UNIVERSIDADE DE LISBOA

FACULDADE DE MEDICINA DENTÁRIA

THE INFLUENCE OF DIFFERENT SURFACE AND HEAT TREATMENTS ON THE BIAXIAL FLEXURAL STRENGTH OF VENEERING CERAMICS FOR ZIRCONIA AND STRENGTH RELIABILITY AND MODE OF FRACTURE OF VENEERING CERAMICS/ ZIRCONIA CORE CERAMICS

José Fausto Pimentel Coelho Lino Carracho

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Thesis supervised by

Prof. Doutor António Vasconcelos Tavares

Dr. Michael Razzoog

José Fausto Pimentel Coelho Lino Carracho

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PREAMBLE

Pursuing a PhD project is a both painful and enjoyable experience. It is just like climbing a high peak, step by step, accompanied with bitterness, adversity, encouragement and frustration. The submission of this dissertation to the Scientific Board of the University Of Lisbon School Of Dentistry is an important key-step in my academic, professional and personal life.

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CHAPTER 1

INTRODUCTION

For decades, an objective of dental restorations has been to replace tooth structure lost by dental disease. However, as dental techniques and materials are improved, both clinicians and patients look for the dental treatments restoring the tooth both functionally and esthetically, not just restoring the function of tooth.

All-ceramic dental materials are becoming the first choice of restorative materials because of their superior biocompatibility and distinct esthetic appeal. However, the major drawback of dental ceramics is the low tensile strength, which results from the presence of surface and internal flaws, a brittleness characteristic of most ceramic materials (O'Brien, 2002; Ritter, 1995).

The brittle behavior of ceramics combined with extreme sensitivity to microcrack-like defects has hampered wider use and limited their application to relatively low stress-bearing areas. Flaws and defects that may grow at the microscopic level have been shown to significantly control their strength characteristics (Tinschert *et al.*, 2000; Kelly, 1995).

A main focus of dental researchers and manufactures has been to improve the strength properties of ceramic materials (Seghi *et al.*, 1990). Various methods and techniques have been recommended to strength dental ceramics (Anderson *et al*., 1993; Anderson *et al*., 1998; Luthardt *et al*., 1999; McLaren *et al.*, 1999; Fischer *et al*., 2001; Fischer *et al*., 2000; Giordano *et al*., 1994; Anusavice *et al*., 1992; Campbell *et al*., 1989; Seghi *et al*., 1995; Rosentiel *et al*., 1993; Burke, 1999; Mclean, 1987).

A strengthened ceramic can be used as the sole material for making an all-ceramic crown, inlay, onlay or veneer restoration or as a core subtract for a ceramic crown (Anusavice, 1996). In an attempt to improve the strength characteristics of all-ceramic systems 2 avenues have been explored. The first is directed toward enhancing the strength of core materials and the second focused on improving processing techniques with the intent of producing a more homogeneous ceramic material. Often both directions are used in the development of new all-ceramic restorative systems with improved strength. Ceramic materials with varied chemical compositions have been developed for use with processing methods combining pressure, high temperature and CAD/CAM (computer-aided design/computer-aided manufacturing) technology (Anderson *et al*., 1993; Anderson *et al*., 1998; O'Brien, 2002).

Until recently, the primary focuses of dental ceramic developers and researchers have concentrated on improving the flexural strength and fracture toughness of the core materials (Anderson *et al*., 1993; Anderson *et al*.; Luthardt *et al*., 1999). Zirconia based restorations are a major example of this progress (Christel *et al*., 1989; Piconi *et al*., 1999). While emphasis has been placed on the development of core ceramics finding stronger veneering porcelains has not been an area of significant research. Thus veneering porcelain strength has remained largely unchanged since the original porcelain jacket crown. Without a major improvement in the strength of the veneering porcelain it is doubtful whether future advances in ceramic core strength will improve the durability of all-ceramic systems.

On the other hand, the development of stronger veneering porcelains, which is now a crucial center of attention of researchers may be the key step to improve the strength of all-ceramic restorations. This idea is supported by different studies that have shown that a thin layer of veneering porcelain fired onto ceramic core material diminish the strength of the 2-layer test specimens (Hopkins, 1989; Zeng *et al.*, 1998). The veneering porcelain is still likely to be the weakest link in the ceramic restoration.

All-ceramic veneering materials, are subjected to different fabrication procedures in the laboratory, and sometimes must be adjusted clinically to allow either proper fitting or occlusion. The processing procedures and/or clinical adjustments are more likely to initiate subcritical flaws or large defects which, upon clinical loading and/or presence of moisture, may grow to a critical situation leading to catastrophic failure. In addition, different surface roughness formed through finishing procedures may cause various stress concentrations and consequently may be accompanied by a reduction in strength (Jager *et al*., 2000).

The high physical properties of many ceramic materials makes surface polishing a difficult task. The development of microcracks during the polishing procedure is not easily avoidable. Moreover, the skill of individual dental technician as well as the adherence to the recommendations of a specific dental material's manufacturer can, to a greater extent, influence the mechanical performance of all-ceramic materials (Chen *et al*., 1999).

The effect of processing procedures, polishing, grinding and glazing on the mechanical properties of some dental materials has been studied by many investigators (Bhrama *et al*., 2002; Giordano *et al*., 1994; Campbell *et al*., 1989; Rosentiel *et al*., 1999; Anusavice, 1991; Giordano *et al*., 1995; Fairhurst *et al*., 1992; Chu *et al*., 2000; Williamson *et al*., 1996; Mecholsky *et al*., 1977; Kosmac *et al*., 1999; Brackett *et al*., 1989; Griggs *et al.*, 1996; Kitazaki *et al.*, 2001; Haharav *et al*., 1999; Denry *et al*., 1999; Anusavice *et al*., 1989; Albakry *et al*., 2003; Isgro *et al*., 2003; Guazzato *et al*., 2003; Guazzato *et al*., 2004). However, there is still controversy concerning the most suitable method that could produce a smooth and strong surface (Williamson *et al*., 1996). The purpose of this study is to contribute to the explanation and resolution of this common clinical problem.

While much research has been conducted to assess the strength of traditional dental porcelain materials, very little information has been reported concerning the relative strength of many of the ceramic materials already in clinical applications. The precision of fit and biocompatibility of specific Zirconia crowns has been studied and found to be excellent for multiple dental applications (Luthardt *et al*., 1999; Piconi *et al*., 1999). However, little data exists regarding the strength and mode of fracture, of dental veneering porcelains used in conjunction with Zirconia based structures (White *et al*., 2005; Aboushelib *et al*., 2006). Therefore, the purpose of this investigation will be to evaluate the influence of different surface and heat treatments on the mechanical properties of Zirconia dental veneering porcelains and the strength, reliability and mode of fracture of Zirconia/veneering porcelain. While it is clear that the long-term clinical performance of Zirconia restorations will depend on many factors, the ability of ceramic materials to withstand fracture is of significant interest.

Ceramic development continues to be a challenge. If a reliable and durable all-ceramic system, which remains yet somewhat elusive, is to be achieved, it is essential to continue the research in order to increase the knowledge about dental ceramics, thereby allowing substantial improvement in the all-ceramic material performance.

History of porcelain

Porcelains have been known for a very long period of time and can be traced back to the early history of human civilization. The Greek word *keramos* means pottery and porcelain traces its ancestry to the primitive potter, whose first attempts were crude, baked in the sun, susceptible to fracture, porous, ugly, and far from perfect. As man experimented to improve them, other elements were added and techniques were enhanced, resulting in the development of three basic types of ceramic materials. Earthenware, that was fired at low temperatures and was relatively porous. Stoneware, which appeared in China about 100 B.C., that was fired at higher temperature, resulted in higher strength and also rendered the material impervious to water. The third material was porcelain or ceramic, obtained by fluxing white china with “China stone” to produce a white translucent stoneware. Porcelain was developed in China about 1000 A.D. and was much stronger and more translucent than earthenware or stoneware (Yamada, 1977; Jones, 1985).

Marco Polo’s experience in China and his return to Florence in 1295 made Europe aware of the beauty of true porcelain. Attempts to uncover the secret of Chinese porcelain manufacture during the seventeenth and eighteenth centuries laid the foundation for the development of a scientific approach to the synthesis of materials. However, it is sad reflection on ceramic science that the secret of Chinese porcelain had to be obtained by an early example of industrial espionage. A Jesuit Father named d'Entrecolles was able to gain confidence of Chinese potters and learn the secret in 1717. It took less than 60 years following this breakthrough for porcelain to be used for the first time as a dental restorative material (Yamada, 1977; Jones, 1985).

The father of modern dentistry “Pierre Fauchard” was a French dentist who is given credit by some for first suggesting the use of porcelain in dentistry as early as 1728 (Yamada, 1977). However, it was Alexis Duchateau who had the idea and Nicholas Dubois de Chemant who fabricated the first pair of all-porcelain dentures in 1790 (Jones, 1985).

The introduction into dentistry of the art of fusing porcelain must stand as one of the most important and significant historic developments in dental materials science. The dental porcelains developed following Duchateau's inspired idea were relatively white and opaque, until 1938 when Elias Wildman was able to formulate a much more translucent porcelain with shades much closer to natural teeth (Felcher, 1932; Clark, 1976). In 1880, porcelain was first applied to restorative dentistry with the development of Richmond and Long & Davis crowns, which attained continuity with the remainder of the tooth by an interfaced layer of metal which was swaged and soldered (McLean *et al*., 1965). Land introduced the porcelain jacket crown, fused on a platinum matrix, in 1887 (Anusavice, 1991). Brewster developed porcelain inlays in 1900 (Anusavice, 1991). Yet porcelain as a restorative material went into a decline soon after this and it was probably due to over enthusiastic use concomitant with ignorance of its physical properties (Clark, 1976; Yamada, 1977; Jones, 1985).

The next step was the introduction of a reinforcing procedure by Swann (Craig, 2006) in which a platinum-iridium-alloy was used as a substructure to which the porcelain was fused. However, technical difficulties in the fabrication of the substructure limited its use. Little more development took place for some years as methylmethacrylate resins began their rise to popularity and it was only after the limitations of resins were realized that the stage was set for the most recent development of ceramics (Clark, 1976).

In 1954 Weinstein (McLean, 1976) patented the use of a castable palladium alloy and a fused-porcelain all-ceramic crown that were the basis of the first ceramic-metal restoration. In no time after that practical investments for high fusing metals emerged, low gold and non-precious alloys were introduced, and the ceramic-metal restoration reached a high degree of sophistication with the development of electronically controlled vacuum-fired ceramic furnaces (Clark, 1976).

In 1965, McLean and Hughes (McLean *et al*., 1965) introduced the aluminous porcelain jacket crown technique by which a core aluminous porcelain was applied and fired on a substrate of platinum foil. Layers of more translucent but weaker porcelain were next applied and fired until the crown form was completed. The foil was subsequently removed from the crown after completion of the firing process (McLean *et al*., 1965). These crowns were more resistant to fracture than the original porcelain jacket crowns but presented high failure rates in the posterior regions of the mouth. To reduce the failure rates of these crowns, McLean and Sced (McLean *et al*., 1976) attempted to strengthen them by bonding the core porcelain to platinum foil which was tin-planted and oxidized. It was believed that the remaining foil would reduce the severity of flaws within the ceramic surface and that improved bonding to the tin oxide layer would reduce the potential for crown debonding and improve the stress distribution in the ceramic. However, clinical data indicates that these crowns should be restricted to restoration of anterior teeth (Grossman *et al*., 1987).

In the past two decades, advances have been made in methods of strengthening of all-ceramic crowns (Andersson *et al*., 1993; Dong *et al*., 1992; Luthardt *et al*., 1999). Today, heat-press injection molded glass ceramics and high strength alumina or Zirconia core ceramics constitute the strongest dental porcelains available (Probster *et al*., 1990; Claus, 1990; Andersson *et al*., 1993; Dong *et al*., 1992; Luthardt *et al*., 1999).

The restoration of a patient's natural tooth requires an esthetic quality that is life-like in appearance and beyond recognition as being artificial. To achieve this goal, the most frequently used restoration has been the porcelain fused to metal crown. This treatment approach offers strength and excellent marginal adaptation and when the metal substructure is combined with porcelain, provides an acceptable esthetic result. However, for some patients, the porcelain fused to metal restoration has not fulfilled their demands for naturalness. As a result, the profession as the potential answer to esthetically demanding situations has suggested the concept of the all-ceramic restoration.

The development of all-ceramic systems offered many improvements such as increased translucency, adaptability and biocompatibility over the porcelain fused to metal restoration (St John, 2007; Bayne, 2005; O'Brien, 2000). All-ceramic materials are inert, resistant to corrosion, and have low temperature and electrical conductivity (Kelly, 2004; Anusavice, 1992). The biggest advantage of all-ceramic crown may be its' natural tooth-like appearance. High strength ceramic copings mimic the light transmission properties of natural tooth by improving the translucency of light through the restoration and the underlying tooth structure. This characteristic of ceramic copings solves the esthetic problem resulting from the opacity of metal substructures of conventional porcelain fused to metal crowns. The absence of metal substructures and translucency of all-ceramic coping materials enhances the final esthetics of restorations by allowing light transmission through the restoration and the underlying tooth or implant abutment structure. The coping is veneered with shaded and translucent porcelains to scatter the light, penetrating the porcelain in a manner similar to natural enamel and dentin.

Composition of dental ceramics

Ceramics, materials largely formed from metallic oxides, have long been used as dental materials because of their biocompatibility, stability, durability, low thermal conductivity, and excellent optical qualities (Kelly, 2004;

Denry, 1996; Anusavice, 1991). Ceramics from the finest porcelain are composed essentially of the same constituents: feldspar (K_2O Al_2O_3 $6SiO_2$), silica (quartz (SiO_2), or flint), kaolin (clay) (Al_2O_3 $2SiO_2$ $2H_2O$), and metallic pigments as opacifiers and color modifiers (Craig, 2006; O'Brien, 2002).

The quality of any ceramic depends on the correct choice and proportioning of these elements and on the control of firing procedures. Only the purest ingredients are used in the manufacture of dental ceramics because of the stringent requirements of fracture and abrasion resistance, low thermal expansion, insolubility, biocompatibility, color stability and translucency.

The various components of the porcelain blended together by the manufacturer result in two principal phases. One is the vitreous (or glass) phase, and the other is the crystalline (or mineral) phase. The vitreous phase formed during the firing process has properties typical of glass, such as brittleness, non-directional fracture pattern, flow under stress and high surface tension in the fluid state. The crystalline phase includes the silica or quartz and certain metallic oxides. The vitreous phase is prominent in dental porcelain powders and contributes to many characteristics properties as well as bonding together the crystalline particles (Craig, 2006).

Feldspar is chemically designated as potassium aluminum silicate with a composition of (K_2O Al_2O_3 $6SiO_2$). When heated to its fusing temperature, approximately 1290°C, it becomes glassy. Unless overheated, feldspar retains its form without rounding, therefore maintaining the contours of porcelain restorations. Feldspar powder its difficult to obtain. Each piece of feldspar needs to be broken with a steel hammer, and only the uniformly light-colored pieces are selected for use in porcelain. These pieces are ground in ball mills until they become a fine powder. Screening to remove the coarser particles carefully controls the final particle size, and flotation processes are used to remove the excessively fine particles. The dry powder is then slowly vibrated down inclined planes equipped with a series of narrow ledges formed by induction magnets that separate remaining iron contaminants and make feldspar ready for use.

Pure quartz crystals (silica, SiO_2) are used in dental porcelain. Traces of iron also may be present in quartz and must be removed to prevent discoloration. The preparation of silica is similar to that of feldspar except that silica is ground to the finest grain size possible. Silica remains unchanged at the temperature normally used in firing porcelain, and this contributes stability to the mass during heating by providing a framework for the other ingredients.

Kaolin is produced in nature by weathering of feldspar, during which acid waters wash out the soluble potassium silicate. The residue (kaolin) is deposited along the banks and the bottom of streams in the form of clay. The kaolin, represented by the formula (Al_2O_3 $2SiO_2$ $2H_2O$), is prepared by repeated washings with water until all foreign materials are separated. The clay is then allowed to settle, and after it has been dried and screened, the nearly white powder is ready for use. Kaolin gives porcelain its opaque quality. When mixed with water, it becomes sticky and aids in forming a workable mass of the porcelain during molding. When subjected to high heat, it adheres to the framework of quartz particles and shrinks considerably.

The coloring pigments added to the porcelain are called “color frits”. These powders are added in small amounts to obtain the delicate shades necessary to imitate natural teeth. Pigments are prepared by grinding together metallic oxides with fine glass or feldspar, fusing the mixture in a furnace, and regrinding it to a powder. The metallic pigments include: titanium oxides (yellow-brown), iron or nickel oxides (brown), manganese oxide (lavender), cobalt oxide (blue), copper or chromium oxides (green), uranium oxide or lanthanide earths (fluorescence) and tin oxide which provides opacity.

The manufacturers do not publicize the exact formulas for their porcelains. Of the formulas published in the literature, feldspar constitutes between 75% to 85% of the total; quartz 12% to 22%; and kaolin 3% to 5%. Pigments constitute a small percentage of the mixture (O'Brien, 2002; Della Bona *et al*., 2002).

Dental ceramics have a composite structure. Materials for metal-ceramic restorations contain a vitreous phase, also called glassy matrix, that represents 75 to 85% by volume and are reinforced by various crystalline phases (Denry *et al*., 1995). The choice of the crystalline phase in compositions for metal-ceramic restorations was initially dictated by the need for matching the thermal contraction coefficient of the porcelain close to that of the metallic infrastructure in order to avoid the development of tensile stresses within the porcelain when cooled. Most ceramics for metal-ceramic restorations contain from 15 to 25 vol% leucite as their major crystalline phase, but changes in the leucite volume fraction can occur during thermal treatment of dental porcelains (Mackert *et al*., 1991). Leucite ($KAlSi_2O_6$) is a potassium alumino-silicate with a high thermal expansion coefficient (Mackert *et al.*, 1996). Its name comes from the Greek word for "white" in allusion to its typical color. At high temperatures, leucite is isometric and will form the isometric trapezohedron crystal form. Interestingly, as leucite cools, an isometric structure becomes unstable and transforms into a tetragonal structure without altering the outward shape. Although the mineral is actually tetragonal, the outward shape is pseudo-isometric and thus the crystal form is actually pseudo-trapezohedral. For this reason leucite is considered a member of the feldspathoid group of minerals. Leucite, like other feldspathoids, is found in silica poor rocks containing other silica poor minerals and no quartz. If quartz were present when the melt was crystallizing, it would react with any feldspathoids and form feldspar. At one time leucite was used as a source of potassium and aluminum. Probably due to the high aluminum to silicon ratio, acids easily destroy its structure and this frees the aluminum ions.

Materials for all-ceramic restorations use a wider variety of crystalline phases as reinforcing agents and contain up to 90% by volume of crystalline phase. The nature, amount, and particle size distribution of the crystalline phase directly influence the mechanical and optical properties of the material (Morena *et al.*, 1986; Kon *et al.*, 1994). The match between the refractive indices of the crystalline phase and glassy matrix is a key factor for controlling the translucency of the porcelain. Similarly, the match between the thermal expansion coefficients of the crystalline phase and glassy matrix is critical in controlling residual thermal stresses within the porcelain.

Brittle materials such as ceramics contain at least two populations of flaws: fabrication defects and surface cracks. Fabrication defects are created during processing and consist of voids or inclusions generated during sintering. Microcracks develop upon cooling in feldspathic porcelains and are due to thermal contraction mismatch between both the crystals and the glassy matrix (Mackert *et al*., 1996) or between the porcelain and the metal or ceramic substrate. Condensation of a ceramic slurry by hand prior to sintering may introduce porosity. Sintering under vacuum has been shown to reduce the amount of porosity in dental porcelains from 5.6 to 0.56% (Anusavice *et al*., 1991). Surface cracks are induced by machining or grinding. The average natural flaw size varies from 20 to 50 µm (Anusavice *et al.*, 1991). Usually, failure of the ceramic originates from the most severe flaw. The size and spatial distribution of the flaws justify the necessity of a statistical approach to failure analysis.

Surface crystallization of leucite can be induced by seeding the surface of a feldspathic glass with leucite particles (Holand *et al.*, 2000). Ceramic materials for all-ceramic restorations are in contact with refractory die materials during firing or pressing at high temperatures. Surface reactions have also been reported between glass-ceramics and the refractory embedment used during the crystallization process, thereby modifying the mechanical properties of the final product (Campbell *et al*., 1989; Denry *et al*., 1993). Diffusion processes are temperature-dependent, and surface reactions are likely to occur between the porcelain and the refractory die material.

Classification of dental ceramics

Dental ceramics are divided into different groups according to their chemical composition (feldspar, leucite, alumina, magnesia, Zirconia, glass alumina, and glass-ceramics), application (tooth reconstruction, ceramic covering metals, veneers, inlay, onlays, crowns and fixed partial dentures), the manufacturing procedure, or the structure of the material (cast metal, burnished metal foil, glass ceramics, CAD/CAM ceramic, and sintered ceramic core).

Sintering, pressing, casting, slip-casting followed by glass infiltration and machining (manually or computer operated) are different manufacturing methods that can be used for making ceramic restorations.

However, dental porcelains are generally classified, according to firing temperatures: high melting point (1201°-1450°C), medium melting point (1051°-1200°C), low melting point (850°-1050°C) and very low melting point (< 850°) (O'Brien, 2002; Craig, 2006;).

High fusing porcelains have high feldspar, low kaolin and low quartz. The resultant mix of minerals is mixed with small quantities of starch or flour, Vaseline and water. The mix is compressed in a mold and heated to gelatinize the organic components. The pieces are extracted from the mold, dried and fired. Formerly, porcelain of this type was used in the fabrication of high fusing porcelain jacket crowns and denture teeth (Southan *et al.*, 1972; Schmitt, 1984). Now its' use is almost entirely confined to manufacturing denture teeth (Engelmeier, 1996). The principal advantage of high fusing porcelains is the ability to be repaired, added to, stained or glazed without distortion (Steppo, 1968).

Medium and low fusing porcelains are modified by the manufacturers with chemical or fluxes of low melting temperature and are refused and reground (Risito *et al*., 1995). This results in narrower fusing ranges and increased tendency for the porcelain to slump during repair or when making additions, staining or glazing (Scherer *et al*., 1991). Refusing and regrinding, however, increase the homogeneity of the powder facilitating handling and fusing operations. Concomitantly, more homogeneity of the powder means that fewer flaws are introduced in the restoration decreasing the potential for early failure (Seghi *et al*., 1990; White *et al*., 1992).

The high and medium fusing porcelains are unstable upon repeated episodes of heating and cooling and for this reason they are only used for denture teeth and occasionally for pontics (Engelmeier, 1996). To the contrary, the low fusing porcelains, by nature of a high proportion of potassium and sodium oxides, may be fired repeatedly without chemical change, and are, therefore, used for single crowns, veneers, inlays and onlays and fixed partial dentures (Drummond *et al*., 2000; Kon *et al*., 2001).

Ceramics with especially low melting points are used to cover titanium frameworks (or titanium-based alloys), since their coefficient of thermal expansion is close to that of the metal. These ceramics can also be used to cover certain low melting type IV gold alloys. However, some of the ceramics with low melting point can also be used for conventional metal-ceramic alloys (highly noble, noble, or no noble metals), since they have sufficiently high coefficients of thermal expansion.

Mechanical properties of dental ceramics

Dental ceramics offer considerable resistance to abrasion, are resistant to degradation in the oral cavity and are biologically compatible (Hanks *et al.*, 1996; O'Brien, 2000; Denry, 1996). Their vitreous structure, consisting of an irregular network of silica, produces physical properties typical of glass, including brittleness and lack of a definite melting temperature (Meyer *et al*., 1976; O'Brien, 2002; Tinschert *et al*., 2000).

Dental porcelain has an inherent fragility in tension. The largely covalent and/or ionic bonded structure of ceramics results in their resistance to chemical degradation in the oral environment, but also imparts brittleness. While the theoretical tensile strength of porcelain is dependent upon the silicon-oxygen bond, the practical strength is 100-1000 times less than the nominal strength extrapolated from the elastic modulus (Ban *et al*., 1990; Evans, 1982).

Flaws are generally present on the surface of glasses that have been melted and fabricated at high temperature. The chief cause of such flaws is abrasion, corrosion (especially by water vapor), and surface devitrification (Anusavice *et al.*, 1991; Campbell *et al*., 1989). Dental porcelains are basically borosilicate and/or feldspathic glass. Unlike glass, dental porcelain is fabricated by powder sintering that can give rise to a distribution of surface flaws and internal voids. These imperfections present in fused dental ceramics limit its strength (Anusavice, 1992; Ritter, 1995; Kelly, 1995; Kelly, 1989).

The strength of porcelain is governed by the presence of small flaws or cracks. When stressed in tension, according to crack propagation theory (Griffith, 1920), small flaws tend to open and propagate, resulting in a low tensile strength. The tendency for crack propagation is resisted by the porcelain when a crack progresses to the metal substructure (metal-ceramic systems), or to a high tensile strength crystal (alumina or Zirconia) within the matrix, as in some all-ceramic systems. Compressive stresses tend to close flaws; hence porcelains are much stronger in compression (Mumford, 1976).

One of the goals when attempting to improve all-ceramic restorations is to maximize the mechanical characteristics of the material. Although the relation between the mechanical properties of a ceramic and its clinical performance is influenced by many variables, some of these properties, namely strength and fracture toughness, have often been the first parameters investigated to understand the clinical potentiality and limits of a dental ceramic (Seghi *et al*., 1990; Seghi *et al*., 1995; Mecholsky, 1995).

Strength is defined as the ultimate stress that is necessary to cause fracture or plastic deformation and is strongly affected by the size of flaws and defects present on the surface of the tested material. The inability of ceramics to reduce the tensile stresses at the tip of the cracks by deforming explains why they are much weaker in tension than in compression, and also why dental restorations normally fail in areas of tensile stresses. Therefore, tensile strength is considered more meaningful compared to compressive strength when testing this property in brittle dental ceramics (Kelly, 1995).

The fracture toughness is defined as the mechanical resistance of the material to crack propagation and the resulting catastrophic failure. Unlike strength, which depends on the size of the initiating cracks present on the surface of that particular specimen, the fracture toughness of a material is generally independent of the size of the initiating crack, the specimen shape, and the stress concentrations acting on the force. Fracture toughness is thus considered a more meaningful property than strength when validating a material's suitability for structural components (Mecholsky, 1995).

Stress is the internal reaction to externally applied forces and is equal in intensity, but opposite in direction, to the external force. Stresses can happen with compression, tension or shearing forces and are distributed over a given area (Flinn *et al*., 1981; Jones, 1983). Brittle materials, such as dental ceramics, have a limited capacity for distributing localized stress at nominal temperatures. The critical strain (change in length per unit length of the body when it is subjected to a stress) of a dental ceramics is low; the material deformation of approximately 0.1% before fracture (Hondrum, 1992; Baran *et al*., 2001).

Failure of a porcelain crown intraorally occurs by a combination of bending and tensional forces on the crown. These forces involve tensile stresses, upon comparatively light occlusal loading, on the inner surfaces of the crown particularly at the cervical third (McLean *et al*., 1976).

Ceramics are much weaker in tension or transverse loading than in compression (Ritter, 1995). According to Griffith's fracture theory (Griffith, 1920) stress concentrations are formed around small flaws. In ductile materials, sufficiently mobile slip systems are present to absorb energy and to allow plastic deformation through which concentration can be relieved. In ceramics, on the other hand, few if any dislocations move under stress. Consequently, stress concentration around cracks in ceramics are high since they lack the ductility to deform and reduce sharp angles and there are no energy absorbing processes resulting from dislocation motion. Tensile or bending stresses tend to extend cracks while compressive stresses tend to inhibit crack propagation (O'Brien, 1985; McLean *et a*l., 1991).

In practice it should be noted that for a wide range of ceramics and glass-like materials, the critical strain at fracture would range between 0.05 and 0.2% (McLean *et a*l., 1991). It has been shown that when crystalline grains of high strength and elasticity were introduced into a glass of similar thermal expansion, the strength and modulus of elasticity of the mixtures increased progressively with the proportion of the crystalline phase (Seghi *et al*., 1990, Anusavice, 1991). It has also been shown, that in this type of system, crack propagation was indiscriminate through both glass and crystal phase. Thus, the energy required for crack propagation had to be higher than required to fracture the glass alone. This method of strengthening glass was the incentive behind aluminous porcelain developed by McLean and Hughes in 1965 (McLean *et a*l., 1965).

Dental ceramic's strength may also be affected by the presence of residual stresses which develop as a result of uneven cooling of fused porcelain or difference in coefficients of thermal expansion among different layers of porcelain fused together (Jones, 1983; Fairhurst *et al*., 1989; Dehoff *et al*., 1989; Twiggs *et al.*, 1989; Mackert *et al*., 1991; White, 1993). Residual compressive stresses which exist on the outer layer of porcelain or in the porcelain along the porcelain-metal interface will inhibit crack propagation and increase strength (O'Brien, 1984; Morena *et al*., 1986).

Static fatigue of dental ceramics, or delayed failure of glass and ceramics, is generally believed to be one of the causes of failure of ceramic restorations in the oral environment (Jones, 1985; White *et al*., 1997). Static fatigue of ceramics is caused by stress-enhancing chemical reactions aided by water vapor acting within the small cracks or flaws in the surface of the porcelain, which causes the flaws to grow to critical dimensions (Jung *et al*., 2000; Zang *et al*., 2005; Okutan *et al*., 2006). This allows spontaneous crack propagation (Mitov *et al*., 2008). Absorbed moisture in static fatigue lowers the energy required at the crack surface to create vacancies at the crack tip, thereby decreasing the apparent activation energy for crack growth.

Mechanical fatigue must also be considered when dental ceramics are being evaluated for their strength. Mechanical fatigue has been defined by the America Society of Testing Materials (1979) as: "The process of progressive localized permanent structural change occurring in a material subjected to conditions which produce fluctuating stresses and strains at some point or points and which may culminate in cracks or complete fracture after a sufficient number of fluctuation" (White, 1993).

Prostheses made from materials which undergo fast rates of mechanical fatigue would be expected to undergo mechanical failure much more quickly than those with lower rates of fatigue. Differing rates of mechanical fatigue could profoundly influence the lifetimes of ceramic prostheses and the selection of ceramics for clinical purposes (Morena *et al*., 1986; Anusavice *et al*., 1989).

Until recently, it has been assumed that ceramic materials do not undergo mechanical fatigue. White has demonstrated, by using an indentation technique, the existence of mechanically induced cyclic fatigue in a feldspathic dental porcelain under ambient conditions (White, 1993). The susceptibilities of a range of dental ceramics to mechanical fatigue, and the possible interaction between mechanical and chemical static fatigue must be investigated so that materials and techniques giving ceramic restorations the longest possible lifetimes can be identified (White *et al*., 1997).

The coefficient of thermal expansion is a very important property of dental materials as it represents the change in length per unit length of a material for a 1°C change in temperature (O'Brien, 2002). The ceramics used for metal-ceramic or all-ceramic restorations must have thermal expansion coefficients compatible with dental alloys or ceramic substructures (Fairhurst *et al*., 1980; Steiner *et al*., 1997; Isgro *et al*., 2004; Ficher *et al*., 2007). The higher expansion is possible by addition of potassium oxide and the formation of a high-expansion phase called leucite ($KAlSi_2O_6$) (Piché *et al*., 1994; Mackert *et al*., 1996; Mackert *et al*., 1996; Tinschert *et al*., 2000).

Porcelain has a coefficient of thermal expansion ($12x10^{-6}$/°C) slightly lower than of tooth structure (Craig, 2006). This is very important to the clinical strength characteristics of all-ceramic materials, especially those that use core made of alumina or Zirconia (de Kler *et al*., 2007; Fischer *et al*., 2007). When porcelains of different expansions are fused together large interfacial stresses develop, which may be sufficient to cause immediate fracture when the restoration is cooling after removal from the furnace. On the other hand, the stresses may not cause an immediate fracture, but additional forces may be generated while trial fitting, during cementation and during mastication. Any or all of these increased stresses may cause fracture (Mumford, 1976; Kon *et al*., 1994; Fischer *et al*., 2008).

Moisture contamination is also a significant clinical factor in weakening of the glass surface, as water plays an important role in the static fatigue of glass and produces a time-dependent reduction in strength (Wang *et al*., 1958 Anusavice *et al*, 1989; Scherrer *et al*., 2001; Lohbauer *et al*., 2008). This process includes the replacement of the alkali ions in glass by hydrogen ions, which interact with water molecules into the spaces originally occupied by the alkali. Static fatigue results from a stress-dependent chemical reaction between water vapor and the surface faults, causing the flaw to grow to critical dimensions and resulting in crack propagation (Tinschert *et al*., 2000; Ritrer, 1995; Kelly, 1995).

The properties of fused porcelain that have been most reported include the linear and volumetric shrinkage (Tinschert *et al*., 2000; Suansuwan *et al*., 2001; Rizkalla *et al*., 2004). The linear shrinkage of glazed porcelain has been reported to be approximately 14% for low-fusing porcelain and 11.5% for high-fusing porcelain. Compensating for the comparatively large shrinkage that occurs during firing is accomplished by the appropriate condensation technique (Suansuwan *et al*., 2001). When porcelain powder is mixed with water or a ceramic mixing fluid and the fluid is removed, the bulk of the mass will shrink until the solid particles touch one another. If during this process the particles are induced to move, they will find a position in closer proximity and will interdigitate with one another. This only occurs while sufficient moisture is present to allow movement, as condensation is a two-part process: agitation of the particles and removal of excess moisture. Correct removal of moisture is a key step not only to limit the shrinkage of dental ceramics but also to eliminate flaws between different layers increasing the final resistance of the restoration (Baker *et al*., 1993; Palin *et al*., 2001).

Ceramics are the hardest restorative materials used in dentistry, being significantly harder than tooth enamel (Mahalick *et al*., 1971; Monasky *et al*., 1971). Therefore whenever porcelain restorations oppose natural dentition there is a possibility for the natural teeth to wear (Clelland *et al*., 2001; Hacker *et al*., 1996). In a study by Jagger and co-workers (Jagger *et al*., 1994) a wear machine was used for abrasive wear tests on unglazed, glazed and polished porcelain opposing to human enamel. Their results showed that the amount of enamel wear produced by both glazed and unglazed porcelain was similar, however, that produced by polished porcelain was substantially less.

Adjusted porcelain surfaces should be reglazed to restore the surface finish, however reglazing is not always convenient or possible. Many techniques for polishing porcelain have been evaluated and the literature is controversial in regards to what produces less enamel wear, reglazed or polished porcelain (Palmer *et al*., 1991; Delong *et al*., 1992; Hudson *et al*., 1995; Sulong *et al*., 1990). Nasr and co-workers (Nasr *et al*., 1989) evaluated and compared the quality of autoglazed, overglazed and polished porcelain surfaces using interference microscopy. They showed that a smoother surface was achieved with the overglaze, followed by autoglaze. They concluded that polishing of porcelain should only be restricted to minute areas of spot grinding. In a study by Haywood and co-workers (Haywood *et al*., 1988) SEM specular reflectance was used to analyze the surface texture of autoglazed and polished porcelain. They found that polished porcelain surfaces could equal or even surpass the smoothness of glazed porcelain. Recent investigations have also shown that polishing porcelain surfaces after adjustment produces a surface which is smoother than that of a glazed porcelain standard and may contribute to less enamel wear (Haywood *et al*., 1988; Brewer *et al*., 1990).

Optical properties of dental ceramics

The appearance of esthetic dental restorations is of crucial importance. Porcelain restorations are required to interact with light in such a way as to mimic normal tooth structure. Knowledge of the optical properties of these materials is thus needed for the purpose of fabricating an esthetically pleasing restoration.

Porcelain is a two-phase system, a matrix of silicate glass and a crystalline second phase dispersed in the primary phase. The secondary phase consists mainly of crystalline quartz, mullite and metal oxides. The approximate composition of dental porcelain is as following: feldspar 81%, quartz 15%, kaolin 4%, and pigments less than 1% (O'Brien, 2002).

During the manufacturing process of porcelain powders finely ground particles of feldspar, quartz and kaolin are fused together at high temperatures to form a frit. During this process kaolin gives rise to some crystalline particles called mullite. These particles are relatively small in size thus scattering light and reducing translucency. Adjusting the ratio of kaolin and feldspar controls the concentration of these particles. Voids in the structure of porcelain also decrease translucency (Ban *et al*., 1998).

The frit is subsequently reground to form glass powder. Mixing high concentrations of finely ground metal oxides with the glass powders forms highly colored frits. Mixture of these highly colored frits and the glass powders are refined to form porcelain frit of the desired color. This frit is then reground to the powder which is finally used to fabricate restorations (Mabie *et al*., 1983).

Porcelain is the most stable tooth colored material available (Heydecke *et al*., 2001; Sailer *et al*., 2007; Samra *et al*., 2008; Yalmaz *et al*., 2008). The oxides used as colorants result in a range of tooth-like colors and do not undergo any change in shade after firing is complete. The smooth glossy surface resists the adherence of exogenous stains and allows regular and diffuse transmission as well as diffuse and specular reflection light (Yalmaz *et al*., 2008). Therefore, porcelain has the potential to reproduce texture, depth of color, and the translucency of the natural teeth. The translucencies of opaque, body, and incisal porcelains differ considerably. Opaque porcelains have very low translucency values to mask metal and ceramic cores substrate surfaces. Body porcelain translucency values range between 20 and 35%. Incisal porcelains have the highest values of translucency ranging between 45 and 50% (Craig, 2006).

The color of the dental porcelain at this point is completely dependent upon the colors of the materials used to make the porcelain powder. If the dental porcelain is manufactured as a single phase glass in which all the oxide constituents are completely taken into solution, the resultant porcelain should be transparent. However, most dental porcelains exhibit a slightly greenish hue. To reduce this effect, the base porcelain frit must be shaded before it is used to fabricate the restoration.

The dental porcelain frit may be shaded by the addition of a concentrated color frit. These shaded glasses are prepared by fritting high temperature-resistant pigments, generally metallic oxides, into the basic glass. The glass will then be highly color-saturated and when ground to a fine powder can be used to modify the uncolored porcelain powder. This may be accomplished with small amounts of shaded frit.

The color pigments used in dental porcelain generally consists of the following:

Pink – Chromium-tin or chromium-alumina. These pigments are stable up to a firing temperature of 1350°C and are particularly useful in eliminating the greenish hue in the glass and giving a warm tone to the porcelain

Yellow – Indium or praseodymium (lemon) are probably the most stable pigments for producing an ivory shade. Vanadium-zirconium or tin oxide plus chromium may be used but they are not as stable.

Blue – Cobalt salts are used to produce this color and are particularly useful for producing some of the enamel shades.

Green – Chromium oxide is the main pigment for producing a greenish color: However, it should be avoided in dental porcelain whenever possible, since green is the characteristic color of glass. In addition, the main complaint from technicians is that some porcelain assumes a greenish hue after baking and the inherent greenness of the basic dental porcelain can be accentuated by over-firing (over-vitrification).

Gray – Iron oxide (black) or platinum gray are useful pigments for producing enamels or for addition to a grayer section of the dentine colors. Incorporation of gray colors can also give an effect of translucency.

The addition of concentrated color frits to dental porcelain is insufficient to produce a life-like tooth effect since the translucency of the porcelain is still too high. In order to address such a common problem opacifying agents are used in the porcelain.

Opacifying agents usually consist of finely ground metal oxides (smaller than 5μ). Metal oxides commonly used for this purpose are: 1) cerium oxide, 2) titanium oxide, and 3) zirconium oxide. Zirconium oxide is the most popular opacifying agent and is usually added along with the concentrated color frit to the uncolored porcelain during the final preparation of the porcelain powder. The actual amounts of opacifying agents used are often determined initially by trial and error until a thin fired porcelain blank assumes just the right degree of translucency and the color saturation is correctly balanced.

Porcelain powders can generally be classified by the amount and type of pigments they contain. Opaque porcelain has a high concentration of opaque pigments. Gingival porcelain may have a slightly different color and a higher concentration of pigments than that used for body porcelain. Incisal porcelain has the least amount of pigments and the greatest translucency.

The manipulation of porcelain during the fabrication of a restoration also affects the optical properties. The two major factors to consider are: 1) the manner and degree of condensation of the powder before firing

(Rasmussen *et al.*, 1997), and 2) the firing procedure used (Barghi, 1982; Uludag *et al*., 2007; Ozturk *et al*., 2008; Celik *et al*., 2008).

The degree of condensation before firing is related to the ability of the powder particles to flow together during the firing process and thus affects the amount of air incorporated into the material. The trapped air acts as a second phase and provides a number of finely dispersed areas of different refractive index than the glass. This is a characteristic optical property of porcelain and is desirable to an extent. However, if large amounts of air are incorporated into the material the optical properties can change substantially. Porcelain powders contain particles of different sizes to allow a greater degree of condensation of the powder than is possible with a powder of uniform sized particles (Meyer *et al*., 1976; Piddock *et al*., 1984; Rasmussen *et al.*, 1997).

During firing the porcelain powder forms glass bridges between the particles. This is called sintering and the degree of sintering also affects the appearance of the porcelain. Bisque bake porcelain has little sintering and a high degree of dispersed air pockets. It thus appears quite opaque and light in color (Cheung *et al*., 2002).

As the degree of firing is increased to a glaze, the sintering process continues. The number of air pockets decreases and the material becomes less opaque. The characteristic surface glaze of porcelain is due to the fact that porcelain is a poor conductor of heat (Cheung *et al*., 2002). The surface layer of porcelain reaches a higher temperature faster and longer than the rest of the material and therefore undergoes more sintering. This layer has fewer air bubbles and thus appears more translucent. If the firing process goes too far, the sintering continues to the point where virtually all of the particles are sintered together and the porcelain loses its desired translucency and becomes transparent.

The particle size also plays a role in the sintering process (Rasmussen *et al.*, 1997). The more compact the condensation the less distance there is for the glass bridges to form and the faster the materials sinters. Using particles of different size increases the degree of condensation. However, using a high number of small particles also increases the surface area which tends to increase the amount of air incorporated into the material, therefore delaying the sintering process.

Fusion of dental ceramics

Dental ceramics restorations can be fused by two different techniques. The first method is by temperature control where the furnace temperature is raised at a constant rate until a specified temperature is reached. The second method of fusing porcelain is by a controlled temperature and specified time. The temperature is raised at a given rate until certain levels are reached, after which the temperature is maintained for a pre-determined period until the desired reactions are completed. The time/temperature method is generally preferred as it is less critical and more likely to produce a uniform product (Anusavice, 1993). Porcelain is a poor thermal conductor (Rosenblum *et al.*, 1997), and for this reason too rapid heating may overfuse the outer layers before the inner portion is properly fused. Also, vacuum firing produces slightly less porosity and less surface roughness than air firing (Anusavice, 1993; Kelly *et al.*, 1996).

During firing the porcelain undergoes several changes (Craig, 2006). The first change involves the loss of water which was added to the powder to form the workable mix. The excess water is partially removed by warming the restoration during the pre-heating cycle. This prevents the sudden formation of steam and subsequent physical damage to the porcelain mass. After the restoration is placed in the furnace, both free and combined water are removed in various stages of heating until a temperature of 480°C is reached.

The second change occurs as the temperature is raised and the particles of porcelain fuse together by sintering. During this densification there is a change in volume, which is inversely proportional to both the viscosity of the mix and the particle size.

The glazing stage represents the last change. Glazing is reached in the last firing and is held only long enough for a glossy surface to form. Overglazing produces to thick a glaze resulting in an increase in porosity, loss of strength, form and color of the restoration. Slow cooling of the fired mass is necessary to prevent surface crazing or cracking.

Methods of fabrication of dental ceramics

Dental ceramics are usually processed by sintering, but in the last few years, processing techniques used for high-technology ceramic materials have been applied to dental ceramics, leading to the development of glass-ceramics, slip-cast ceramics, heat-pressed ceramics, and CAD/CAM ceramics. When applied to ceramic materials, the sintering process is defined as the "transformation of an originally porous compact to a strong, dense ceramic". It can be described as a complex sequence of high-temperature reactions occurring above the softening point of the porcelain and leading to partial melting of the glassy matrix, with coalescence of the powder particles. During sintering, the density of the porcelain greatly increases and is associated with volume shrinkage of between 30 and 40%. Clinically, the amount of shrinkage still constitutes a problem in metal-ceramic restorations with all-porcelain margins, decreasing the marginal fit.

Dental porcelains are typically comprised of a fine powder of glass-like particles. To fabricate a dental restoration, water or some suitable liquid is added to the powder. A wet, sandy mix is created which can be formed into desired shapes and then fused by heat to produce a solid substance similar to glass. In this manner, porcelain may be enameled to a metal or simply baked into a solid mass of pure porcelain. Restorations are usually fabricated on a replica or die of the prepared tooth. Materials may be added to the porcelain powders which improve color and strength (O'Brien 2000). Several methods for the fabrication of dental all-ceramic porcelain restorations are described in the literature; however four main techniques are usually employed.

The first glass-ceramics were developed in the late 1950s (Stookey, 1958). Glass-ceramics are polycrystalline solids prepared by the controlled crystallization of glasses. The crystallization is achieved when the glass is submitted to a heat treatment during which crystal nucleation and growth are thermodynamically possible. Proper control of the crystallization heat treatment is necessary to ensure the nucleation of a sufficient number of crystals and their growth to an effective size. The dual nature of glass-ceramic materials confers upon them the esthetic, mechanical, and chemical qualities of ceramics as well as the ability to be cast and processed as glasses. These characteristics are of great interest for dental applications. Machinability is another property desirable for the maximum utility of glass-ceramics as dental materials. The ability of glass-ceramics to be machined is closely related to the nature and particle size of the crystalline phase that develops during the crystallization heat treatment. Machinable glass-ceramics for industrial as well as dental applications often contain mica as a major crystalline phase.

Hot-pressed ceramics constitute another application of high technology to dentistry. This process relies on the application of external pressure at elevated temperatures to obtain sintering of the ceramic body. Hot-pressed ceramics are also called "heat-pressed" ceramics. Hot-pressing classically helps avoid large pores caused by non-uniform mixing. It also prevents extensive grain growth or secondary crystallization, considering the temperature at which sintering is obtained. This technique is initiated by creating the restoration in wax. The wax pattern is lifted from the die and invested or surrounded by a mix of "plaster-like" material which is allowed to harden. A channel or opening leads from the outer surface of the investment into the wax pattern. Wax is eliminated from the investment during a burnout procedure. The dental porcelain, provided in powder or ingot form, is placed in a special hot press and is melted and forced under pressure into the opening of the investment. The melted material fills the void created by the wax pattern. After cooling, the hardened ceramic is broken out of the investment. Where desired, color or feldspathic porcelain can be baked onto the surface of the restoration to simulate tooth color.

The mechanical properties of many ceramic systems are maximized with high density and small grain size. Therefore, optimum properties can be obtained by hot-pressing techniques. In spite of their excellent esthetic qualities and their good biological compatibility, dental ceramics, like all ceramic materials, are brittle. They are susceptible to fracture at the time of placement or during function.

Another method is the slip-cast technique. Slip-casting has long been used in the ceramic industry to make sanitary ware. The fabrication of dental all-ceramic restorations by this method requires that they be made on a special gypsum die of the tooth preparation. Slip-casting involves the condensation of an aqueous porcelain slip on a refractory die. The porosity of the refractory die helps condensation by absorbing the water from the slip by capillary action. The piece is then fired at high temperature on the refractory die. This firing process allows the grains of the material to partially fuse at their grain boundaries while the gypsum die shrinks. Usually, the refractory shrinks more than the condensed slip, and the piece can be separated easily after being fired. The core may then be lifted off the die. Final shaping must be done at this stage since the subsequent infusion of the glass matrix makes finishing operations very difficult because of the hardness of the final ceramic. After shaping, the porous material is ready for the infusion of the glass matrix. A specially prepared low-fusing glass of matching thermal expansion is painted over the external surface of the core, and then placed on a thick piece of platinum foil. The fired porous core is later glass-infiltrated, a unique process in which molten glass is drawn into the pores by capillary action at high temperature resulting in a very dense interpenetrating phase composite structure that has a minimal shrinkage of about 0.21% (Campbell *et al*., 1995). Materials processed by slip-casting tend to exhibit reduced porosity, fewer defects from processing, and higher toughness than conventional feldspathic porcelains. The core is then all-ceramic crowned with the appropriated low fusing porcelain.

A revolutionary method of fabricating dental restorations involves the computer-aided-design/computer-aided-manufacturing (CAD/CAM) technique. In such method, a 3-dimensional photo or image is captured of the prepared tooth over which a dental restoration is to be placed. The digitized image is supplied to the CAD/CAM system software, displaying the 3-dimensional picture on a viewing screen. The dental practitioner selects the most suitable tooth form from a plurality of tooth forms stored in the CAD/CAM system and projects the image of the selected tooth form over the prepared tooth until an optimum positioning and fit of the dental restoration is obtained. The digital data concerning the dental restoration thus formed are supplied to a numerically controlled milling machine operating in three dimensions, which precisely cuts a blank of a solid piece of metal or fully fused dental porcelain, on the basis of the digital data. The obtained core is then all-ceramic crowned with the appropriated low fusing porcelain.

Strengthening mechanisms of dental ceramics

Progress in dental ceramics is limited by the inherent problems of clinical dentistry: space, esthetics and occlusal forces. The human tooth is a very translucent object covered with a thin layer of enamel approximately 1-

2mm thick (Nanci, 2003). The inner core of dentin nourished by the pulp is more flexible and supports the brittle enamel. Dentin consists of approximately 70% hydroxyapatite crystals bonded in a collagen matrix (Goldberg *et al.*, 2003). Human enamel may transmit up to 70% light on a 1mm-thick specimen, whereas dentin is more opaque and varies between 20 to 40% light transmission, depending upon the age of the tooth (McLean, 1979).

In order to duplicate this translucency, dental porcelains must contain a high proportion of glassy material. Ceramics are harder than human enamel and may cause extensive wear during chewing (Metzler *et al*., 1999; Derand *et al*., 1999). However, they are comparatively weak materials when compared with gold alloys used in dentistry (Al-Hiyasat *et al*., 1998; Ramp *et al*., 1997).

Metals possess high fracture toughness and are not as dependent on surface condition as ceramics. Indeed, many dental researchers are now realizing that the integrity of the surface of ceramic restorations plays a major role in the longevity of the restoration, and that a high-strength ceramic with a badly flawed surface may perform worse in a clinical situation than a weaker ceramic with comparatively flaw-free surface (de Jager *et al*., 2000; Albakry *et al*., 2004; Guazzato *et al*., 2004; Guazzato *et al*., 2005). For this reason, the margin of safety required in ceramics is always greater than that in metals, particularly when variables in dental technology are taken into account.

Dental porcelains are brittle, vitreous ceramics with high glass content that fail under tensile or bending stresses because of the presence of flaws (cracks or pores) in the material, which are created during fabrication (O'Brien, 2002). Tensile or bending stresses open and widen the flaws in the material and cause failure; compressive stresses close cracks. A pane of glass placed on a very flat floor can be walked on without damage, but if the glass is picked up and bent, any existing flaws will enlarge and cause the glass to break.

Brittle materials have a related weakness to mechanical shock – the stresses from dropping or applying pressure to porcelain will cause any existing flaws to widen and fracture. These flaws can be reduced in the fabrication process, and the modern high-strength glass fibres are made with fewer surface flaws and very high tensile strengths.

A number of methods for strengthening ceramic restorations have been applied in the dental field. What follows is a description of the different methods used until 2010.

Enameling of metals

The most predominant method of strengthening dental porcelain restorations involves enameling of metals. Weinstein and co-workers were the first to describe commercially the production of metal-ceramic restorations using porcelain powders containing 11-15% K_2O frits (Weinstein M, Katz S, Weinstein AB. Fused porcelain-to-metal. US Patent 3,052,982.1962).

Glasses in the $Na_2OK_2OAl_2O_3SiO_2$ system containing not less than 11% K_2O when subjected to heat treatments at room temperatures from 700-1200°C produced expansion glasses suitable for bonding to metal at 13-15 x10^{-6}/°C. The required thermal expansion resulted from the crystallization of leucite. The proportion of leucite is governed by chemical composition, firing temperature and time of heat treatment. The basic change required to produce porcelain of thermal expansion necessary for metal bonding is to increase the K_2O content to the required level (McLean, 1991).

To achieve a strong bond to gold or palladium alloys, certain conditions must be fulfilled. The glass must wet the metal and the stresses resulting from thermal expansion and contraction must not exceed the tensile strength of the glass (Craig, 2006). Alloys used for attachment to porcelain must have high temperature strength and produce thin films of oxide for porcelain bonding

(Jochen *et al*., 1986). Dental porcelain will wet and adhere to any clean, gas free metal, provided that the metal is covered with an adherent layer of oxide, and the temperature is raised to the point where this oxide partially dissolves into glass (McLean, 1991). Excessive oxide production can produce weak bonding, as sometimes occurs with nickel-chromium alloys (Lubovich *et al*., 1977; Mackert *et al*., 1984).

High gold alloys containing around 80% pure gold have been used for many years, but lately less expensive alloys have been produced that are proving clinically successful (McLean, 1988). A quiet revolution has occurred in the production of high palladium alloys that have been in use for over 30 years. The reduction in cost of these alloys and resistance to creep at high temperature has lent an even more competitive edge to the metal-ceramic restoration, which is still regarded as the strongest and most durable ceramic restoration available today.

Bonding porcelain to a metal substructure prevents the propagation of cracks and flaws in two ways. In the first, the metal strengthens the under layer where tensile stresses from mastication forces can be the highest; a high tensile metal coping resists the stresses without failing. The second mechanism involves protective residual compressive stresses produced in the porcelain from a slight mismatch in the thermal expansion coefficients of the alloy and porcelain. An alloy will produce protective compressive stress if it has a slightly higher thermal expansion coefficient than the porcelain; this effect is formed after firing the porcelain when the alloy substructure tries to contract more than the porcelain layer, as the restoration cools. A porcelain with a higher thermal expansion coefficient than the alloy would produce tensile stresses at the interface that weaken the porcelain. For the strengthening mechanism to be successful, the porcelain needs to chemically bond to the alloy substructure and form a hybrid layer of porcelain and oxides on its surface (Mackert *et al*., 1988). Coating the alloy with pure gold for esthetics before adding the porcelain will prevent the bond from forming and will result in a weaker crown and possible failure.

Metal-ceramic crowns have been widely accepted as the treatment of choice for single or fixed partial denture restorations because of the advantages of high strength, reasonable esthetics, and long-term predictability (Reitemeier *et al*., 2006; Näpänkangas *et al*., 2008). Its only disadvantage lies in the use of metal copings to reinforce the ceramic. The metal requires masking with highly opaque porcelains and as a result the natural translucency of human teeth is lost.

The translucency of metal ceramic crowns is often affected by the metal coping, which restricts the transmission of light through the restoration and may increase light reflectivity of the crown (Chiche *et al*., 1994). Two problems that are commonly encountered in metal ceramic crowns are (1) the high value (brightness) and excessive opacity at the cervical one-third and (2) dark overlying gingival tissues.

The metal copings of these crowns completely mask the color of underlying structures, and the opacity of metal coping does not allow light transmission through the restoration and the underlying tooth structure (McInnes-Lesoux *et al*., 1987). Moreover, even when placed subgingivally, a dull grayish background may give the soft tissue an unnatural bluish appearance. This darker coloring effect may entail substantial esthetic impairment and contribute to an unsatisfactory treatment outcome.

Effective masking of the color of underlying metal structure can be achieved by increasing the amount of opaque pigments in the porcelain and the thickness of the coping veneering material (Yaman *et al*., 1997). Increasing the thickness of the restoration may create clinical problems. If the restoration is overcontoured food remnants may easily be entrapped around the cervical area of the restoration and become difficult to be cleaned out. This may cause periodontal disease resulting in gingival inflammation and destruction of soft and hard tissues (Parkinson, 1976). Also, overcontoured restorations cannot create the natural look of teeth regarding to such factors affecting final appearance such as emergence profiles, line angles, or embrasures (Chiche *et al*., 1994). Addition of more opaque porcelain pigments has been reported to have a positive effect on final color of the restorations (Yaman *et al*., 1997; Davis *et al*., 1992). Typically 5% to 15% opaque porcelain is required to achieve optimal masking. Exceeding 15% opaque porcelain dramatically reduces light penetration and results in a significant loss of natural appearance of restoration (Sturdevant *et al*., 1995).

The driving force for the developments in the dental ceramic field has been the immense difference in reliability between metal-ceramic systems and all-ceramic systems and a public perception that metal-free restorations are more esthetic. The disadvantages of the metal-ceramic systems include radiopacity, some questions centering on metal biocompatibility and lack of natural esthetics; important features in today's consumer conscious dental market. It is hardly surprising that research has therefore concentrated on producing high-strength ceramic restorations that allow similar light transmission as that of the human tooth.

Crystallization of glasses

Glass is an inorganic material comparable to enamel in hardness (Seghi *et al.*, 1991). Because molten glass flows easily, it can be cast to any desire shape and the casting accuracy is usually very good. However, glass is weak and brittle due to the surface and internal imperfections. If fine-grained crystals with comparable thermal expansion coefficients could be incorporated into the glass matrix by powder sintering or internal crystallization, the strength of the material could be greatly enhanced (Olcott, 1963).

In order to develop this idea, Stookey in 1952 at the Corning Glass

Works (Stookey, 1958) introduce the crystallization of glass. Controlled crystallization of glass depended upon the fact that glass, at ordinary temperature, is a super cooled liquid, which does not crystallize on cooling. It can be made to crystallize by reaching to suitable temperature with crystal seeds or nuclei present. The glass is then converted to a dense mass of very tiny interlocking crystals. This original ideal was the first step towards the introduction of glass ceramics. Glass ceramics have an intermediate position between glasses and ceramics: glass ceramics are melted and formed as a glass but after forming they are subjected to an additional heat treatment resulting in a material composed of uniformly distributed crystals embedded in a glassy matrix. Many properties of glass ceramics are mainly determined by those of the crystals because these usually occupy a predominant volume fraction. Thus, glass ceramics frequently resemble ceramics of similar composition more closely than they resemble chemically identical base glasses. Strengthening by crystalline reinforcement involves the introduction of a high proportion of the crystalline phase into the ceramic to improve its resistance to crack propagation. However, the crystalline phase must be carefully selected. Important selection criteria include the coefficient of thermal contraction, toughness, and the modulus of elasticity. Different crystal seeds or nuclei are used to fabricate glass ceramics. What follows is a description of different systems used on the fabrication of dental glass ceramics.

Lithium zinc silicate glass ceramic (LZS) - Li_2O-ZnO-SiO_2 system (Lithia-based)

MacCulloch was the first researcher to recognize the potential use of glass-ceramics in dentistry (Grossman, 1985). He found that Li_2O-ZnO-SiO_2 glass based ceramics showed a translucency similar to the natural tooth and was stronger than feldspathic porcelain. The use of compact white glass ceramics of the Li_2O-ZnO-SiO_2 system, in the field of dentistry, was first applied for the manufacture of artificial ceramic denture teeth. In his paper, MacCulloch also suggested that it should be possible to manufacture crowns and inlays using centrifugal casting method (MacCulloch, 1968). However, he was not able to develop such a technique to a practical stage.

On the basis of such discovery was the fact that normal glasses do not show volume crystallization on heat treatment. Volume crystallization is induced in glass ceramics by small additions of nucleating agents has previously mentioned; components that readily dissolve in the glass melt but tend to segregate at lower temperatures thus forming nuclei for the crystallization of the major crystal phases. The number of nuclei per unit volume may be so high that the average size of the crystals growing on them remains well below the wavelength of visible light and thus, transparent glass ceramics may be formed. MacCulloch used continuous glass molding procedures to form ceramic denture teeth.

Lithium disilicate glass ceramic (LDS) - Li_2O-SiO_2 system

(Lithia-based)

Glass ceramics are polycrystalline materials prepared by controlled crystallization of the crystal phase in glasses. By regulating the temperature in a controlled manner well-shaped crystal growth occurs which is basically random in nature. A simple composition of 20% (wt) Li_2O and 80% (wt) SiO_2 was investigated as a glass-ceramic for use in dental application by Hench in 1971 (Hench, 1971). The main crystal phase produced in the ceramic matrix was lithium disilicate (Li_2O-$2SiO_2$). The diametrical tensile strength of this glass ceramic was reported to be between 15.000-18.000 psi, compared to fused conventional feldspathic dental porcelain of 9.000 psi. However, the presence of small cracks surrounding the crystals, arising from a local volume change during thermal crystallization lowered the strength of the material, and the glass ceramic produced by this system was abandoned (Grossman, 1988). Only 30 years later was that system re-utilized, and it is now one of the most prominent types of ceramic material.

Lithium disilicate glass ceramic (LACS) - Li_2O-Al_2O_3-CaO-SiO_2 system (Lithia-based)

Further improvements to lithium disilicate based glass ceramics were performed through the addition of CaO and Al_2O_3 (Hench, 1973). Glass-ceramics can be obtained from a wide variety of compositions, leading to a wide range of mechanical and optical properties, depending on the nature of the crystalline phase nucleating and growing within the glass. Experimental glass-ceramics in the system Li_2O-Al_2O_3-CaO-SiO_2 were the object of extensive research work. The choice of adequate additives was critical in the development of tougher and higher-strength glass-ceramics (Anusavice *et al*., 1994). Differential thermal analysis could be efficiently used to determine the heat treatment leading to the maximum lithium disilicate crystal population in the shortest amount of time, thereby optimizing the nucleation and crystallization heat treatment of this type of glass-ceramic. These additions substantially improved both the castability and the chemical durability of the glass ceramics. A further improvement in the mechanical properties was found by incorporating the combined nucleating agents resulting in a fine-grained crystalline phase uniformly dispersed within the glass matrix (Ritter *et al*., 1979).

Calcium phosphate glass ceramic (CP) - $CaO-P_2O_5$ system

(Phosphate-based)

Different materials were tested as glass ceramics in the following years for dental applications. One of these was the calcium phosphate material in light of its' characteristics. As with natural teeth, these materials are composed primarily of phosphorus and calcium, and are high in affinity with gingival tissue. Accordingly, they could be cast by the lost wax process. The preparation of calcium phosphate based glass-ceramic crowns by lost-wax technique was first described by Abe and co-workers (Abe *et al*., 1975).

The disadvantages of such calcium phosphate glass-ceramics was that they had been regarded to have drawbacks such a low degree of strength and a tendency of easily breaking (brittleness). With the intent of improving the strength and toughness of the calcium phosphate glass ceramics, compounds of rare earth elements were added into the glasses prior to crystallization. For dental materials the elements added were one type, or two or more types of compounds selected from oxides of iron, manganese, cerium, titanium, nickel, zinc, cobalt, tungsten, chromium, and vanadium as color component. Also, if necessary, Al_2O_3 and/or SiO_2 were contained as shading assistant agent in that composite. The rare earth oxide, shading component, and shading assistant agent added were incorporated in crystals of calcium phosphate.

Kihara and co-workers reported this new system of crystallization of glasses to form the $CaO-P_2O_5$ glass ceramic (Kihara *et al*., 1984). The flexural strength of this glass ceramic was reported as 17.000 psi (116Mpa) and the glass was cast at 1050°C in a gypsum-bonded investment mold. The fabrication of the ceramic by this procedure was by heat-treating the clear glass at 645°C for 12 hours. This glass ceramic material had a very close match in hardness to the natural tooth enamel as measured by Vickers Microhardness Test (Fukui *et al*., 1977).

The added rare earth compounds were dispersed by becoming oxides during the vitrification of calcium phosphate composites, and when the calcium phosphate glasses were crystallized, those rare earth oxides acted to accelerate the formation of a large number of crystal nuclei in the interface with the glasses, while inhibiting the growth of crystal grains. As a result, the calcium phosphate glass was crystallized into aggregates of fine crystal grains. In this manner, the strength and the toughness were improved markedly. They were added in the form of oxides, and also of carbonates, hydroxides, and nitrates. The rare earths were used by mixing one type or two or more types of them, and it was desirable that yttrium (Y), lanthanum (La), or cerium (Ce) was contained in the rare earths to be mixed.

Tetrasilicic fluormica glass ceramic (TSFM) - K-Mg_{25}-Si_4O_{10}-F_2 system (Mica-based)

As described earlier, glass-ceramics are obtained by controlled devitrification of glasses with a suitable composition including nucleating agents. Depending on the composition of the glass, various crystalline phases can nucleate and grow within the glass. The advantage of this process is that the dental restorations may be cast by means of the lost-wax technique, thus increasing the homogeneity of the final product compared with conventional sintered feldspathic porcelains. The development of glass ceramics by the Corning Glass Works (Corning, NY, USA) in the late 1950's has led to the creation of a dental ceramic system based on the strengthening of glass with various forms of mica. However, it was only in 1983 that this development finally resulted and a crown was marked under the trade name Dicor® (Dentsply International, Inc., York, PA, USA), based on the work of Grossman and Adair (Adair, 1984; Grossman, 1987). The ceramic material contains tetrasilicic fluromica crystals ($KMg_{25}Si_4O_{10}F_2$) and small amounts of Al_2O_3 and

ZrO_2, which because of its flexibility and plate-like morphology adds strength and resistance to fracture propagation. The tetrasilicic mica system nucleates readily at a temperature of 650°C to 1075°C: The residual glass phase occupies approximately 45% (by volume) of the glass-ceramic. The casting is then heat-treated or "cerammed", during which tetrasilicic fluromica crystals are formed to increase the strength and toughness of the glass ceramic.

Dicor® is a mica-based machinable glass-ceramic. The machinability of Dicor® glass-ceramic is made possible by the presence of a tetrasilicic fluromica ($KMg_{25}Si_4O_{10}F_2$) as the major crystalline phase (Grossman, 1987). Micas are classified as layer-type silicates. Cleavage planes are situated along the layers, and this specific crystal structure dictates the mechanical properties of the mineral itself. Crack propagation is not likely to occur across the mica crystals and is more probable along the cleavage planes of these layered silicates. In the glass-ceramic material, the mica crystals are usually highly interlocked within the glassy matrix, achieving a "house of cards" microstructure (Grossman, 1987). The interlocking of the crystals is a key factor in the fracture resistance of the glass-ceramic, and their random orientation makes fracture propagation equally difficult in all directions. After being cast, the Dicor® glass is converted into a glass-ceramic by means of a single-step heat treatment with a six-hour dwell at 1070°C. This treatment facilitates controlled nucleation and growth of the mica crystals. However, it is critical to re-invest the cast glass restoration prior to the crystallization heat treatment, to prevent sagging or rounding of the edges at high temperature. The match in the thermal expansion coefficients of the glass and the investment is achieved by use of a leucite-based gypsum-bonded investment. The interaction of the glass-ceramic and the investment during the crystallization heat treatment leads to the formation of calcium magnesium silicate at the surface of the glass-ceramic (Denry *et al*., 1993). This crystalline phase could be formed by decomposition of the mica into magnesium silicate that later reacts with the gypsum-bonded investment. This surface layer, called the "ceram layer", has been reported to decrease the strength of glass-ceramic crowns significantly (Campbell *et al*., 1989; Kelly et al, 1989). The effects of alumina and Zirconia additions on the bending strength of Dicor® glass-ceramic have been investigated. It was found that alumina additions successfully increase the bending strength of Dicor® glass-ceramic, whereas Zirconia additions had no effect (Denry *et al*., 1996)

The method of construction of the Dicor® crown was, and still is, very appealing, since like all glass-ceramics, it may be cast by lost-wax technique process. After fusing the resulting ceramic crown was semi-crystalline but still translucent like human enamel, so that it could be colored by surface staining or light glazing or veneered with aluminous porcelain.

Unfortunately, the strength of Dicor® was no greater than aluminous porcelain with values of 120MPa which contra-indicated its use on molar areas or to fabricate fixed partial dentures (Grossman *et al*., 1987; Malament *et al*., 1999). However, when used on anterior teeth, Dicor® had some distinct esthetic advantages. Because of high translucency, it had a chameleon-like effect and merge with the surrounding teeth. Dicor® was particularly useful for matching the adolescent tooth (Malament *et al*., 1990).

In order to increase the range of applications, the Dicor® Plus crown was launched in 1991 (McLean, 1991). The material was used as a cast coping that was stratified with matched expansion feldspathic porcelain of the aluminous type. This second dental version was developed for CAD/CAM dental procedures. This cerammed glass was provided in an already heat-treated state from the manufacturer. In this latter technique an optical scan of a prepared tooth is loaded into a computer and a milling system was used to produce the restoration. The restoration was then “bonded” to the remaining tooth structure using a dental based composite resin. This technique offered the opportunity of building porcelain color in depth and utilizing high translucency of the coping to reduce shadowing at the critical cervical margin (McLean, 1991). However, the design of the coping influenced the fit. When wall thickness of the coping was reduced to bellow 0.5mm, there was a danger of pyroplastic flow causing distortion during the fitting of the all-ceramic crown porcelain. Also, on cooling, cracking of the glass all-ceramic crown could occur.

The flexural strength of the Dicor® Plus crown was about 22.000psi (152MPa) which would not appear to be high enough for use in posterior teeth (Duffin *et al*., 1989; Grossman, 1985). Although the Dicor® crown showed low resistance to fracture, the use of an acid-etch procedure along the inner surface, and the use of resin cements increased the resistance which was reported to be low with the zinc phosphate cement (Malament *et al*., 1992).

Apatite glass ceramic (HMPO) - CaO-MgO-P_2O_5-SiO_2 system

(Hydroxyapatite-based)

The formulation of a hydroxyapatite ceramic through reaction of glass ceramics with moisture was described by Hobo and co-workers (Hobo *et al*., 1985). This material, designated commercially as CeraPearl® (Kyocera Corp, San Diego, CA, USA), is an apatite ceramic classified as a CaO-MgO-P_2O_5-SiO_2, that when reheated becomes crystalline oxyapatite ($Ca_{10}(PO_4)6O$). This structure when exposed to moisture becomes the crystalline hydroxyapatite (Hobo *et al*., 1985).

CeraPearl® crown was fabricated in a manner similar to the Dicor® ceramic crown. Crystallization resulted in a crown that was highly dense, strong and chemically stable. The hardness and thermal conductivity were similar to that of human enamel and the material was reported as having excellent biocompatibility (Savil, 1987). As with similar ceramics, the final shading was accomplished by external surface staining. There are no long-term published studies concerning the performance of this system clinically, and also, no reliable testing of the strength of the material.

Chemical strengthening (ion exchanging)

An effective method of strengthening glass-like materials, such as dental porcelain, is to produce compressive stresses in their surfaces (Olcott, 1963; Stookey, 1965). This may be achieved chemically by modifying the atomic structure of the surface regions of glass by ion exchange (Southan, 1970). Ion exchange yields a “crowding” or stress in the surface, produced by larger ions taking the place of smaller ions, that is not relieved, and permanent compression in the surface remains. The principle of chemical strengthening relies on the exchange of small alkali ions for larger ions below the strain point of the ceramic. Since stress relaxation is not possible in this temperature range, the exchange leads to the creation of a compressive layer at the surface of the ceramic (Denry *et al*., 1996). Finally, any applied load must first overcome this built-in compression layer before the surface can be placed into tension, resulting in an increase in fracture resistance. This technique involves the use of alkali salts with a melting point lower than the glass transition temperature of the ceramic material. Ion exchange strengthening has been reported to increase the flexural strength of feldspathic dental porcelain up to 80%, depending on the ionic species involved and the composition of the porcelain (Anusavice *et al*., 1992; Anusavice *et al*., 1992; Seghi *et al*., 1992; Fischer *et al*., 2001; Fischer *et al*., 2005). The depth of the ion-exchanged layer has been shown to be greater than 50 micrometers (Anusavice *et al*., 1994). However, this technique is diffusion-driven, and its kinetics is limited by factors such as time, temperature, and ionic radius of the exchanged ions.

Ohno and co-workers in 1985 (Ohno *et al*., 1985) introduced a simple method by which dental porcelains can be strengthened by an ion exchange slurry while maintaining the translucency and color tone of the porcelain, without the use of any specialized equipment. Araki in 1989 (Araki *et al*., 1989), demonstrated an increase of 20% in three-point bending strength and a 23% increase in the Knoop hardness of a feldspathic porcelain after a 30-minute firing at 400°C of an ion exchange agent consisting of 35.0 wt % K_2HPO_4, 15.0wt % SiO_2, 49.5 wt% distilled water and 0.5 wt% of a viscosity controlling agent.

In 1990, an ion exchange agent called GC Tuf-Coat (GC Int. Corp., Tokyo, Japan) was introduced for strengthening feldspathic porcelain via a Na+ - K+ exchange process. This material is believed to be similar in composition to the one used by Araki. A study investigated the effect of this agent on seven different feldspathic porcelains and reported an increase in three-point bending strength from 20% to 83% (Seghi *et al*., 1990). A different study found, by means of a ball-on-ring flexure test, that the same ion-exchange agent was more effective in strengthening aluminous core porcelain than dentin porcelain (Piddock *et al*., 1991). This study also reported that the depth of ion exchange could be as much as 100μm and that the steepest gradient developed over the first 10μm.

Thermal tempering (physical toughening)

Thermal tempering is commonly used to strengthen glasses and is based on the creation of temperature gradients between the surface and the bulk part of the glass piece. This method of strengthening glasses, and consequently dental porcelain, can be achieved physically, by thermally quenching the glass object just bellow its softening temperature (DeHoff *et al*., 1989; DeHoff *et al*., 1990). The technique involves heating of the glass to a temperature above the glass transition region and below the softening point. It is then cooled to room temperature in a jet of air or, in some cases, in an oil bath. The residual stresses arise from differences in cooling rates for surface and interior regions. The result is similar to that obtained with chemical strengthening with the formation of a surface compressive layer that results in increased strength. This technique has been successfully applied to feldspathic dental porcelain and resulted in mean flexure strength values 2.6 times greater than the corresponding value for slow-cooled specimens (Anusavice *et al*., 1991). The stress relaxation behavior of dental porcelain when reheated can be characterized by stress relaxation testing under compression at high temperature or by acoustic emission techniques. The principal effect of tempering is the inhibition of crack formation rather than the retardation of crack growth (Anusavice *et al*., 1991; Hojjatie *et al*., 1993). However, the combination of thermal tempering and subsequent ion exchange does not lead to a significant increase in the mean biaxial flexural strength values (Anusavice *et al*., 1992). This study also showed that tempering treatment was more effective in strengthening porcelain than was the ion exchange process as measured by the biaxial flexural strength test. However, the results of initial crack size induced by the microhardness tester showed that ion exchange yielded a surface that was more resistant to crack initiation than that yielded by the tempering treatment. Furthermore, the study showed that there was evidence of exchange between Na^+ within the porcelain surface and K^+ from the ion exchange agent applied on the surface.

Anusavice and co-workers have shown that tempering of feldspathic porcelain by forced convective cooling in air reduces the size of cracks induced within the surface by a microhardness indenter, and that the compressive stress induced by tempering was approximately 78MPa (Anusavice *et al*., 1991).

Bonding to foils

Bonding of the porcelain to metal foils can strengthen feldspathic dental porcelain. This procedure appears to eliminate open surface defects from which tensile failure may originate (Sced *et al*., 1977). A platinum twin foil technique was advocated by McLean and Sced in 1976 for this purpose (McLean *et al*., 1976). This method could increase the shell strength of the core porcelain to over 300 MPa, providing the platinum foil was left intact with the porcelain (McLean, 1991). When the platinum foil was removed, strength fell to approximately 180Mpa (Piddock *et al*., 1984).

In 1979, Rogers reported a rather ingenious method of making metal copings by electroforming (Rogers, 1979). He used a tin oxide coating to attach the porcelain to the gold coping, and conventional feldspathic metal-bonding porcelain was used as the all-ceramic crown.

In 1981, Hopkins introduced a new method called the gold-coated platinum foil porcelain crown (Hopkins, 1981). In this technique, porcelain formulated for the metal-ceramic technique could be built over platinum matrix coated with a thin layer of gold. A 75% increase in tensile strength had been claimed (Hopkins, 1981).

Schoessow, in 1983 presented a method of bonding feldspathic porcelain to a special noble metal foil in fabrication of metal-ceramic crown (Schoessow, 1983). This system was marketed in the U.S. under the name Renaissance crown system® (Williams Gold Refining CO, NY, USA) and in Europe as Ceplatec crown system® (Ceplatec, Krefeld, Germany). The metal foil was supplied in umbrella-shaped sheets with eight pleats and consists of seven layers, the two outermost layers were 100% gold. Next two layers were ceramic gold alloy composed of 85% gold, 5% platinum and 7% palladium. The next two layers were composed of pure palladium and the inner layer was a ceramic gold alloy composed of 85% gold, 5% platinum and 6% palladium giving a total thickness of 51-70μm (Scharer *et al*., 1987).

The pleated metal foil was adapted to the die by means of crimping, burnishing and swaging. After the foil was adapted to the die, it was heat treated, thereby forming a gold ceramic alloy and making the form rigid. An interfacial alloy was applied to the surface of all-ceramic crowns. The matted surface of interfacial alloy after fusion at 1000°C provided mechanical retention to the all-ceramic crown porcelain. The compressive strength of the cemented Renaissance crowns was evaluated by different studies (Burkl *et al*., 1987; Vrijhoef *et al*., 1988). They found that the Renaissance crowns were weaker than the metal-ceramic crowns, Cerestore crowns and aluminous porcelain jacket crowns. However, the fracture strength of the Renaissance crowns was higher than the expected average chewing forces (Haraldson *et al*., 1989).

A different bonding foil technique was the Sunrise system (Tanaka Enterprises, CA, USA) that was a gold-foil-supported ceramic system that used Ceramco II porcelain (Dentsply International, Inc., York, PA, USA) (Gregory *et al*., 1992). Its foil compressing system could be used to adapt platinum foils as well as the system's gold foil. This technique offered a yellow background from which to work, but no core porcelain was recommended. The strength of the crowns made with this system is unproven and questionable.

Bulk strengthening (compositional strengthening)

Another method to improve mechanical properties of dental porcelain is bulk strengthening. This means producing a porcelain with a uniform, increase in strength. Dental porcelains are basically aluminosilicate with residual quartz crystals and a small percentage of pigment agents. In a composite system with a continuous glass matrix, crystalline dispersions limit the size of Griffith flaws and strengthen the system. For crystals to act as effective strengthening agents, however, they should possess a thermal expansion similar to the matrix phase to prevent the introducing of stress at the matrix-crystal interface (Backer *et al*., 1993). McLean and Hughes reported on replacing the quartz crystalline phase with alumina and observed a significant improvement in strength (McLean *et al*., 1965). This resulted in part from the lower expansion of alumina particles that enable them to remain in intimate contact with the glass matrix. Southan has classified modern dental porcelains containing quartz filler particles into two groups: one group having an intact vitreous matrix and the other having a disrupted vitreous matrix (Southan, 1970). The first group includes aluminous porcelains and any feldspathic porcelain having matrices that are sufficiently plastic at 573°C to accommodate the volumetric shrinkage of the quartz inversion. The second group includes feldspathic porcelains having matrices that are rigid at 573°C. When porcelains in this group are cooled, the glass matrix is stressed to the point where microcracks are formed. Southan´s data indicated that aluminous porcelains were not significantly stronger than matrix intact feldspathic porcelains. The implication of this concept is that strengthening may be achieved with quartz filler particles if the viscosity or rigidity of the glass matrix is such that the volumetric contraction of the quartz particles does not produce stresses at the quartz-glass interface.

Although the presence of crystals, whether quartz, alumina or leucite can strengthen a dental porcelain under proper conditions, fracture may also propagate through the glass in the regions between crystalline particles. Therefore, an inquiry into methods of strengthening the glass matrix is warranted (Backer, 1993).

Dispersion strengthening

Glassy materials such as dental porcelain may be strengthened by dispersing ceramic crystals of high strength and elasticity in the glass matrix. If the glass has a similar thermal expansion to the crystals, the strength and elasticity of crystal-glass composite materials may increase progressively with the proportion of the crystalline phase. A dispersed material with a higher thermal expansion coefficient will produce greater strengthening because a compressive stress is formed at the dispersed phase interface. For example, compare leucite, a potassium-alumina-silicate in a glass matrix that has a high thermal expansion coefficient of 17ppm/°C, to a glass with a coefficient of 10ppm/°C. This was previously discussed in the section on the strengthening mechanisms of ceramic crystallization of glasses. Several all-ceramic dental porcelain systems use nowadays dispersion strengthening without relying on a metal substructure. The most common crystals used are leucite, alumina, magnesia, lithium disilicate, and Zirconia. There are several techniques and methods in fabricating crystals-reinforced porcelain crowns that utilize the principle of dispersion strengthening. What follows is the description of this methods and/or respective all-ceramic systems.

Leucite-strengthened ceramics

Leucite has been widely used as a constituent of dental ceramics to modify the coefficient of thermal expansion since 1950's. The addition of leucite (crystals of a potassium-alumina-silica complex) increased the strength of porcelain by limiting crack propagation. Unfortunately, the use of ceramics restorations with high-leucite content increased the potential for wear of opposing teeth and also reduced opalescence.

In order to solve this problem, a new wave of ceramics appeared since the 1980´s aiming to improve the esthetics and address one of the most important drawbacks of porcelains - the potential for catastrophic failure. The introduction of pressed leucite reinforced ceramic systems, had leucite in a different role. In these systems the ceramic material relies on an increased volume of fine leucite particles to increase flexural strength of the dental porcelain (Dong *et al*., 1992).

Historically, a patented heat-press technique was first described in 1936 for the construction of ceramic complete dentures (Dong *et al*., 1992). In 1969, Dröge described a ceramic press technique based on the hot-press resin technique (Dröge, 1969). In improving Dröge´s technique, McPhee was able to produce complete-coverage metal-ceramic restorations that accurately duplicated occlusal surfaces (McPhee *et al*., 1977). A heat-press technique was introduced in 1983 at the University of Zurich. The development proceeded in conjunction with the Ivoclar Company, which in 1991, introduced the IPS Empress® system (Ivoclar, Schaan, Liechtenstein) an injection molded glass ceramic.

The IPS Empress® system caused a major revival of the all-ceramic crown concept. All-ceramic crowns had been somewhat dormant in the profession for a period of years after significant failure of some previously used crown types. The IPS Empress® system is a highly esthetic hot pressed glass ceramic material for fabrication of ceramic restorations. Similar versions using finely dispersed leucite grains to increase toughness, strength and modify wear patterns and rates were then developed and are now available.

IPS-Empress® 1

IPS-Empress® 1 is a fine-grained high strength pressed ceramic material which is leucite reinforced. The heat-pressed technique has been described to construct single unit crowns, inlay/onlays and veneers using precerammed and precoloured glass-ceramic ingots. The crowns produced by this system contain around 40 to 50% leucite dispersed in a lower expansion glass. The leucite improves the strength and fracture resistance of the feldspathic glass matrix. Leucite crystals are formed through various temperature cycles. The technique requires that restorations be waxed to full contour and invested in a special flask with a correspondent developed investment. The invested pattern is placed into the Empress furnace for burnout at 800°C. The selected shade ingot and an aluminum oxide plunger are placed in a cylindrical opening at the investment ring and heated to 1100°C so that the leucite-reinforced ceramic becomes plasticized. The ingot is pressed into the investment mold where it is held under pressure to allow for complete and accurate adaptation. The casting procedure is fully automated. After divesting the restoration, there are two ways to reproduce the appropriate shade: (1) surface characterization: a heavily pigmented characterization color in the form of superficial stains is applied and then covered with translucent, extremely fine glazing material to a thickness of 50-60μm; (2) all-ceramic crowning technique: the restoration is reduced by grinding to allow space for placement of a selected shade all-ceramic specially formulated fired ceramic that will create excellent anatomy and a realistic, translucent esthetic result. This technique is indicated to achieve superior esthetics in anterior crowns. Lüthy and co-workers demonstrated that there is no difference in tensional strength between these two methods (Lüthy *et al*., 1993). Internal crown surfaces need to be roughened by etching with hydrofluoric, silaned and bonded to tooth structure using standard dentin and enamel bonding techniques and resin cement in order to obtain consistent bond strength (Ayad *et al*., 2008). IPS Empress® restorations are very translucent and flexural strengths of 140 MPa to 180 MPa have been reported (Dong *et al*., 1992). These materials are being used successfully as veneers and anterior full crowns (Fradeani *et al*., 2002; Fradeani *et al*., 2005).

Posterior full crowns are serving with acceptability, but more research is necessary (Fradeani *et al*., 2002).

Optimal Pressable Ceramic®

The popularity of Empress has stimulated other companies to produce similar products. The OPC® (Optimal Pressable Ceramic®) (Jeneric/Pentron Inc., CT, USA) restorations were virtually fabricated in the same manner as the IPS Empress® 1 crown. The only differences between the two technology systems were the price and the ceramic ingots supplied to fabricate the ceramic restorations. The strength reported for the OPC® crown was similar to that of IPS Empress®1 around 140Mpa to 180Mpa (Sobrinho *et al*., 1998; Cattel *et al*., 1999). The ceramic was used for anterior crowns, veneers, inlays, onlays, and posterior regions with low pressure. No long-term data is available for this crown system and the product is no longer available.

Optec HSP

Optec HSP material (Jeneric/Pentron Inc., CT, USA) was a leucite reinforced feldspar ceramic that condensed like alumina ceramic and was sintered like traditional feldspar ceramic. The manufacturing process was done on refractive dies, differently from the OPC system. Because of the moderately opaque core, this ceramic was more transparent than crowns made on aluminum oxide cores or with glass/aluminum oxide cores. Optec HSP material was a feldspathic porcelain containing up to 45 vol% tetragonal leucite (Denry *et al*., 1995). The greater leucite content of Optec HSP porcelain compared with conventional feldspathic porcelain for metal-ceramics lead to a higher modulus of rupture and compressive strength. The large amount of leucite in the material contributed to a high thermal contraction coefficient (Katz, 1989). In addition, the large thermal contraction mismatch between leucite (22 to 25 x 10^{-6}/°C) and the glassy matrix (8 x 10^{-6}/°C) resulted in the development of tangential compressive stresses in the glass around the leucite crystals when cooled. These stresses acted as crack deflectors and contributed to increase the resistance of the weaker glassy phase to crack propagation. After heat treatment of Optec HSP for one hour at temperatures ranging from 705 to 980°C, a second metastable phase identified as sanidine ($KAlSi_3O_8$) formed at the expense of the glassy matrix. The crystallization of sanidine was associated with a modification of the optical properties of the material from translucent to opaque. However, sanidine did not appear when the porcelain was heated to 980°C, since sanidine is metastable in the temperature range 500-925°C. The precipitation of sanidine has been reported as well upon isothermal heat treatment of conventional feldspathic porcelain for metal-ceramics (Mackert, 1988).

Optec HSP contained a higher concentration of leucite crystals than feldspar ceramics. Due to the opacity caused by leucite crystals, it was not necessary to apply core ceramics. The outer layer consisted of conventional ceramics, so that this layer showed a more inferior fracture resistance than the leucite reinforced ceramic core. The flexural strength reported by manufacturer was approximately 172 MPa. The flexural strength reported by Seghi and co-workers was only 105 MPa (Seghi *et al*., 1995). This system was used in the fabrication of inlays, onlays, and all-ceramic crowns for anterior teeth. No independent long-term clinical data are available for these restorations. The only study available for crown evaluation at 5 years revealed satisfactory results (Hankinson *et al*., 1994). However, because of commercial problems the product is no longer available.

Alumina-strengthened ceramics

Alumina is the oxide of aluminum commonly extracted from the mineral bauxite that is mainly a hydrated aluminum oxide. To produce the alumina bauxite is finely ground and digested in hot caustic soda solution to dissolve the Gibbsite (aluminum tri-hydroxide or "hydrate"). Digestion of Gibbsite is achieved at a comparatively lower temperature and pressure compared to other aluminum materials. After digestion, insoluble bauxite residue is removed from the hydrate rich solution by settling. The clarified hydrate-rich solution is cooled, seeded and cooled again in stages to precipitate the alumina trihydrate crystals that are separated by filtration and then washed to remove the final traces of caustic soda and other impurities. The alumina trihydrate is converted to aluminum oxide by calcinations usually in a rotary kiln at a temperature of 600°C that drives off the chemically combined water in the hydrate to form gamma-aluminum oxide.

For ceramic applications the calcined aluminum oxide is generally ball milled and commercially supplied as a fine powder, usually below 10 or 20 μm in size. A binder such as methyl cellulose and a release agent may also be added to facilitate molding into simple and intricate shapes. The molded articles are then slowly oven dried and fired at fusion temperatures of up to 1650°C, the resulting product being a hard impermeable ceramic of high strength and chemical resistance.

The firing of high-aluminum oxide ceramic at high temperatures is a solid-state sintering process in which the aluminum oxide grains fuse at their grain boundaries and atomic diffusion across interfaces occurs. Volume diffusion, whether along grain boundaries or through lattice dislocation, is followed by shrinkage. The initial stage of sintering is by the neck growth between the original powder particles and involves slight increase in density of about 10%. The beginning of the intermediate stage coincides with the beginning of grain growth. During this stage, particles grow to a grain-like structure with pores as channels lying on three-grain edges. The final stage starts when the cylindrical pores are transformed into spherical voids at about 5% porosity resulting in a dense crystalline structure with an average grain size of 4 μm (Andersson *et al*., 1993). The shrinkage of aluminum oxide during sintering to full density is about 15-20%, which has made it impossible to manufacture individual tooth copings of acceptable accuracy in pure aluminum oxide with the powder technology available in the 1970's.

High purity alumina is biocompatible and has been used as implant material since 1968 when Sandhaus used this material for tooth replacements (Sandhaus, 1968). McLean and Hughes were the first to describe a method to reinforce dental porcelain crowns using alumina, in their development of the aluminous jacket porcelain crown (McLean *et al*., 1965). Aluminous core porcelain is a typical example of strengthening by dispersion of a crystalline phase. Alumina has a high modulus of elasticity (350 GPa) and high fracture toughness (3.5 to 4 $MPa.m^{0.5}$). Its dispersion in a glassy matrix of similar thermal expansion coefficient leads to significant strengthening of the core. The first aluminous core porcelains contained 40 to 50% alumina by weight (McLean *et al*., 1965). The core was baked on a platinum foil and later veneered with matched-expansion porcelain. Flexural strengths of over 120MPa were obtained for these materials. It may be generally stated that the strength and opacity of alumina-reinforced porcelain is a function of its crystals or particle size. The finer the crystal size the greater the strength and opacity.

There are several techniques and methods in fabricating alumina reinforced porcelain crowns that utilize the principle of dispersion strengthening.

Traditional platinum foil technique

McLean popularized the modern Aluminous Jacket Crown, probably more commonly known as the Porcelain Jacket Crown in the mid 1960's. The fabrication of this crown utilized a technique using a platinum foil (0.0005-0.002" inch) that was meticulously and intimately adapted to the die. The adapted platinum foil served as a scaffold for build-up and firing of the porcelain and also acted as a die spacer to provide the required space of the luting agent. Platinum was used because its coefficient of thermal expansion ($9x10^{-6}/°C$) is slightly greater than that of the core porcelain so that upon cooling, the porcelain was subjected to compressive forces. To achieve maximum strength in such restorations, it was essential that the load-bearing area be reinforced with at least 1.0mm thick section of core porcelain. The ideal aluminous porcelain coping for incisors needed to be extended similarly to a metal coping in which the lingual surface collar was at least 2.0mm in height, and the incisal labial surface was thinned to 0.3mm for esthetic needs since this area was considered a low stress-bearing (McLean *et al*., 1976).

After the build-up of the core, a specially formulated all-ceramic crown porcelain with similar coefficient of thermal expansion and less alumina crystal content was stratified and fired to complete the crown fabrication. The platinum foil was then peeled off the internal surface of the crown before cementation. The disadvantages of this technique were the unreliable accuracy of the crown fit and the exposed defects in the internal surface caused by incomplete wetting of platinum foil by the molten porcelain that created porosity along the interface of the foil and the porcelain crown affecting the compressive strength of the restoration (Munoz *et al*., 1982).

The platinum twins foil technique

With the objective of improving the strength and consequently the fracture resistance of the porcelain jacket crown fabricated with the traditional foil technique, Sced and McLean reported a procedure for a reinforcement of the jacket crown, as a result of inhibiting the propagation of cracks into brittle materials, so called the twins foil technique (Sced *et al.*, 1977). In this technique, a platinum foil was adapted to the die to act as a spacer and a matrix as in the conventional alumina crown technique. A second platinum foil was then adapted over the first and plated with 0.2-2.0μm of tin and oxidized. Alumina core porcelain, which was modified to mask the grayness of the foil, was fired onto the plated foil. Enameling porcelain was then built to complete the crown fabrication. The inner foil was removed, but the plated foil remained as a permanent part of the completed crown.

The purpose of the plated foil was to increase the wettability of the platinum by the core porcelain. Ordinarily, platinum tenaciously retained absorbed carbons. The tin oxide coping prevented the platinum surface from acting as a contact catalyst for carbon compounds. As a consequence a chemical bond between the plated oxidized foil and the core porcelain resulted. The oxide could bond to both the metal and the porcelain or the oxide could dissolve into the glass and bring the porcelain into atomic contact with the metal (Sarkar *et al.*, 1981). As microcrack propagation was inhibited by the bond, this resulted in an increase in fracture strength up to 83% over the conventional aluminous porcelain crown (Sced *et al.*, 1977). The other advantage of this technique was that the bonded foil could inhibit the weakening effect of moisture on the strength of porcelain crowns.

As with the conventional platinum foil technique, the fit of the platinum foil porcelain crown was criticized as the weakest point of the technique. The platinum twins foil technique exhibited even poorer overall fit than the conventional technique along the axial walls and the margin of the crown, which weakened the strength of the crown after cementation (Munoz *et al*., 1982; Philip *et al*., 1984). Using the same technique, different core materials were tried posteriorly trying to solve the most important problems associated with the aluminous jacket crown - strength, porosity, and misfit. One of these materials was the magnesia jacket crown developed by O'Brien in 1985 (O'Brien, 1985). This material could be used in the construction of all-ceramic jacket crowns with the same body and enamel porcelains used for porcelain fused to metal crowns. Jacket crowns could be constructed using a modified platinum foil technique with greater accuracy and higher strength. The magnesia core ceramic developed as an experimental material had a high thermal expansion coefficient (14.5 x 10^{-6}/°C) that closely matched that of body and incisal porcelains designed for bonding to metal (13.5 x 10^{-6}/°C). The flexural strength of unglazed magnesia core ceramic was twice as high (131 MPa) as that of conventional feldspathic porcelain (65 MPa). The core material was made by reacting magnesia with a silica glass within the 1100-1150°C temperature range. These treatments lead to the formation of forsterite (Mg_2SiO_4) in various amounts, depending on the holding time. The proposed strengthening mechanism was the precipitation of fine forsterite crystals. The magnesia core material could be significantly strengthened by glazing, thereby placing the surface under residual compressive stresses that have to be overcome before fracture could occur (Wagner *et al*., 1992). The main advantage was a stronger jacket crown with exceptional esthetics without the need for special equipment or long processes (Hondrum, 1988; Hondrum *et al*., 1988).

The refractory die technique

In order to improve the strength of the porcelain jacket crown an alternative method was developed to fabricate this crown using a refractory die material. The refractory die was a phosphate-bonded type investment and had a thermal expansion comparable with aluminous porcelain. The core porcelain used for this technique had not been opacified and contained little or no quartz (McLean, 1988). When correctly fired, the light transmission of the core and all-ceramic crown materials approached that of dentin and enamel respectively.

The refractory die technique was reported to improve the internal surface of the crown. In light of this claim, Southan did a study to compare the fracture load of aluminous porcelain discs made on refractory dies formed in polyvinylsiloxane impressions to fracture load of the discs of the control group (Southan, 1987). He found that there was no difference in fracture strength. However, the fracture loads of disc prepared on refractory dies formed in the alginate impressions and those with deliberately flawed surfaces were significantly weaker than the samples formed on refractory dies produced from polyvinylsiloxane impressions.

The marginal discrepancy of these crowns was still poor and consequently the fracture resistance. However, Southan and co-workers found that a refractory die could be wetted better than the platinum foil and thus felt that they could get better marginal adaptation (Southan *et al.*, 1973).

Subsequently, the Hi-Ceram® system (Vident™, CA, USA) was developed to make an aluminous porcelain jacket crown (Schmidseder, 1986). The alumina core was built on a refractory die with subsequent buildups applied using standard feldspathic porcelain powders. The flexural strength of this porcelain crown was approximately 150 MPa (Oilo, 1988). This system was used in the fabrication of inlays, onlays, veneers, and all-ceramic crowns for anterior teeth (McLean *et al*., 1994). Although the inherent problems of fracture resistance persisted the development of the Hi-Ceram® system was an important step toward the development of other systems.

Injection molded core porcelain

In 1983, Sozio and Riley introduced a unique injection molding process alumina core porcelain system and called it Cerestore® (Coors Biomedical, CO, USA – Johnson & Johnson, USA) (Sozio *et al*., 1983). The Cerestore® crown system relied on the enameling of a 60% alumina body that was transfer-molded in the plastic state onto an epoxy die. This material possessed some interesting features, since it was the first non-shrink ceramic produced in dentistry. Essentially aluminum magnesium oxides were mixed with a barium glass frit that, on firing, produced a magnesium aluminate spinell. The spinell occupied a higher volume than the original mixed oxides, and compensated for firing shrinkage. A reinforcing core similar to the aluminous porcelain crown was constructed in this material onto which matched-expansion feldspathic all-ceramic crown porcelain was baked in the conventional manner to create the crown form (Sozio *et al*., 1985). The Cerestore® crown failed for two main reasons: high cost of the system and crowns with inadequate strength (89Mpa -150Mpa) when compared with metal ceramic restorations (Oilo, 1988; Anusavice, 1991). The manufacturer states that several thousand crowns are currently in use. However, to date, the failure rates for anterior and posterior crowns have not been documented in controlled clinical studies. The only documentation available reported a four-year failure rate of 18.5% for molar crowns.

Slip-cast alumina ceramics

Slip-casting is the art or science of preparing stable suspensions and forming ware by building up a solid layer on the surface of a porous mold that sucks up the liquid phase by means of capillary forces. The most common material used in slip-casting is plaster of Paris. The process has been used in forming clay bodies for at least 200 years, but it is only recently that the principle has been applied to nonplastic materials.

Slip casting is generally carried out with most particles between 1-5µm ranges. The bulk of the particles should be in a size range where their interactions are beginning to be governed by surface forces rather than by gravity. If however, the bulk of particles are too fine, it is difficult to exercise proper rheological control over the slip. Minor changes in the ionic atmosphere surrounding such particles can exert considerable influence on the forces of interaction and thereby cause sudden undesirable viscosity changes.

In-Ceram® Alumina

The slip-casting technique was posteriorly adapted to crown and bridge fabrication to produce a high strength alumina coping that was marketed under the trade name In-Ceram® (Vita® Zahnfabrick, Germany) (Probster *et al.*, 1992). This system was first described by Sadoun and was introduced to the dental community at the 1989 International Dental show in Stuttgart. In-Ceram® is a high strength aluminous core ceramic with extremely high flexural strength of the core which is derived from slip-casting of alumina (content in excess of 85%), and lanthanum-silicate glass infiltration into voids and pores in a second firing process.

The pure alumina slip-cast is made on a special gypsum die of the tooth preparations. The greenware is dried and slowly brought to a temperature of 1100°C for 2 hours. This firing process allows the alumina grains to partially fuse at their grain boundaries while the gypsum die shrinks. The core may then be lifted off the die. The fit of the partially sintered alumina cores can be very accurate. Final shaping must be done at this stage since the subsequent infusion of the glass matrix makes finishing operations very difficult because of the hardness of the final ceramic. After shaping, the porous alumina is ready for the infusion of the glass matrix and has a minimal shrinkage of about 0.21% (Campbell *et al*., 1995). A specially prepared low-fusing glass of matching thermal expansion is painted over the external surface of the core, and then placed on a thick piece of platinum foil. The glass melts at 800°C and when the temperature is raised to 1100°C it diffuses through the porous alumina by capillary action resulting in a very dense alumina/glass interpenetrating phase composite structure. The core is then all-ceramic crowned with the appropriated low fusing porcelain. The different thermal expansion coefficients of glass and alumina contribute to the strength, and effectively limit crack propagation (Probster, 1993). Flexural strength of the In-Ceram® Alumina core determined by three-point, four-point, and biaxial flexural strength was found to vary from 236.15 to 530 MPa (Seghi *et al*., 1995; Giordano *et al*., 1995; Wagner *et al*., 1996; Zeng *et al*., 1998; Tinschert *et al*., 2000; Chen *et al*., 2008). When tested with its porcelain laminate, the flexural strength of In-Ceram® Alumina was found to range from 174.2 to 240 MPa using biaxial bending test (Zeng *et al*., 1998). The strength value depends on the different equations used to derive the formula for bilayer model and the thickness of the laminate (Zeng *et al*., 1998). The strength of the In-Ceram® Alumina core was lower when tested with its porcelain laminate (White *et al*., 1994).

This material was intended for single anterior and posterior crowns, and anterior three-unit bridges. The high alumina content of the In-Ceram®

core makes it resistant to acid etching; therefore conventional cements are used for cementation. However, the crowns can be adhesively luted with resin cement after sandblasting (Probster *et al.*, 1992). Margins accuracy is acceptable independently of the method used for cementation (Pera *et al.*, 1994)

In-Ceram® Alumina has proven to be an acceptable treatment alternative for single crown as well as anterior fixed partial dentures. The 5-year survival rate of In-Ceram® Alumina crowns range from 91.7% to 100% and is similar to the survival rate of conventional metal-ceramic crowns. The 5-year survival rate of single-retainer In-Ceram® Alumina resin-bonded fixed partial dentures (RBFPDs) was 92.3%, which is higher than that of 2-retainer RBFPDs (Wassermann *et al.*, 2006). Although the material presented excellent results, and is still today one of the most preeminent ceramic systems used, from the esthetic and strength perspective, two problems where usually found. The material could not be used in highly esthetic situations and in the fabrication of posterior fixed partial dentures. The first problem is related with the opacity derived from the high alumina content, which is around 70%; and the second problem is related with insufficient strength to resist high masticatory forces. In order to solve these problems the Vita® Company developed the In-Ceram® Spinell system and the In-Ceram® Zirconia system.

In-Ceram® Spinell

The In-Ceram® Spinell manufacturing resembles that of In-Ceram® Alumina, with the exception that the core is made of a glass infiltrated magnesium alumina (Spinell $MgAL_3O_4$). The indication for the In-Ceram® Spinell crowns exists only for anterior single crowns when more translucency is desired and where problems evident in transiluminating light caused by the semi opaque oxide core material of the In-Ceram® Alumina. The new core material was able to offer glass-like light transmission and, accordingly, a more natural appearance to the final restoration (Paul *et al.*, 1995; Magne *et al*. 1997) In-Ceram® Spinell crowns are cemented and adjusted in the same way as in the In-Ceram® Alumina crowns. However, the crowns are more susceptible to fracture than the In-Ceram® Alumina crown, mainly because the flexural strength of the material is lower, around 350 MPa (Seghi *et al*., 1995; Fradeani *et a*l., 2002). The Spinell material has shown that it possesses encouraging characteristics if used correctly under normal-colored abutment teeth to maximize its translucency properties. The material is contraindicated in situations where teeth are dark do to endodontic treatment or over metal cores. The survival rates of the material are similar to those of In-Ceram® Alumina (Wassermann *et al*., 2006).

In-Ceram® Zirconia

The In-Ceram® Zirconia will be discussed in this section instead of in the Zirconia strengthened ceramics section, because contrary to the other Zirconia based ceramics systems, the manufacturing procedure is different and the Zirconia content within the material is also different. The In-Ceram Zirconia manufacturing resembles that of In-Ceram® Alumina, with the exception that the core is made of a glass infiltrated Zirconia/alumina ($ZrO_2AL_3O_4$). The difference is the addition of 35% partially stabilized Zirconia to the slip composition (McLaren *et al*., 1999). The partially stabilized Zirconia oxide undergoes phase transformation in the presence of propagating crack, resulting in a 3% to 5 % volumetric expansion (Garvie *et al*., 1975; Seghi *et al*., 1995). The volumetric change subjects the vicinity of the transformed Zirconia to a state of compression, which contributes to strengthening of the ceramic (Piconi *et al*., 1999). The indication for the In-Ceram® Zirconia exists only for anterior or posterior single crowns and fixed partial dentures (McLaren *et al*., 1999). However, because of the opacity of the Zirconia core, the use of the material for anterior crowns should be correctly evaluated (Heffernan *et al*., 2002; Hefernan *et al*., 2002). The flexural strength of the material was reported to be between 500 and 620 MPa (Guazzato *et al*., 2002; Chong *et al*., 2002; Chai *et al*., 2007; Chen *et al*., 2008). The high alumina and Zirconia content of the In-Ceram® Zirconia core makes it resistant to acid etching; therefore conventional cements are used for cementation. However, the crowns can be adhesively luted with resin cement that increases the bond strength between tooth preparation and the material (Della Bona *et al*., 2007). The marginal and internal fit of all-ceramic three-unit fixed partial denture fabricated by slip-cast technique showed satisfactory results (Bindle *et al*., 2007).

Long term clinical results and survival rates for In-Ceram® Zirconia crowns or FPDs are insufficient. Then no statement can be made presently regarding the reliability of the material (Wassermann *et al*., 2006). The only study available for fixed partial dentures reveals success rates of 95% at 3 years (Suárez *et al*., 2008).

CAD-CAM alumina ceramics

CAD/CAM technology has been developed since the first interactive graphics was developed in 1960s. CAD uses computer graphics and software to enable professionals to do most of design activities needed to complete professional design that include creating parts in 3D, assembling them, and producing software drawings. CAM generates tool path (i.e., NC [numerical control] part program) with reference to CAD database for geometric references. The CAD/CAM technology is a result of advancement of computer hardware and software technologies and it is used in almost all areas of industries in many different forms helping people to do efficient jobs in design and manufacturing activities.

In 1993 Matts Andersson and colleagues developed a new technique for manufacturing an individual all-ceramic crown built on a coping of dense sintered high purity alumina which when combined with dental porcelain could be used as the core for an all-ceramic restoration (Andersson *et al*., 1993). The all-ceramic restoration was named the Procera® AllCeram Alumina (Procera®, Nobel Biocare™ AB, Sweden) and it was developed by Nobel Biocare (Andersson *et al*., 1996). This crown was produce using the technology CAD-CAM, and it was the first application of this technology to restorative dentistry.

Procera® AllCeram Alumina

The Procera® system appears to fulfill the long-range research plan for the National Institute of Dental Research in the decade of the 90s that called for the development of cost-effective computer assisted systems for fabricating dental restorations. The Procera® system is one all-ceramic system that embraces the concept of CAD/CAM to fabricate dental restorations. Initially, the Procera® system was used to fabricate crowns and fixed partial dentures by combining a titanium substructure with a low-fusing veneering porcelain with promising results (Andersson *et al*., 1989). This CAD/CAM technology has been used to produce the Procera® AllCeram crown. This crown is composed of a densely sintered, high purity aluminum oxide coping (Al_2O_3>99.9%) with 500ppm MgO that is combined with a low-fusing AllCeram veneering porcelain. The system relies on the production of an aluminum oxide core as a substitute for the metal framework. One problem of dispersion strengthening is that if too much crystalline material is added to a glass, it becomes too viscous to fire to a glaze in an ordinary porcelain furnace, and other forming methods are needed. The Procera® porcelain core avoids this problem using unique forming processes. Because the core has so much alumina as explained previously, it cannot be fired in an ordinary furnace and instead is machined using high precision CAD/CAM technology.

The Procera® system consists of a computer-controlled design station in the dental laboratory that is joined through a modem link to the production facility in Sweden, where the coping is fabricated. At the design station, a scanning device controlled by a personal computer maps the surface of the die of the prepared tooth. A die of the tooth preparation is ditched below the finish line to clearly define the extent of the preparation. A sapphire ball forms the tip of the scanner probe that contacts the surface of the die as it rotates around a vertical axis. Light pressure of 20g maintains the probe in contact with the die. As the platform rotates, one data point is collected at every degree around the 360-degree circumference of the die. During each rotation of the die, the probe is automatically and continuously elevated 200μm by the computer and another scan line is read until the entire surface contour of the die has been mapped, thus describing the tooth through the use of approximately 50,000 measured values. When the scanning is completed, the data is evaluated for completeness. Marking of the finish line on the three-dimensional plots is the next step. At every 10 degrees of rotation, the operator marks the finish line, and the software interpolates the segment between the marks. The finish line is further refined by the operator, who repeats this process by marking the margin at 5-degree increments. The next step is to establish the thickness of the coping to be fabricated. A default of 600μm is customary for the coping thickness; however, for special situations the operator may choose to change this dimension. The emergence angle of the coping from the tooth is selected, and the relief space for the luting agent is automatically established by a computer algorithm. When the design of the coping has been completed, the file is saved in the computer and is ready for transmission via modem to the production station (Nobel Biocare) (Andersson *et al*., 1998).

The data of the preparation and the design of the coping are transferred via modem to the facility production in Sweden, where the coping is manufactured with advanced powder technology and the CAD/CAM technique. This process takes into account the sintering shrinkage of approximately 20% by enlarging a model of the preparation that is used in the manufacturing process. A high-purity aluminum oxide powder is compacted against the enlarged preparation model, with an industrial dry pressing technique, against the enlarged replica. The compacted alumina is pre-sintered to a green stage. The core is then removed from the die and sintered unsupported at 1550°C. After cooling, the outer surface of the core is formed by grinding and milling the alumina and adjusting the coping along the preparation to a predetermined dimension. Sintering of the high alumina core occurs at 1550°C, and the wall thickness of the sintered coping is from 0.5 to 1mm with standard core thickness of 0.6mm (Andersson *et al*., 1998). The color of the dense sintered alumina is close to A3 in the Vita shade system. The coping is examined for quality control and is sent by mail to the dental laboratory, where the ceramist finalizes the restoration by the addition of the AllCeram veneering porcelain. The Procera® coping is veneered with AllCeram ceramic, which is specially adapted to the coefficient of thermal expansion $7x10^{-6}$ µm/mol of the aluminum oxide framework. Further layers of the veneering porcelain include specific dentins, enamels, and transparent masses. The Procera® AllCeram crown is recommended for single crown restorations anterior and posterior, and 3 unit bridges restoration from 2nd to 2nd premolar.

The flexural strength of the aluminum oxide Procera® coping has been reported in several studies ranging from 510 to 670MPa (Anderson *et al*., 1993, Wagner *et al*., 1996). Mean gap dimensions for marginal openings, internal adaptation, and precision of fit for this crown are reported to be below 70 microns for premolar and molars (May *et al*., 1998) independently of the luting agent or finishing lines used (Quintas *et al*., 2004). Although the fracture resistance may be different depending on the cement used, the values obtained are well above the maximum masticatory forces (Al-Makramani *et al*., 2008). The clinical success of these crowns is well documented with survival rates between 97 and 99% at 5 and 7 years respectively, and 93.5% at 10 years (Odman *et al*., 2001, Zitzmann *et al*., 2007).

Lithium-disilicate-strengthened ceramics

Increasing interest in ceramic fixed prostheses has followed improvements in strength, esthetics, and ease of processing. Such advances included introduction of lithium disilicate ($Li_2O{\cdot}2SiO_2$) reinforced glass ceramics for dental use. The increased strength and improved esthetics of these systems are well-documented in the dental literature (Holand *et al*., 2000). The development of these systems, with lithium disilicate as the main crystalline phase, presume that the material could be used for the fabrication of single crows in the anterior or molar regions and three-unit fixed partial dentures extending to the premolar region. The ceramic restorations produced by these systems follow the common fabrication of injection pressed ceramics. The waxed pattern of the restoration is invested in a refractory material, which is preheated at 850 °C for one hour to eliminate wax and create a mold, which is subsequently transferred to a special pressing furnace. The pre-cerammed ingots, supplied by the manufacturer in a variety of shades, are then placed in the open end of the mold and pressed by a thermal resistant plunger attached to the furnace. After yielding through the sprues (connectors), the cavity of the mold is filled by the viscous flow of the glass-ceramic. In this first step, only the framework of a crown or fixed partial denture is hot-pressed. The esthetic characterization of the lithium disilicate glass-ceramic can be achieved by the layering technique. The final shape of the restoration is obtained by applying a sintered glass-ceramic in layers, which are fired in a conventional porcelain furnace. Several systems are employing the lithium disilicate ceramics in the fabrication of restorations with satisfactory results. However, some reports have suggested that they may have some biological risks (Brackett *et al*., 2008). The results of these reports suggest that lithium disilicates are not biologically inert, and that many have a similar cytotoxicity dynamic regardless of small differences in composition or processing.

IPS-Empress® 2

The IPS Empress® 2 or IPS Empress® Eris (Ivoclar, Schaan, Liechtenstein) was first presented in November 1998. The material is composed of a lithium disilicate glass-ceramic that exhibits higher fracture strength then IPS Empress® 1. The material is fabricated in the same way as IPS Empress® 1 by a hot-pressing technique. The glass ceramic is pre-cerammed by the manufacturer and supplied in ingots for pressing in a pressing furnace. The crystalline phase that forms during the ceramming of this glass is lithium disilicate ($Li_2O{\cdot}2SiO_2$), which makes up about 65% of the volume of this glass ceramic. This microstructure is unusual because it consists of many small interlocking plate-like crystals that are randomly oriented. The interlocking nature of the crystals, as well as their high density gives the glass ceramic very high flexural strength (350/400MPa), and high fracture strength (Oh *et al*., 2000; Albakry *et al*., 2003; Albakry *et al*., 2003).

Lithium disilicate crystal is a laminated silicate that exhibits a tight cross-linking of the SiO_3 tetragonal lattice. The strengthening mechanism of lithium disilicate is attributed to a higher percentage volume reduction of the particles compared to the surrounding glass matrix upon cooling. The higher percentage volume reduction of the crystal is accounted for by its higher coefficient of thermal expansion in comparison with the glass matrix and a high-to-low temperature phase transformation. The result of pressing the material at a temperature of 920°C is a primary crystalline phase of needle-shape lithium disilicate crystal (0.5 to 5mm), which comprises approximately 65% volume (Albakry *et al*., 2004; Guazzato *et al*., 2004). The volume differential between the lithium disilicate particles and the glass matrix causes residual stresses that place the surrounding glass matrix in compression, which must be counteracted by tensile stresses before cracks propagate. Irrespective of the large percentage of lithium disilicate crystals, IPS Empress® 2 exhibits a high level of translucency due to the optical properties of the crystal structures. In addition to lithium disilicate crystals, the material contains minor quantities of lithium orthophosphate (Li_3PO_4) with particle sizes between 0.1 and 0.3mm.

The IPS Empress® 2 ceramic is etchable with hydrofluoric acid and needs to be cemented with a resin base luting agent. A special apatite fluoride glass-ceramic was developed for veneering of the IPS Empress® 2 frameworks. This veneering ceramic can be sintered at 800°C. During this process, a part of the apatite crystals of the glass matrix is precipitated out of the glass-ceramic. These crystals help to achieve a higher level of biocompatibility and optical characteristics such as translucency, brightness, and light-scattering of the veneering material.

The framework of the material may be luted to the dental preparation using conventional or adhesive cementation techniques opposing the IPS Empress® 1 system that can only be resin cemented. However, when retentive areas are small, retention may be inadequate, and an adhesive luting agent is desirable because bond strength can be enhanced (Spohr *et al*., 2003). The results of 5-year clinical evaluation suggest that IPS Empress® 2 ceramic is an appropriate material for the fabrication of single crowns with success rates around 95% (Toksavul *et al*., 2007; Marquardt *et al*., 2006). Survival rates of fixed partial dentures however, reveal a very low success rate between 50 and 70 % at 2 and 5 years respectively (Marquardt *et al*., 2006; Taskonak *et al*., 2006). Strict conditions should be considered before the use of IPS Empress® 2 material for the fabrication of three-unit fixed partial dentures.

3G-OPC®

The 3G-OPC® (Optimal Pressable Ceramic) (Jeneric/Pentron Inc., CT, USA) is a pressable ceramic with a needle-like crystalline structure made of lithium disilicate. The glass ceramic material restorations are virtually fabricated in the same manner as the IPS-Empress® 2 crown. The only differences between the two technology systems are the price and the ceramic ingots supplied to construct the ceramic restorations. The strength reported for the 3G-OPC® crown was similar to that of Empress® 2 around 350 MPa (Jin *et al*., 2004), but less then Procera® and In-Ceram® Alumina. The 3G OPC® is based on lithium disilicate glass-ceramic and crystal-free overlay porcelain. Due to the fact that the interlocking lithium disilicate crystals prevent crack propagation, 3G-OPC® has higher fracture toughness and flexural strength when compared to former OPC products. For instance, its fracture strength is two times as that of OPC and it is sufficient for use in three-unit anterior fixed partial dentures. The ceramic can be used for anterior and posterior single unit crowns and three-unit anterior fixed partial dentures. Because of its fluorescing core and overlay porcelain, it can achieve good esthetic results. The fact that the overly porcelain is crystal free, it causes less abrasive wear of the opposing natural dentition. No long term data is available for this crown system.

Zirconia-strengthened ceramics

The increased demand for optimal esthetics has made metal-ceramic restorations no longer adequate to meet the expectations of both clinicians and patients. In this respect, the all-ceramic crown set a standard in esthetics that is difficult to match by the metal-ceramic crown because of the absence of underlying metal and increased light transmission through the restoration. These all-ceramic crowns with high strength still remain susceptible to a high mechanical loading of chewing force. The absence of reinforcement by a metal substructure results in relatively weak flexural strength and fracture resistance. Therefore, currently available all-ceramic restorations have limited clinical application in areas of high stress, such as fixed partial dentures. In order to overcome such a mechanical weakness of all-ceramic crown systems, several all-ceramic companies have recently investigated a high strength new ceramic material composed of Zirconia for the coping of all-ceramic crown. Its superior mechanical behaviors over alumina ceramic have been reported (Christel et al., 1989).

Currently available all-ceramic crowns are practically limited to single tooth restorations. The indication that Zirconia containing ceramics exhibit durability in a highly loaded environment makes them attractive for use in dentistry and increase the broad of clinical indications to posterior molar areas and posterior fixed partial dentures.

Zircon has been known as a gem from ancient times. The name of the metal, zirconium comes from the Arabic Zargon (golden in color) that in turn comes from the two Persian words Zar (Gold) and Gun (Color). Zirconia, the metal dioxide (ZrO_2), was identified as such in 1789 by the German chemist Martin Heinrich Klaproth in the reaction product obtained after heating some gems, and was used for a long time blended with rare earth oxides as pigment for ceramics (Piconi *et al*., 1999).

Good chemical and dimensional stability, mechanical strength and toughness, coupled with a Young's modulus in the same order of magnitude of stainless steel alloys was the origin of the interest in using Zirconia as a ceramic biomaterial (Piconi *et al*., 1999). The first paper concerning biomedical application of Zirconia was published in 1969 by Helmer and Driskell (Helmer *et al*., 1969), while the first paper concerning the use of Zirconia to manufacture ball heads for Total Hip Replacements (THR), which is the current main application of this ceramic biomaterial, was introduced by Christel (Christel *et al*., 1989).

Zirconia is a polycrystalline ceramic without any glass component. It is a polymorph that occurs in three forms: monoclinic (M), cubic (C), and tetragonal (T). The cubic phase is stable but brittle, while the tetragonal phase is tough but unstable. Pure Zirconia is monoclinic at room temperature. This phase is stable up to 1170°C. Above this temperature it transforms into tetragonal and then into cubic phase at 2370°C. Pure Zirconia has a high melting point (2700°C) and a low thermal conductivity. Its polymorphism, however, restricts its widespread use in ceramic industry.

This results from the fact that noticeable changes in volume are associated with these transformations. During the monoclinic-to-tetragonal transformation, which occurs when Zirconia is heated, there is a 5% volume decrease; conversely, a 3% increase in volume is observed during the cooling process. Stresses generated by the expansion originate cracks in pure Zirconia ceramics that break into pieces at room temperature. These phenomena have been shown detrimental to the mechanical behavior of Zirconia because the stresses induced during the phase transformations resulted in crack formation inhibiting the use of pure Zirconia in many applications. This phase transformation of zirconium oxide can be inhibited by the addition of stabilizing oxides, like CaO, MgO, CeO_2 and Y_2O_3 to pure

Zirconia. Thus the undesirable phase transformation is prevented and allows the material to generate multiphase materials known as Partially Stabilized Zirconia (PSZ) whose microstructure at room temperature generally consists of cubic Zirconia as the major phase, with monoclinic and tetragonal Zirconia precipitates as the minor phase (Subbarao, 1981; Piconi *et al*., 1999). These precipitates may exist at grain boundaries or within the cubic matrix grains. Usually PSZ consists of larger than 8 mol% of MgO, 8 mol% of CaO, or 3-4mol% of Y_2O_3. PSZ is a transformation toughened material and distinguished by a crack initiation mechanism.

Two kinds of microstructures can be generated. In the ZrO_2-MgO or ZrO_2-CaO systems, materials are sintered in the cubic state and small tetragonal precipitates are formed during cooling has a result of partial transformation of the cubic phase. In the ZrO_2-Y_2O_3 system, the extent of the stability range of the tetragonal phase in terms of temperature and amount of yttrium oxide allows sintering of fully tetragonal fine-grained materials. Thus, using Y_2O_3 as a stabilizing agent, it is possible to produce zirconium oxide ceramic made of 100% small metastable tetragonal grains. The volume change related to the tetragonal to monoclinic phase transformation results in a prestressed material. In this respect, a propagating crack can release the stresses on the neighboring grains that then transform from the metastable state into the monoclinic phase. The associated volume expansion results in compressive stress at the edge of the crack front and extra energy is required for the crack to propagate further. Thus, it is believed that the main energy absorbing mechanism is due to the martensitic-like transformation occurring at the crack tip.

Zirconia is usually produced from the zircon, $ZrSiO_4$. To produce Zirconia from zircon, zircon is first converted to zirconyl chloride ($ZrOCl_2.8H_2O$). There are two methods used to make Zirconia from the zirconyl chloride: thermal decomposition and precipitation. Once the zirconyl chloride is heated to 200°C, it starts dehydration and becomes dehydrated $ZrOCl_2$. On next step, $ZrOCl_2$ decomposes into chlorine gas and becomes Zirconia at a much higher temperature. Zirconia lumps obtained from the calcinations then undergo a size reduction process, such as ball milling, into the particle size range needed, usually up to 325 meshes. However, it is not easy to produce Zirconia powders with high purity and fine particle size by this method. Precipitation method, on other hand, uses chemical reactions to obtain the Zirconia hydroxides as an intermediate. Obtained Zirconia hydroxides go through wash, filtration, freezing dry, and calcination to turn into Zirconia powder. By this method, the grain size, particle shape, agglomerate size, and specific surface area can be modified within certain degree by controlling the precipitation and calcination conditions. Furthermore, using this process its purity is also easier to be controlled.

In order to prepare stabilized Zirconia powders, stabilizers (magnesia, calcia, or yttria) must be introduced into pure Zirconia powders prior to sintering. Stabilized Zirconia can be formed during a process called in-situ stabilizing. Before the forming processes, such as molding, pressing or casting, fine particles of stabilizer and monoclinic Zirconia are well mixed. Then the mixture is used for forming of green body. The phase conversion is accomplished by sintering the doped Zirconia at 1700°C. During the sintering, the phase conversion takes place. High quality stabilized Zirconia powder is made by co-precipitation process. Stabilizers are introduced during chemical processing, before the precipitation zirconium hydroxide. A cubic (or tetragonal) phase Zirconia is formed during calcination of chemically precipitated intermediates.

In the early stages of the development all these solid solutions (ZrO_2-MgO, ZrO_2-CaO, and ZrO_2-Y_2O_3) were tested for biomedical applications. But in the following years the research efforts appeared to be more focused on Zirconia-yttria ceramics (ZrO_2-Y_2O_3), characterized by fine grained microstructures known as Tetragonal Zirconia Polycrystals (TZP). The sintering behavior of Zirconia-TZP does not allow the manufacturing of abutments and crowns/bridges by direct sintering. Therefore abutments and crowns/bridges of industrial manufactured Zirconia-TZP ceramics can only be fabricated by grinding. In addition, copy grinding machining techniques requires CAD/CAM procedures. Luthardt and coworkers reported that prosthetic restorations manufactured using CAD/CAM technologies reached a level of quality (fracture strength, fitting accuracy) that warrants broad clinical application (Luthardt *et al*., 1999). Zirconia-TZP is being used as application in space shuttle, automobiles, cutting tools, and combustion engines because of its good mechanical and dimensional stability, such as mechanical strength and toughness. In vitro evaluation of the mutagenic and carcinogenic capacity of the high purity Zirconia ceramic confirmed that it did not elicit such effects on the cells (Covacci *et al*., 1999).

As mentioned previously yttrium-tetragonal Zirconia polycrystals (Y-TZP) is now posed to replace alumina ceramic because of its superior mechanical behavior. This material Y-TZP, exhibits an improved fracture toughness (KIC = 9-10 MN/m3/2) related to the transformation of tetragonal ZrO_2 grains into monoclinic phase and the associated compressive stresses occurring at the crack front. Y-TZP has a higher bending strength as compared to alumina (900-1200 vs. 400 MPa) associated with a much lower modulus of elasticity (200 GPa vs. 350 GPa), and a smaller grain size (0.5 µm vs. 7 µm). In addition, the ZrO_2 based material exhibits a lower Young's modulus pointing to an interesting elastic deformation capability.

The enhanced strength of this material can be explained by their characteristic microstructural differences: Y_2O_3-partially-stabilized ZrO_2 ceramic has a higher density and a smaller particle size than Al_2O_3 ceramic. Moreover, the hardness of both ceramics is essentially different. The main reason for the superior fracture strength of ZrO_2 ceramic, however, lies in the metastable tetragonal crystalline structure at room temperature. This structure represents an efficient mechanism against flaw propagation and has a strong impact against flaw sub-critical crack growth. However, ZrO_2 ceramic exhibits 1 to 10 lower thermal conductivity than Al_2O_3 ceramic (Munz *et al*., 1995). Temperature peaks can alter the metastable tetragonal crystalline phase of partially stabilized ZrO_2 ceramic. Therefore ZrO_2 ceramic materials should be more endangered than Al_2O_3 ceramic materials by heat producing surface treatments, which produce high temperature spots because of the very slow heat dissipation.

Although many types of Zirconia-containing ceramic systems are currently available, only three are used to date in dentistry. These are yttrium cation-doped tetragonal Zirconia polycrystals (3Y-TZP), magnesium cation-doped partially stabilized Zirconia (Mg-PSZ) and Zirconia-toughened alumina (ZTA).

Zirconia containing ceramics systems

Yttrium cation-doped tetragonal Zirconia polycrystals (3Y-TZP)

Biomedical grade Zirconia usually contains 3mol% yttria (Y_2O_3) as a stabilizer (3Y-TZP) (Piconi *et al*., 1999). While the stabilizing Y^{3+} cations and Zr^{4+} are randomly distributed over the cationic sites, electrical neutrality is achieved by the creation oxygen vacancies (Eichler, 2001; Fabris *et al*. 2002). 3Y-TZP has been used to manufacture femoral heads in total hip replacement prostheses since late eighties but its used in orthopedic surgery has since been reduced by more than 90% mostly due to a series of failures that occurred in 2001 (Chevalier, 2006). 3Y-TZP is available in dentistry for the fabrication of dental crowns and fixed partial dentures. The restorations are processed either by soft machining of presintered blanks followed by sintering at high temperature, or by hard machining of fully sintered blocks (Filser *et al*., 2003).

The mechanical properties of 3Y-TZP strongly depend on its grain size (Burger *et al*., 1997; Ruiz *et al*., 1996). Above critical grain size, the material is less stable and more susceptible to spontaneous tetragonal to monoclinic transformation whereas smaller grain sizes (1µm) are associated with a lower transformation rate (Heuer *et al*., 1982). Moreover, bellow certain grain size (~0.2µm), the transformation is not possible, leading to reduce fracture toughness (Cottom *et al*., 1996). Consequently, the sintering conditions have a strong impact on both stability and mechanical properties of the final product as they dictate the grain size. Higher sintering temperatures and longer sintering times lead to larger grain sizes (Chevalier *et al*., 2004).

Currently available 3Y-TZP for soft machining of dental restorations utilizes final sintering temperatures varying between 1350 and 1550°C depending on the manufacturer. The fairly wide range of sintering temperatures is therefore likely to have an influence on the grain size and later the phase stability of the material for dental applications. From the phase diagram established by Scott, 3Y-TZP contains some amount cubic Zirconia (Scott, 1975). Chevalier and co-workers demonstrated that the presence of cubic Zirconia is not desirable in 3Y-TZP for biomedical applications and is caused by uneven distribution of yttrium stabilizer ions. The cubic grains are enriched in yttrium while the surrounding tetragonal grains are depleted and therefore less stable (Chevalier *et al*., 2004). As mentioned earlier, restorations produced by soft machining are sintered at later stage (i.e. following the forming steps), this process prevents the stress-induced transformation from tetragonal to monoclinic and leads to a final surface virtually free of monoclinic phase unless grinding adjustments are needed or sandblasting is performed. Most manufacturers of 3Y-TZP blanks for dental applications do not recommend grinding or sandblasting to avoid the tetragonal to monoclinic transformation and the formation of surface flaws that could be detrimental to the long-term performance, despite the apparent increase in strength due to the transformation-induced compressive stresses. In contrast, restorations produced by hard machining of fully sintered 3Y-TZP blocks have been shown to contain a significant amount of monoclinic Zirconia (Guazzato *et al*., 2004). This is usually associated with surface microcracking, higher susceptibility to low temperature degradation and lower reliability (Huang, 2003). Liu and co-workers studied the fatigue behavior o 3Y-TZP (Liu *et al*., 1991). The pre-existing processing flaws were identified as the fracture origin in all cases and microcracking was shown to be the dominant mechanism of fatigue damage. More recently, Zhang and co-workers studied the effect of sharp indentation damage on the long-term performance of this material. It was shown that both sandblasting and sharp indentations even at very low loads are detrimental to the long-term performance of 3Y-TZP when tested in cyclic loading (Zhang *et al*, 2005; Zhang *et al*., 2004). These studies pointed out the importance of controlling the final surface state of 3Y-TZP for biomedical applications. In summary, even if high strength might appear as beneficial property for dental applications, long-term performance and reliability should also be considered.

Several authors have reported that annealing at 900°C for 1h or relatively short heat treatments in the temperature range 900-1000°C for 1 min induce the reverse transformation from monoclinic to tetragonal (Kosmac *et al*., 2000; Sundh *et al*., 2005). This phenomenon was accompanied by the relaxation of the compressive stresses at the surface and a decrease in strength. The firing of veneering porcelain during the fabrication of dental restorations is therefore likely to promote the reverse transformation with the consequences listed above. In addition, the reversibility of the transformation should not be confused as providing a mechanism for healing of the flaws previously introduced.

The microstructure of 3Y-TZP ceramics for dental applications consists of small equiaxed grains (0.2-0.5μm in diameter, depending on the sintering temperature) (Guazzato *et al*., 2004). The mechanical properties are well above those of all other available dental ceramics, with a flexural strength in the 800-1000 MPa range and fracture toughness in the 6-8Mpa $m^{0.5}$ range. The Weibull modulus strongly depends on the type of surface finish and the processing conditions (Kosmac *et al*., 1999).

Glass-infiltrated Zirconia-toughened alumina (ZTA)

Another approach to advantageously utilize the stress-induced transformation capability of Zirconia is to combine it with an alumina matrix, leading to a Zirconia-toughened alumina (ZTA) (Lange, 1982; Lange, 1982). These materials have recently received interest as potential bioceramics (Deville *et al*., 2003; Deville *et al*., 2004). One commercially available dental product, previously discussed, In-Ceram® Zirconia was developed by adding 33 vol% of 12 mol% ceria-stabilized Zirconia (12Ce-TZP) to In-Ceram® Alumina (Guazzato *et al*., 2004). In-Ceram® Zirconia can be processed by either split-casting or soft machining. Initial sintering takes place at 1100°C for 2h, prior to this porous ceramic composite being glass infiltrated. The glass phase represents approximately 23% of the final product. One of the advantages of the split-cast technique is that there is very limited shrinkage. However, the amount of porosity is greater that of sintered 3Y-TZP and comprises between 8 and 11% (Guazzato *et al*., 2003). This partially explains the generally lower mechanical properties of In-Ceram® Zirconia when compared to 3Y-TZP dental ceramics. It should be pointed out, however, that Ce-TZP ceramics usually exhibit better thermal conductivity and resistance to low temperature degradation than Y-TZP under similar thermo-cycling or aging conditions (Tsukuma *et al*., 1985).

In-Ceram® Zirconia for machining is thought to exhibit better mechanical properties due to more consistent processing compared to the split-cast ceramic. Conversely, Guazzato and coworkers reported a significantly higher flexural strength for In-Ceram® Zirconia processed by split casting (630 ± 58 MPa) compared to the machined material (476± 50 MPa) (Guazzato *et al.*, 2005). There was no significant difference in fracture toughness. The two materials exhibited a very similar microstructure with large alumina grains (6 μm long, 2μm wide) together with clusters of small Zirconia grains (less than 1 μm in diameter). Some faceted Zirconia grains (2μm) were also observed. The crack patterns were consistently transgranular for ZrO_2 and intragranular for Al_2O_3. In some of the newly developed ZTA for biomedical applications, excellent mechanical properties are obtained by promoting a fine and uniform dispersion of Zirconia grains in an alumina matrix; such dispersion is readily obtained by sol-gel processing (Tanaka *et al.*, 2002). An advancing crack triggers the tetragonal to monoclinic transformation. The associated increase in volume creates microcracks in the alumina matrix surrounding the transformed particle. The toughness is therefore enhanced by microcracking. (Porter *et al.*, 1977)

Magnesia cation-doped partially stabilized Zirconia (Mg-PSZ)

Although a considerable amount of research has been dedicated to magnesia partially stabilized Zirconia (Mg-PSZ) for possible biomedical applications, this material has not been successful due mainly to the presence of porosity, associated with a large grain size (30-60μm) that can induce wear (Piconi, 1999). The microstructure consists of tetragonal precipitates within a cubic stabilized Zirconia matrix. The amount of MgO in composition of commercial materials usually ranges between 8 and 10 mol% (Green *et al.*, 1988). In addition, to a high sintering temperature (between 1680 and

1800°C), the cooling cycle has to be strictly controlled, particularly in aging stage with a preferred temperature of 1100°C (Green *et al.*, 1988). Precipitation of the transformable tetragonal phase occurs during this stage, which volume fraction is a critical factor in controlling the fracture toughness of the material (Hannink *et al.*, 1994). Due to difficulty of obtaining Mg-PSZ precursors free of SiO_2, magnesium silicates can form that lower the Mg content in the grains and promote the tetragonal to monoclinic transformation (Leach, 1987). This can result in lower mechanical properties and a less stable material. Denzir-M® (Denzir®, Cadesthetics AB, Skellefteå, Sweden) is an example of Mg-PSZ ceramic currently available for hard machining of dental restorations.

<u>Zirconia surface treatments</u>

Soft machining of pre-sintered blanks

Since its development in 2001 (Filser *et al.*, 2003); direct ceramic machining of pre-sintered 3Y-TZP has become increasingly popular in dentistry and is now offered by a growing number of manufacturers. Briefly, the die or a wax pattern is scanned, an enlarged restoration is designed by computer software (CAD) and computer aided machining mills a pre-sintered ceramic blank. The restoration is then sintered at high temperature. Several variations of this process exist depending on how the scanning is performed and how the large sintering shrinkage of 3Y-TZP (~25%) is compensated for. For example, both contact scanners and non-contact scanners are available. Overall, non-contact scanners are characterized by higher density of data points and a greater digitizing speed compared to contact scanners.

Typically the 3Y-TZP powder used in the fabrication of the blanks contains a binder that makes it suitable for pressing. The binder is later eliminated during the pre-sintering step. It also contains about 2 wt. % HfO_2, classically difficult to separate from ZrO_2. These powders have only minor variations in chemical composition. The powders consist of spray-died agglomerates (about 60 µm in diameter) of much smaller crystals that are about 40 nm in diameter. These blanks are manufactured by cold isostatic pressing. The mean pore size of the compacted powder is very small in the order of 20-30 nm with a very narrow pore size distribution (Filser *et al*., 2001).

The binder is eliminated during pre-sintering heat treatment. This step has to be controlled carefully by manufacturers, particularly the heating rate and the pre-sintering temperature. If the heat rate is too fast, the elimination of the binder and associated burn out products can lead to cracking of the blanks. Slow heating rates are therefore preferred. The pre-sintering temperature of the blanks affects the hardness and machinability. These two characteristics act in opposite direction: an adequate hardness is needed for the handling of the blanks but if the hardness is too high, it might be detrimental to the machinability. The temperature of the pre-sintering heat temperature also affects the roughness of the machined blank. Overall higher pre-sintering temperatures lead to rougher surfaces. The choice of a proper pre-sintering temperature is thus critical (Filser *et al*., 2001). The density of each blank is carefully measured so that the appropriated compensating shrinkage is applied during final sintering. The final density of the pre-sintered blanks is about 40% of the theoretical density (6.08g/cm^3). The density gradient within the blanks is lower than 0.3% of the theoretical density in all directions (Filser *et al*., 2001).

Machining is better accomplished in two steps. A first rough machining is done at a low feed rate while the final fine machining is performed at a higher feed rate (Filser *et al*., 2001; Filser *et al*., 2003).

Restorations can be colored after machining by immersion in solutions of various metal salts such as cerium, bismuth, iron or a combination thereof.

The color develops during the final sintering stage. The concentration of the solution strongly influences the final shade. Concentrations as low as 0.001 mol% are sufficient to produce a satisfactory coloration. The final sintering temperature influences the color obtained. Careful respect of the manufacture's instructions is therefore important. Coloration with various solutions does not appear to affect the crystalline phases or mechanical properties of the final product. Alternatively, colored Zirconia can be obtained by small additions of various metal oxides to the starting powder (Cales, 1998).

Sintering of the machined restoration has to be carefully controlled, typically by using specifically programmed furnaces. Shrinkage starts at 1000°C and reaches ~25%. Sintering conditions are product-specific. Final sintering temperatures between 1350 and 1550°C with dwell times between 2 and 5 h lead to densities greater than 99% of the theoretical density. These variations in sintering conditions are likely to be due the initial chemical composition of the 3Y-TZP powder. For example, small additions of alumina have been shown to act as a sintering aid, allowing the use of lower sintering temperatures and times. Prior to sintering, the frameworks are placed on Zirconia sintering beads to avoid deformation. The minimum thickness for the copings is 0.5mm, bellow which warpage could occur. The restorations are furnace-cooled to a temperature bellow 200°C to minimize residual stresses. As mentioned earlier, the sintering temperatures and times strongly influence the grain size (Matsui *et al*., 2003). Chevalier and coworkers also demonstrated that the amount of cubic phase in 3Y-TZP increases when the sintering temperature reaches 1500°C with a sintering time of 5h (Chevalier *et al*., 2004). The presence of larger cubic grains is detrimental to the resistance of the ceramic to low temperature aging. This point out the importance of carefully controlling the sintering step.

The restorations are finally veneered with porcelains of matching coefficient of thermal expansion. The nature of the interface between 3Y-TZP and the veneering porcelain has not been thoroughly studied. The veneering porcelain is baked at ~900°C, with a hold time of 1min. Although diffusion processes are time-dependent, chemical reactions could occur between the two ceramic materials. This point will be examined in greater detail later. Representative systems utilizing soft machining of 3Y-TZP for dental restorations are Lava™ (3M ESPE, Germany), Procera® Zirconia (Procera, Nobel Biocare AB, Sweden), YZ cubes for Cerec InLab (Vident, Baldwin Park, CA, USA) and Cercon® (Dentsply International, Inc., York, PA, USA).

Hard machining of 3Y-TZP and Mg-PSZ

At least two systems, Denzir® (Denzir, Cadesthetics AB, Skellefteå, Sweden) and DC-Zirkon® (DCS Dental AG, Germany) are available for hard machining of Zirconia dental restorations. Y-TZP blocks are prepared by pre-sintering at temperatures bellow 1500°C to reach a density of at least 95% of the theoretical density. The blocks are then processed by hot isostatic pressing at temperatures between 1400 and 1500°C under high pressure in an inert gas atmosphere (Piconi *et al*., 2006). This latter treatment leads to a very high density in excess of 99% of the theoretical density.

The blocks can then be machined using a specially designed milling system. Due to the high hardness and low machinability of fully sintered Y-TZP, the milling system has to be particularly robust. A study by Blue and co-workers demonstrated that Y-TZP was significantly harder to machine than fully sintered alumina with lower material removable rates (Blue *et al*., 2003). This was confirmed by Yin and co-workers who also reported that coarse diamond burs were more efficient for material removal with Y-TPZ, while machining with fine burs led to a more ductile type of damage (Yin *et al*., 2004). Huang studied the effect of grinding speed on the type of machining damage in Y-TZP and reported both brittle and ductile removal modes at high speed with less subsurface damage (Huang *et al*., 2003). On the other hand, the fine grain size of Y-TZP leads to very smooth surfaces after machining. As mentioned earlier, all surface treatments cause some degree of tetragonal to monoclinic transformation at the surface of Y-TZP. Kosmac and co-workers showed that sandblasting was more efficient than grinding in inducing the transformation, thereby promoting a greater increase in strength (Kosmac *et al*., 1999). Conversely, coarse grinding caused the formation of deep defects as well as reverse transformation with elimination of the compressive stresses and decrease in strength. Guazzato and Curtis on Y-TZP confirmed these results for dental applications (Guazzato *et al*., 2005; Curtis *et al*., 2006)

The influence of residual stresses on the susceptibility of Y-TZP to low temperature degradation has been thoroughly examined by Deville and co-workers (Deville *et al*., 2003). It was concluded that the presence of residual stresses was more influential than the final roughness in promoting low temperature degradation. Smooth polishing led preferential transformation after aging around the residual scratches. A thermal treatment at 1200°C for 2h induced the relaxation of the stresses and a lower susceptibility to aging than the polished state. Grant and co-workers reported that hot isostatically pressed (HIPped) 3Y-TZP had a lower susceptibility to low temperature degradation than the unHIPed material (Grant *et al*., 2001). The aging susceptibility of HIPped 3Y-TZP for dental applications is likely to follow a different scheme as the material is later machined. However, the difficulty of comparing the results of the numerous studies dedicated to surface treatments of Y-TZP should be pointed out, as there is no standardization of the treatments applied. In summary, questions remain about surface state remaining after hard machining of Y-TZP, while soft machining seems to lead to a more consistent final state, provided that the machined restoration is left intact after sintering.

Clinical studies of ZrO_2 fixed prostheses

There are approximately fifteen major long-term studies of Zirconia prostheses underway at this time (Denry *et al*., 2008). It seems noticeable that these studies mostly involve multi-unit and posterior prostheses. It is apparent that sponsoring manufacturers have some confidence in the structural potential of Zirconia frameworks. This also signals some comfort regarding the performance of this core ceramic for the restoration of single anterior teeth; reflecting the clinical finding that many less/strong tough all-ceramic systems are found to have 90% or higher survival at 5-6 years. It is also interesting that, other than for a few papers and IADR/AADR abstracts, there has been little reporting of early results (von Steyern *et al*., 2005; Larsson *et al*., 2006; Raigrodski *et al*., 2006). This stands in contrast to other ceramic product launches for which 2-year and even 1-year study results were quickly published.

Bulk fracture appears to be quite uncommon in all studies to date, even with the majority of study prostheses being multi-unit replacing first molars or second premolar (Denry *et al*., 2008). The fractures that have occurred mostly involve connectors of multi-unit prostheses or second molar abutments. Results for single-unit molar prostheses may turn out to be at least as good as for alumina-based core systems; such supposition being tempered of course by the relatively limited observation times. It is also obviously too early to judge whether microstructural or processing differences among Zirconia systems will be reflected in clinical performance. That said, it is rather remarkable to have such an emphasis on clinical examination of a new technology in dentistry.

Problems with the porcelain veneer seem to trouble all studies. In three published reports of four separate systems, 8, 15, 25 and 50% of prostheses developed crazy or cracking with minor loss of material after only 1-2 years of observation (von Steyern *et al*., 2005; Larsson *et al*., 2006; Raigrodski *et al*., 2006). Investigators involved with studies not yet published admit to porcelain problems as well. However, this picture is somewhat confused by non-research clinical experience. This may signal that the difficulties are material-specific, as was the conclusion in one published study of two systems exhibiting, respectively, 8 and 50% incidence of porcelain cracking (Larsson *et al*., 2006). It may also indicate that non-materials factors such as thickness ratios or framework design play a role in porcelain cracking. For comparison, porcelain problems on metal-ceramic prosthesis over a 10 years observation period was reported to be on the order of 4% for a gold-palladium alloy, no higher than 6% for most alternative alloys, and only as high as 15% for one nickel-based alloy without beryllium (Anderson *et al*., 1993). Consistent findings have been reported for another gold-based alloy, with 98% completely intact porcelain at 5 years (Walter *et al*., 1999). Lower survival percentages are reported for porcelain on titanium (84-87% survival at 5 years) (Lovgren *et al*., 2000); a metal known to have an issue involving development of a weak "alpha case" layer during porcelain firing. Thus, porcelain-Zirconia compatibility appears problematic in light of past experience with metal-ceramic systems.

Ceramic-ceramic compatibility is not easily characterized. All manufacturers appear to be using standard slow-heating dilatometry measurers of expansion coefficients (α) and thermal shock testing during product development. Most manufacturers provide veneering porcelains having a slight mismatch (Δα) between their porcelain and Zirconia, with the porcelain having approximately 1α unit ($\Delta L/L \times 10^{-6}\ K^{-1}$) lower than the Zirconia, which generally have an α in the range of $(10.5\text{-}11.0) \times 10^{-6}\ K^{-1}$. This approach is used for most metal-ceramic systems and non-Zirconia all-ceramic systems. Therefore, if a compatibility issue is occurring with Y-TZP it is likely not due to a simple thermal expansion coefficient mismatch between the bulk materials.

Crazing or chipping during function signals the presence or development of tensile stresses, likely associated with the Zirconia-porcelain interface. Since the origin of such stresses does not appear to be related to bilk thermal expansion/contraction mismatches, perhaps surface property changes are involved. Silicate glasses are known to be aggressive solvents towards refractory materials at high temperatures (Sandhage *et al*., 1990). Aluminum oxide has been shown to be a soluble in dental porcelains under firing conditions (Kelly, 1989). More recently both cerium and zirconium were shown to diffuse into a glass used to infiltrate a partially sintered Ce-TZP powder (Denry *et al*., 2008).

Depletion of stabilizing dopants (e.g., Y and Ce) might conceivably lead to local changes in unit cell tetragonality resulting in destabilizing of the tetragonal-phase or development of local thermal expansion anisotropy. If significant cubic- phase is present near grain-boundaries or triple-points, destabilization might result in cubic to metastable transformation with quite high local associated strains. Liquid silicate penetration of grain boundaries may be another consequence to consider, perhaps analogous to water penetration of Y-TZP at moderately elevated temperatures (Kobayashi *et al*., 1981). All manufacturers of porcelains for dental Y-TZP ceramic now provide “liner” materials, presumably to increase porcelain bonding as well as to provide some chroma and fluorescence. Although “bonding” does not appear to be at issue, perhaps these liners help assure wetting or have chemistries adjusted to reduce possible interactions with the Y-TZP. It does not appear that prostheses have needed to be replaced in any studies due to porcelain crazing or minor chipping.

Zirconia all-ceramic systems

Ceramic restorations, suitable for the anterior as well as the posterior region, simultaneously satisfying the demand for high strength, longevity, and esthetics, are an increasingly important field for the dental professional. Because of its outstanding mechanical properties and esthetics with a proven track record in other industrial areas, Zirconia is emerging in the dental industry. As the manufacturing method of choice, CAD/CAM is important for the dental laboratory; however, in the final analysis, the primary focus will be on the material properties and the clinical performance of the result of the CAD/CAM process - in this case, Zirconia crowns and bridges. This is especially true since some concepts do not require the acquisition of a CAD/CAM system at all.

Several companies are offering Zirconia materials in dentistry (Lava™, 3M™ ESPE™; Cercon® Smart Ceramics, Dentsply Ceramco®; Procera® Zirconia, Nobel Biocare™; Vita® InCeram® YZ, Vident™). Even though Zirconia can be chemically similar it is not necessarily the same, as previously explained. Bread is often chemically similar; however the color, consistency and taste can be very different. Many other factors outside of chemistry influence the final result including the order in which ingredients are mixed, the grain size or consistency of the mixture, and time and temperature used for preparing the material. Although the Zirconia ceramic is chemically similar, once processed, it can exhibit different mechanical and optical characteristics. Working with Zirconia, one can experience the differences in machinability (e.g. wet milling and dry milling) and in sintering (e.g. different sintering temperature). Several factors should be evaluated when selecting the proper material: (1) processing parameters for pre-sintered Zirconia since they may affect performance attributes; (2) differences in the Zirconia powder because they affect the strength/long-term stability and translucency of the restoration;

(3) the pressing condition and pressing method since they can affect the marginal fit, strength and translucency of the restoration (4) the pre-sintering conditions may affect the strength of the pre-sintered material and its mill ability (5) the material and method used for coloring the Zirconia because it can affect the marginal fit, strength and translucency of the material.

Although this parameters may be important at long-term, different studies show that, independently of the Zirconia material used the restorations produced can have the potential to withstand occlusal forces applied in the posterior region of the mouth and can therefore represent interesting alternatives for replacing porcelain-fused-to-metal restorations (Att *et al*., 2007; Pittayachawan *et al*., 2009)

A technique using either the traditional wax-up technique or by advanced Computer assisted design (CAD) using computer and special software provided by the manufacturers can be used to design the Y-TZP-based framework for crowns or FPDs. This designing software is unique and different from individual Y-TZP based manufacturers.

Y-TZP based frameworks are white in color. This can present clinical limitations for its use in the esthetic zone. To overcome this problem several all-ceramic systems have now the Zirconia framework for crown and bridges available in different shades. This is achieved by staining or coloring the framework before final sintering. Coloring is made by immersing or dipping, depending on the system, the framework into a salt solution before sintering, so that the zirconium oxide infiltrates with the liquid. This staining allows the achievement of the final shade from intaglio surface to external surface of veneering ceramic. Different studies evaluated the effect of such staining on the flexural strength of the Zirconia framework and concluded that there was no difference in flexural strength of uncolored and colored Y-TZP ceramic (Pittayachawan *et al*., 2006; Shah *et al*., 2008). However, a decrease in flexural strength at the higher salt concentrations can occur and is attributed to an increase in open porosity. The addition of shading pigments to Zirconia frameworks result in structural changes that require different surface treatment prior to veneering. To prevent delamination and chipping failures of Zirconia veneered restorations, careful selection of both framework and veneer ceramic materials, in addition to proper surface treatment, are essential for maintaining good bond strength (Aboushelib *et al*., 2008)

Tooth preparation guidelines are as comparable to metal-fused to ceramic crowns and bridges preparations. It is advisable to use manufacturer's recommendation and to use advised preparation kit. The axial reduction of approximately 1.2 to 1.5 mm, occlusal reduction should be 1.5 to 2.0 mm. The axial taper of crown preparation should be of 5 to 6 degrees. All the sharp edges of the crown need to be smoothened. The gingival finish line should be uniform and can be at the gingival margin or 0.5 mm sub gingival. Recommended cervical finish line is 0.8 or 1.2 mm deep chamfer or shoulder with rounded internal angle. Recent publications recommend that, for crown the chamfer or shoulder finishing line and for bridges the shoulders with rounded internal angle type design to be used for a favorable distribution of occlusal stresses to abutment teeth during function (Komine *et al*., 2007; Reich *et al*., 2008; Beuer *et al*., 2008). However, it was found that the finish line design seemingly wielded no influence on marginal adaptation of ZrO_2 ceramic copings and crowns. It was also observed that the marginal and internal adaptation values were all within the clinically acceptable range. The accuracy of fit achieved by different Zirconia systems was shown by another study within the range of clinical acceptability (Gonzalo *et al*., 2008).

Full coverage Y-TZP based restorations can be cemented using either conventional cements or be bonded using adhesive cementation. Studies show that the use of composite resin cements with a bonding agent do not yield higher coping retention compared to other cements (Ernst et al., 2005; Palacios et al., 2006). Before cementation the inner surface of the Zirconia frameworks should be sandblasted since it was demonstrated that sandblasting may provide a powerful technique for strengthening Y-TZP in clinical practice. (Guazzato *et al*., 2005; Kosmac *et al*., 1999; Kosmac *et al*., 2000)

Procera® AllZircon system

The CAD/CAM technology employed to produce the Procera® AllCeram alumina crown, is now used for the fabrication of a densely sintered high-purity Zirconia framework. The Y-TZP Procera® (Procera®, Nobel Biocare™ AB, Sweden) material can be used to fabricate copings for crowns, one-piece or multi-unit all-ceramic fixed partial dentures, and ceramic implant structures and abutments. These ceramic units are then combined with low fusing veneering porcelain that creates the anatomic form of the restoration and the occlusal morphology. The technique used for fabricating these ceramic restorations is the same described previously for the Procera® system. The individual Zirconia oxide substructures are manufactured by Nobel Biocare™ at their facilities and then returned to the laboratory of origin for veneering porcelain application and final finishing.

The Procera® system consists of a computer-controlled design station in the dental laboratory that is joined through a modem link to the production facility in Sweden, where the substructure is fabricated. At the design station, a scanning device controlled by a personal computer maps the surface of the die/s of the prepared tooth/teeth and ridge in case of a fixed partial denture. When the scanning is completed, the data is evaluated for completeness and displayed on the monitor computer as a three-dimensional image. Marking of the finish line on the three-dimensional plots is the next step. The design of the framework on the screen, e.g. the insertion of a pontic (from a library) or the design/modeling of the connections is done with the keyboard, mouse and software support. The next step is to establish the thickness of the substructure to be fabricated. The emergence angle of the tooth is selected, and the relief space for the luting agent is automatically established by a computer algorithm. When the design of the substructure has been completed, the file is saved in the computer and is ready for transmission via modem to the production station.

The data of the preparation and the design of the substructures are transferred via modem to the facility production in Sweden. At this point the fabrication of single copings and fixed partial dentures frameworks differs. In the former, the sintering shrinkage is compensated through the computerized milling of an enlarged die. After that, zirconium oxide powder is compacted under pressure onto the die and fired. In the latter, the frameworks are virtually enlarged to balance the sintering shrinkage and milled out of a solid piece of high-purity zirconium oxide, which is then sintered (Polack, 2006). The color of the dense sintered Zirconia is traditionally white; however the manufacturer has now available Zirconia substructures in different shades for copings. The Zirconia structure is examined for quality control and sent by mail to the dental laboratory, where the ceramist finalizes the restoration by the addition of the veneering porcelain. Available data demonstrates superior mechanical fracture strength of the Zirconia material when compared to alumina (Snyder *et al*., 2005). Margins accuracy is acceptable independently of the method used and is acceptable for clinical use (Gonzalo *et al*., 2008; Beuer *et al*., 2009). No long-term clinical trials are available for this material until this date. The only study available made in private practice environment revealed that most crowns studied (78%) were placed on premolars and molars areas. The clinical outcome of the crowns was favorable. No Zirconia core fractured and no caries were observed on the abutment teeth. Some types of complication were recorded for 32 (16%) crowns or abutment teeth. The most severe complications, in total 12 restorations (6%), were recorded as failures: abutment tooth was extracted (5), remake of crown due to lost retention (4), veneer fracture (2) and persistent pain (1). The criteria for 25 crowns were rated favorably, and patient satisfaction with the Zirconia crowns was in general high. The authors concluded that porcelain-veneered Zirconia crowns showed good clinical results, were well accepted by the patients, and only few complications were reported over the 3-year follow-up period. (Ortorp *et al*., 2009)

LAVA™ system

The Lava™ All-Ceramic System from 3M™ ESPE™ comprises also a CAD/CAM procedure for the fabrication of all-ceramic crowns and fixed partial dentures for anterior and posterior applications. The framework ceramic consists of Zirconia supplemented by a specially designed veneer ceramic. The frameworks are fabricated using CAD/CAM procedures (scanning, computer-aided framework design and milling) from presintered Zirconia blanks; the size of which are increased to compensate for shrinkage during sintering in a special high-temperature furnace.

The unit consists of the non-contact, optical scanning system Lava™ Scan a PC with monitor and the Lava™ CAD software. When the sectioned model has been positioned in the scanner, individual preparations and the ridge are recorded automatically and displayed on the monitor as a three-dimensional image (recording of the model situation including preparations, gingiva and occlusal record). The preparation margins are scanned and displayed automatically. The design of the framework on the screen, e.g. the insertion of a pontic (from a library) or the design/modeling of the connections is done with the keyboard, mouse and software support. The data is then transferred to the Lava™ Form milling unit for calculation of the milling path. The 3D shape is milled from a pre-sintered ZrO_2 blank using hard metal tools.

Manual finishing can be carried out before sintering takes place. The coloring of the frameworks also takes place before the sintering process according to the prescribed shade (7 possible shades, keyed to Vita® Classic). The fully-automated, monitored sintering process then takes place with no manual intervention in a special furnace, the Lava™ Therm and compensates the volume shrinkage of the sintering process. The coefficient of thermal expansion (CTE) of the specially developed, integrated overlay or porcelain ceramic system has been matched closed (-0.2 ppm) to that of Zirconia. The 16-shade system is based on the Vita-Lumin range. The biaxial flexural strength of the material was reported to be 1100MPa (Pittayachawan *et al*., 2007. The marginal and internal fit of all-ceramic three-unit fixed partial denture fabricated by CAD/CAM technique showed satisfactory results (Beuer *et al*., 2009). Long term clinical results and survival rates for Lava™ Zirconia crowns are insufficient.

Cercon® smart ceramic system

The Cercon® smart ceramic system (Dentsply International, Inc., York, PA, USA) utilizes the conventional wax pattern method for designing infrastructure for anterior and posterior crowns and fixed partial dentures up to 2 pontics with specific thickness. An optical laser scans the wax pattern into a computer, where it is virtually enlarged and sintered. The collected data is then transferred to the Computer Aided Manufacturing (CAM) unit where the framework is automated milled from partially sintered Y-TZP blanks. The partially sintered blanks used compensated by increasing the framework size the sintering shrinkage of 20 to 25 %. The Cercon® system uses different types of CAD software with different designing options and features. The Zirconia structure is examined for quality control and finalized the restoration by the addition of the veneering porcelain.

Mean gap dimensions for marginal openings, internal adaptation, and precision of fit for this crown are reported to be within the clinically acceptable range (Komine *et al*., 2007). Although the fracture resistance may be different depending on the cement used, the values obtained are well above the maximum masticatory forces and around 1140MPa (Yilmaz *et al*., 2007). The clinical success of these crowns is not yet well documented.

In-Ceram® 2000 Vita® YZ CUBES

In-Ceram® 2000 Vita® YZ CUBES (YZ Zirconia) (Vita®, Zahnfabrick, Germany) is a newly developed yttria-stabilized Zirconia ceramic containing approximately 95% zirconium oxide (ZrO_2) and 5% yttrium oxide (Y_2O_3). The material is marketed as presintered Y-TZP blocks used together with a computer-aided design/computer-assisted manufacture (CAD/CAM) system (Cerec® 3/InLab, Sirona Dental Systems Inc., NY, USA). The blocks are available in different sizes depending on the number of elements to be prepared. The prepared model and dies are scanned with a special designed scan and the information is displayed on a computer where the framework is planned. The ceramic blank is positioned in the milling machine and the information transferred from the computer. The same similar enlarging and sintering process is needed in this system to fabricate single copings and fixed partial denture frameworks of up to 2 pontics. The milled presintered ceramic blank is then fired in a high-temperature furnace to complete sintering. The restoration is finalized by the ceramist adding veneer porcelain and pigments. Flexural strength of the material has been reported to be around 1100 MPa (Chai *et al*., 2007). The marginal gap values and internal adaptation of this material were reported to be within satisfactory acceptable clinical range (Att *et al*., 2009) .The clinical application appears sufficiently promising although no long-term clinical studies were reported.

DCS- Smart-Fit system

The DCS Smart-Fit (DCS Dental, Bien Air, Switzerland) is a CAD/CAM system that instead of employing partially sintered Y-TZP milling blocks utilizes fully sintered Y-TZP under hot isostatic pressure called DC-Zirkon®. This results in an extremely hard and dense ceramic that requires a mill time of around 2 to 4 hours for a coping (McLaren *et al*., 2005).

The working model tooth/teeth preparations and die/s are measured and digitized using a laser scan and the information is transferred to a computer. The restoration is then designed and calculated using CAD software. The resulting control and milling data are forwarded to the milling machine were the framework is prepared from different shaped bocks, depending on the framework that is necessary. The manual finishing operations are performed and the framework is veneered by the ceramist using conventional procedures. The system can be used to fabricate anterior and posterior crowns and fixed partial dentures. Systems that use fully sintered blanks (HIP) take longer time for milling due to increased hardness of blank. Studies show superior marginal fit by virtue of not having sintering shrinkage (Denry *et al*., 2008). However, the results of margin accuracy in all system studied are within clinical acceptance. The mean strength of the material was reported to be around 1120 MPa (Chai *et al*., 2009). The same study reveled contradictory and unexpected results, when compared to other studies with concern to the strength of different Zirconia based materials. In this study the strength of Lava™ was significantly lower than that of DC-Zirkon® but significantly higher than that of Cercon®. The authors concluded that the probability of failure of DC-Zirkon® was significantly lower than that of Lava and Cercon. The clinical behavior of Zirconia-based fixed partial dentures made of DC-Zirkon® at 3-year demonstrate satisfactory results with a sufficient success rate under clinical conditions (Tinschert *et al*., 2008).

Veneering Ceramics

A strengthened ceramic can be used as the sole material or as the core substrate for making an all-ceramic crown, inlay, onlay, veneer, or fixed partial denture restoration (Anusavice, 1996). Two development directions are recognized in efforts to improve the strength characteristics of all-ceramic systems. The first is directed toward enhancing the strength of the core materials, while the second focuses on improving processing techniques designed to produce more homogeneous ceramic materials and veneering porcelains. Often both directions are used in the development of a new all-ceramic restorative system with improve strength. Ceramic core materials with new chemical compositions have been developed for use with processing methods combining different technologies (Dong *et al*., 1992; Anderson *et al*., 1993; Probster *et al*., 1992; Luthardt *et al*., 1999).

Yttria stabilized Zirconia provides a sufficient mechanical strength to be used in frameworks for all-ceramic crowns and fixed partial dentures (Lüthy *et al*., 2005). However, for esthetical reasons, these frameworks have to be veneered with an appropriate veneering ceramic. In clinical applications, the veneering ceramic revealed to be the weakest link in such reconstructions (Sailer *et al*., 2006; Sailer *et al*., 2007; Vult von Steyern *et al*., 2005). Chipping of the veneer is described to be the most frequent reason for failure with a failure rate of 15.2% after service time of 35.1± 13.8 months (Seiler *et al*., 2007).

Among other reasons failure of a veneer may be caused by insufficient bond-strength, excessive tensile stress due to a thermal mismatch between veneer and framework or excessive load due to premature contacts (al-Sheri *et al*., 1996; Isgro *et al*., 2003; De Jager *et al*., 2005; Aboushelib *et al*., 2005; Drummond *et al*., 2000). The bond strength was intensively investigated (Luthard *et al*., 1999; Aboushelib *et al*., 2006; Al-Dohan *et al*., 2004). It revealed to be in the range of that measured with metal-ceramic systems. Furthermore, it was demonstrated that independently of the surface treatment the bond strength of veneering ceramics to Zirconia was similar to metal ceramic systems (Fischer *et al.*, 2008). The tensile stress in the veneering ceramic is established during cooling after firing, when an unequal thermal contraction of both layers happens. The coefficients of thermal expansion should be adjusted in a way that during cooling a slight compression of the veneering ceramic occurs to enhance its strength (Bagby *et al.*, 1990). In the metal-ceramic systems, excessive thermal creep of the alloy, i.e. plastic flow, may compensate stress to some extent especially if a high gold alloy is used (Anusavice *et al.*, 1987). In all-ceramic systems, the ceramic framework is rigid and does not yield to the stress induced by a thermal mismatch to that extent. Therefore, the risk of destructive stress formed in the veneer layer might be higher in all-ceramic systems and thus would require a high mechanical strength for veneering materials for all-ceramic systems. Hence, the strength of the veneering ceramic is a crucial parameter for the clinical long-term success. For metal-ceramic restorations failure rates after 5 years, caused by chipping of the veneer are reported to be 0.4% for single crowns and 2.9% for fixed partial dentures. For this reason, veneering ceramics for Zirconia should at least show a flexural strength, which is similar to that of veneering ceramics for alloys. In fact, this similarity was demonstrated by Fisher and co-workers using a three-point flexural strength test (Fischer *et al.*, 2008).

Ceramic veneering materials do not show the strength one would expect from their molecular structure. Small defects, for example, scratches, can be found on the surface of almost any material and these scratches are the reason for the lower strength (Guazzato *et al.*, 2003). Such surface defects are comparable to sharp cuts with crack tips that can be as narrow as the distance between two atoms. The stress concentration that results from the defect results in a local increase in tensile stress. However, the theoretical strength of the material is based on the assumption that there is an even distribution of stress in the entire structure. If the tensile stress exceeds the strength limit at the tip of a defect, the chemical bond breaks at this tip, and a crack stars propagating. The tensile stress at the tip of the crack remains until the crack has propagated through the entire material or has reached another crack, a pore, or a crystalline particle, so that compression occurs and the stresses are distributed. This phenomenon explains why some materials fail at pressures far bellow their theoretical strength values (Isgro *et al*., 2003; Albakry *et al*., 2004; White *et al*., 2005; Denry *et al*., 2008).

The failure of ceramics and their low tensile values can be explained on the basis of how stress concentrations are generated at surface inclusions. Under certain conditions inclusions can also initiate crack within the material. Since ceramics have no other stress distribution mechanism available to deal with tensile loads besides crack growth, crack can continue to grow under low-pressure conditions through the entire material. Therefore the flexural strength of ceramics and glass is essential lower than their compressive strength (Anusavice *et al*., 1992; Marchack *et al*., 2008).

Complex stresses develop in the mouth. The maximum pressure appears at the surface of the restoration. Therefore, surface inclusion is especially important in judging the strength of a ceramic. Removing or reducing the number of surface inclusions can result in a considerable improvement of the fracture resistance. This is one of the reasons why it is necessary to polish and or glaze dental ceramics. The fracture strength of the material can be improved in two ways: (1) through introducing a compressive stress within the material surface, and (2) through interrupting crack propagation in the material. The fracture strength of the ceramic veneering material can be improved by intrinsic strengthening mechanisms as explained previously or by minimizing failures.

Several methods can be employed in order to minimize failures. They include: (1) use of harder and more stable ceramics to resist higher pressure without crack formation; (2) limitation of sharp edges induced during preparation which cause areas of high tensile stress within the restoration, particularly when these regions have to resist bending forces; (3) reduction of inclusions located at the outer ceramic margins and surface of a crown which lead to high local tensile stress. For rough ceramic surfaces a self-glazing procedure is recommended since strength increases in contrast to unglazed ceramics. The glaze also decreases the risk of crack propagation. If the glaze is removed by grinding, the strength is reduced by half in comparison to a surface with intact glaze layer. As studies have shown, certain ceramics with highly polished surfaces have comparable strength to similar ceramics that are both polished and glazed Polishing has been advocated to strength ceramics (Giordano *et al*., 1995; Chen *et al*., 1999). This observation is of clinical importance, because it is common practice to adjust the occlusion by grinding the ceramic surface after the ceramic crown has been cemented. By doing so, the glaze is removed and a relatively rough surface exposed that weakens the ceramic significantly. In order to cope with this problem, the surface should be polished with disks, cups, tips and diamond polishing paste. A smooth surface also reduces the abrasion on the opposing teeth; (4) minimizing the number of firing cycles. The goal of ceramic firings is to achieve denser sintering of the power particles and to manufacture a relatively, smooth, glassy layer on the surface. In some cases, a layer of color is fired to adapt to the color of the natural teeth or to simulate color lines or craze lines. During firing, different chemical reactions occur of which the most important one is the increase in concentration of crystalline leucite crystals in the ceramics. Leucite is a crystal that has a high thermal expansion. The leucite crystal permanently influences the thermal expansion of the ceramic. Several firing steps clearly increase the leucite content, which could cause a disparity in the coefficients of thermal expansion between the ceramic and the metal or ceramic substructure. During the cooling phase, this can lead to tension that finally causes crack in the ceramic; (5) laboratory control of cooling. Proper cooling of the ceramic restoration from the firing temperature to room temperature is very important, particularly if the coefficient of thermal expansion of the ceramic is higher that of the metal or ceramic substructure. In this case, faster cooling is desirable in order to minimize tensile stresses that arise during cooling.

If a more stable material is used as a core in an all-ceramic crown and if there is a stable bond with the outer ceramic, cracks can occur only if the stronger inner material is deformed or broken. The fracture risk of the entire ceramic can be reduced to a minimum if the process is carried out properly and if the physical properties of the ceramic and the substructure are well balanced. Current processing technologies cannot make Zirconia frameworks as translucent as natural teeth, nor can they provide internal shade characterization or facilitate customized shading. Therefore, Zirconia core or frameworks must be veneered with porcelain to achieve acceptable esthetics. A series of studies on layered all-ceramic structures showed that a veneer of a relative weak porcelain may result in failure at low loads should the porcelain veneer be placed in tension. (White *et al*., 1994; White *et al*., 1996; Kelly *et al*., 1995; Lang *et al*., 2001).

Although clinical failure of all-ceramic restorations is a very complex process involving patient variables, dynamic loads, restoration geometry, material properties, fatigue phenomena, and multiple failure modes, in vitro models may help to elucidate mechanical parameters known to influence fracture by tensile failure (Kelly, 1995; Kelly *et al*., 1999; White, 1993; White *et al*., 1997. Tensile failure is believed to be the dominant clinical failure mechanism of all-ceramic restorations (Kelly *et al*., 1989; Kelly *et al*., 1990; Thompson *et al*., 1994).

In a recent review of the use of contact testing in the characterization and design of all-ceramic crown-like layer structure, Lawn and co-workers have pointed out that the lifetime of a restoration can be improved by increasing the strength of the core material and avoiding the creation of spurious flaws in the surface (Lawn *et al*., 2001). All-ceramic materials are subjected to different fabrication procedures in the laboratory, and sometimes must be adjusted clinically to allow either proper fitting or occlusion. The processing procedures and/or clinical adjustments are more likely to initiate subcritical flaws or large defects, which upon clinical loading and/or presence of moisture may grow to a critical situation leading to catastrophic failure. In addition, different finishing procedures can cause various stress concentrations and consequently may be accompanied by a reduction in strength (Jager *et al*., 2000). These flaws may be introduced at different stage of the fabrication of the restoration. The most common steps of the fabrication of all-ceramic restorations are grinding, sandblasting, polishing and heat treatment. Previous studies indicate that the influence of these steps on the ultimate strength of a restoration is contradictory and related to the nature of the material investigated and the operation conditions (Denry, 2008).

Grinding is commonly involved during machining of an all-ceramic framework and adjustments by ceramist and dentist to improve occlusion and proper fitting. The effect of grinding on the surface of ceramic and its mechanical properties is contradictory. Giordano and co-workers have shown that grinding induce flaws of a depth of 30-40μm in feldspathic porcelain and are responsible for up to 80% strength degradation (Giordano *et al*., 1995). However, Guazzato and co-workers showed that if grinding is followed by heat treatment the ultimate flexural strength degradation could be avoided (Guazzato *et al*., 2003). On the other hand, grinding increases the strength of phase transforming ceramics, such as Zirconia and may increase the strength of glass-ceramics when dimensions of the initial cracks are greater than those induced by grinding (Samuel *et al*., 1989; Green *et al*., 1983).

This apparent contradiction may be clarified by considering the effect of grinding on the surface of a ceramic. The effect of the diamond grains of the grinding disk has been compared to a number of closely spaced indentations (Lange *et al*., 1983; Sines *et al*., 1983). Each single indentation dislocates a corresponding volume of material, creating radial compressive stresses. The overlapping of plastically deformed regions generated by neighbouring indentations creates a layer of uniform compressive stresses (Lange *et al*., 1983). These stresses become tensile within a depth of several microns below the surface and then gradually decline to zero at greater depth (Samuel *et al*., 1989). The near-surface compressive residual stresses contribute to increase the flexural strength of the material by reducing the stress intensity factor of a surface crack to an applied stress, whereas opposite action is expected by underlying layer of tensile stress (Lange *et al*., 1983). Mecholsky and co-workers have investigated the influence of grinding orientation on flexural strength of alumina specimens and have shown that grinding always generates two steps of flaws with different geometries (one parallel to the grinding orientation and the other perpendicular) (Mecholsky *et al*., 1977). In addition, grinding perpendicular to the tensile axis generates flaws that are 60% deeper with slightly greater stress intensity factor than flaws resulting from parallel grinding. A greater strength reduction is therefore expected from testing with the tensile axis perpendicular to the grinding direction (Mecholsky *et al*., 1977). However, different studies have shown that orientation of grinding in respect of the direction of the tensile stress do not influence the ultimate tensile strength (Guazzato *et al*., 2003; Albakry *et al*., 2003). The influence of grinding on the ultimate strength of a ceramic can therefore be explained by tacking into account factors that may alter the combined effect of the surface flaws and the residual stress layers. Some of these factors are: the magnitude of the residual stress (which is also related to the composition and microstructure of the ceramic); the ratio of the crack length to surface compressive layer depth (Lange *et al*., 1983); the effective size of grinding particles (Johnson-Walls *et al*., 1986); the dimensions of the pre-existing flaws (Tuan *et al*., 1998); and the orientation of grinding (Mecholsky *et al*., 1977).

The influence of grinding on the flexural strength of Zirconia ceramics is also contradictory and related to the volume percentage of transformed Zirconia, which in turn depends on the metastability of the tetragonal to monoclinic phase transformation, the grinding severity and the locally developed temperatures as previously explained (Kosmac *et al*., 1999; Kosmac *et al*., 2000; Gupta, 1980; Green, 1983; Guazzato *et al*., 2005). In Zirconia grinding has been recommended to create a surface region of compressive stresses that increases the mean flexural strength of Zirconia ceramics (Gupta, 1980; Guazzato *et al*., 2005). Swain and co-workers showed that hand grinding is more effective than lapper-machine grinding in inducing the tetragonal to monoclinic transformation (Swain *et al*., 1989). They demonstrated that in the case of machine grinding the local development of temperatures exceeded the monoclinic to tetragonal transformation temperature, causing the reverse monoclinic to tetragonal transformation. In this instance, the deep defects introduced by grinding are no longer counteracted by the transformation-induced compressive stresses and act as stress concentrators, lowering the mean flexural strength of the ceramic. More recently, Xu and co-workers reported an improvement in strength of Y-TZP upon fine grinding with 25 μm grit size diamond wheels, whereas coarser grinding resulted in strength reduction (Xu *et al*., 1997). However, this study did not correlate strength to the relative amount of transformed monoclinic phase obtained upon surface treatment. The effect of such alterations in bi-layered specimens, Zirconia/veneering porcelain has not yet been established, and would wonder if such alterations would have any significance.

Sandblasting is used to remove excess glass from glass-infiltrated core materials and also recommended to create micro-retentive surface to increase the adhesion of the cement of any metal or ceramic inner surface restoration (Kern *et al*., 1994; Blixt *et al*., 2000). Due to the brittleness and elasticity of glass and ceramics, the impact of the sand particles on the surface of the materials creates stresses that are sufficient to generate cracks, even if the impact energy is low (Guazzato *et al*., 2003). The coalescence of two or more cracks and the impact by another particle or the elastic response of the ceramic causes the removal of small portions of the material (Guazzato *et al*., 2003). Kosmac and co-workers investigated the influence of sandblasting, wet and dry grinding (carried out by hand with a 150 μm grit size diamond burr mounted on a high-speed hand piece) in dry pressed and sintered 3 mol% Y_2O_3 TZP (Kosmac *et al*., 1999). They showed that sandblasting was more effective than grinding in inducing the tetragonal to monoclinic transformation and therefore, increasing the flexural strength of the ceramic. On the basis of the study conducted by Swain and Kosmac and co-workers inferred that locally developed temperature must have exceeded the monoclinic to tetragonal transformation temperature. In this instance, the deep defects introduced by grinding were no longer counteracted by the transformation-induced compressive stresses and acted as stress concentrators, lowering the mean flexural strength of the ceramic. On the other hand, sandblasting was described as a process able to induce transformation without developing high temperatures or creating severe surface damage, and therefore, strengthening the material. Guazzato and co-workers in a similar study concluded that sandblasting and grinding may be recommended to increase the strength of dental Y-TZP, provides they are not followed by heat treatment (Guazzato *et al*., 2005). They also suggested that fine polishing might remove the layer of compressive stresses and therefore, lower the mean flexural strength of the material. Once more, the effect of sandblasting on the strength of bi-layered specimens it’s yet to be determined. However the influence that it may have on the bond strength between the core material and the veneering porcelain was already demonstrated to be unnecessary (Fischer *et al*., 2008). The results from these studies suggest that sandblasting the inner surface of a Zirconia framework before cementing may have the potential of increasing the flexural strength of the restoration since it increases the flexural strength of the Zirconia core. In addition, air abrasion combined with pretreatment with a metal primer seems to be an appropriate method for improving bond strength between Zirconia based restorations and abutments (Lindgren *et al*., 2008).

Polishing has been advocated to strength ceramics. (Giordano *et al*., 1995; Chen *et al*., 1999). The strengthening mechanisms are due to the generation of compressive stresses (as seen for grinding) and removal of part of the pre-existing flaws and radial cracks (Johnson-Walls *et al*., 1986). However, if the dimensions of the radial cracks pass the limits of the compressive layer or the flaws are not entirely removed in their depth, the polishing procedure may be inconsequent to eliminate the damaged zone and thus increase the strength of the ceramic material. Studies results from the influence of polishing on the surface of ceramic materials are contradictory. In one study, polishing alone did not strengthen the ceramic when compared to untreated, sandblasted and ground ceramic groups (Guazzato *et al*., 2003). However, in another study by the same authors, polishing produced significantly highest mean flexural strength values when compared to untreated, sandblasted and ground groups (Albakry *et al*., 2003). It has been speculated that more than 50% strength increase can be achieved after fine polishing (O'Brien, 2002). The ability of polishing to eliminate various defects and flaws from treated surface is considered responsible for such strength increment (Albakry *et al*., 2003). The high physical hardness of many ceramic materials makes surface polishing a difficult task and the development of microcracks is not easily avoidable. The increment in strength can be accomplished by polishing with a diamond paste material since it produces the smoothest surface and the highest strength tensile values when compared to diamond polishing wheels (Guazzato *et al*., 2004). Moreover, skills of individual dental ceramists as well as the adherence to the recommendations of a specific dental material's manufacturer can, to a great extent, influence the mechanical performance of all-ceramic materials (Chen *et al*., 1999). The influence of polishing in the flexural strength of Zirconia based restorations has not yet been determined. Available results for polishing have only been presented for Zirconia ceramic studied alone (Guazzato *et al*., 2005; Kosmac *et al*., 2000). These results revealed that polishing had little effect upon mechanical strengthening of the Y-TZP ceramics. In some instances, polishing may remove the layer of compressive stress and therefore, lower the mean flexural strength of Zirconia. It was demonstrated by the authors that polishing had neither been able to induce transformation, nor to remove all of the strength- determining grinding-induced flaws. They speculated that further polishing might minimise the size of flaws and result in greater flexural strength.

After the fabrication of the Zirconia framework, the core material is veneered with feldspathic veneering porcelain by firing and glazing at a temperature of approximately 900°C. Glazing can be either the application of a low fusing glass overcoat or auto glazing which is based on firing for a certain time, held at the maximum ceramic temperature. Auto glazing and/or the application of glazing material after grinding is believed to increase the strength of ceramic materials by reducing the depth and/or sharpness of critical flaws (Baharav *et al*., 1999). The overglazing surface treatment is a routine procedure in a dental laboratory. It produces crowns with smooth and shiny surfaces and has a positive effect on the biaxial flexural strength of ceramic materials (McLean, 1979). However, this effect is still uncertain (Fairhurst *et al*., 1992). Several reports in the literature show that auto glazing has no effect on strength (Anusavice *et al*., 1989; Fairhurst *et al*., 1992; Griggs *et al*., 1996; Denry *et al*., 1999; Albakry *et al*., 2003). This is attributed to the fact that heat treatment after polishing or grinding may degrade strength, which is though to be a result of realising compressive stresses that normally develop during polishing or grinding. Conversely, other studies revealed that heat treatment procedure should always follow grinding and polishing procedures in order to avoid strength degradation of the ceramic material, and that the overglazed surface treatment increases the strength of ceramic core and veneering porcelain materials (Isgro *et al*., 2003; Guazzato *et al*., 2003). The term “overglazing” describes the firing of a low-fusing colourless glass on the ceramic core or veneering porcelain. This thin layer of about 4 μm of glass, produced after 60 seconds of hold time at the final temperature reduces the size of flaws present on the surface (probably introduced during the fabrication or clinical adjustment of the restoration), thus increasing the strength of the materials. Furthermore it is used to provide large surface compression, which strengthens the ceramic body.

While much research has been conducted to assess the strength of traditional dental porcelain materials and Zirconia based frameworks very little information has been reported concerning the relative strength of many of the ceramic materials already in clinical applications. The precision of fit and biocompatibility of Zirconia crowns have been studied and found to be excellent for multiple dental applications (Luthardt *et al*., 1999; Piconi *et al*., 1999). However, little data exists regarding the strength and mode of fracture, of dental veneering porcelains used in conjunction with Zirconia based structures (White *et al*., 2005; Aboushelib *et al*., 2006; Fischer *et al*., 2008).

The effect of processing procedures, polishing, grinding and glazing on the mechanical properties of some dental materials has been studied by many investigators (Bhrama *et al*., 2002, Giordano *et al*., 1994; Campbell *et al*., 1989; Rosentiel *et al*., 1999; Anusavice, 1991, Giordano *et al*., 1995; Fairhurst *et al*., 1992; Chu *et al*., 2000; Williamson *et al*., 1996; Mecholsky *et al*., 1977; Kosmac *et al*., 1999; Brackett *et al*., 1989; Griggs *et al*., 1996; Kitazaki *et al.*, 2001; Haharav *et al*., 1999; Denry *et al*., 1999; Anusavice *et al*., 1989; Albakry *et al*., 2003; Isgro *et al*., 2003; Guazzato *et al*., 2003; Guazzato *et al*., 2004). However, there is still controversy concerning the most suitable method that can produce a smooth and strong surface (Williamson *et al*., 1996). The purpose of this study is to contribute for the explanation and resolution of such a common clinical problem.

Fracture mechanism/Mode of fracture of dental ceramics

The fracture mechanism of ceramics and metals are different due to their different structure and bonding. Covalent and ionic bonds in ceramics are associated with large inter-atomic forces and hence a strong resistance to plastic deformation as compared with metals. Ceramics fail because of crack propagation at a critical strain of 0.1%. Therefore increases in strength can be achieved by an increase in the elastic modulus.

External loads will cause stress concentrations at a crack tip instead of relaxation by plastic flow (Kvam *et al*., 1991). Even within the all-ceramic systems fracture behavior of the restorations is different. It depends on the bonding and interaction between different ceramic layers and the occlusal loading rate and direction. Conservatively, restorations in the mouth should withstand masticatory force of 200 N and more than 10^7 cycles at contacts between opposing cusps of characteristic radii of 2 to 4 mm (Anusavice, 1989; Craig, 2008).

To predict the fracture modes and fracture resistance of bilayered ceramic restorations with different designs, one should perform analyses using a generalized model. There are several factors that are associated with the stress state created in dental ceramic restorations. These include: (1) thickness of ceramic layers; (2) mechanical properties of ceramics; (3) elastic modulus of supporting substrate materials; (4) direction, magnitude, and frequency of applied loads; (5) size and location of occlusal contact areas; (6) residual stresses induced by processing; (7) restoration-cement interfacial defects; and (8) environmental effects (Wakabayashi *et al*., 2000).

Dental structures are essentially layered composites. This is true of both natural teeth (enamel/dentin) and restorations such as crowns, inlays, onlays, veneers and fixed partial dentures (Claus, 1990; Probster *et al*., 1992; Probster, 1993; Rinke *et al*., 1995; Hornberger *et al*., 1996; Wolf *et al*., 1996; Kelly, 1997). The most esthetic restorations usually consist of at least one ceramic component, with net outer thickness ranging from 1.5 mm down to 0.5 mm or less near the margins, on a soft dentin interior from 1 to 4 mm thick. Sometimes there is a relatively stiff intervening core, such as alumina or Zirconia, ideally 1 mm thick but again sometimes less. Often there is a thin underlying "bond" layer, e.g., dento-enamel junction (DEJ) in natural teeth or luting cement in crowns. As with many other biomechanical systems, the properties of layered structures can be superior to those of their constituent material components.

High masticatory forces may induce fracture or deformation in the dental restorations, either of which can lead to premature failure. Tooth contacts can be closely simulated by indentation with spheres-the so-called Hertzian contact tests (Peterson *et al*., 1998a). Hertzian indentation testing is a method used for evaluating the role of microstructure in the mechanical response of dental ceramics. This test uses a hard sphere that is mounted on the underside of the crosshead on the mechanical loading machine. During the testing crosshead is lowered until the sphere is brought into contact with the specimen and the load is then increased to a peak value. A major advantage of Hertzian indentation over more traditional fracture methodologies is that it emulates the loading condition experienced by dental restorations. In ideally brittle materials contact failure occurs by the growth of a single macroscopic tensile cone crack (“brittle” mode), in tougher ceramics, failure occurs by evolution of a diffuse subsurface zone of microscopic shear cracks or faults (quasi-plastic” mode) The quasi-plasticity is macroscopically analogous to the yield zones that occur beneath contacts in metals. Microscopically the shear fault mechanism differs fundamentally from the dislocation motion that characterizes flow in metallic structures (Lawn *et al*., 1994). The Hertzian test has been especially valuable in demonstrating the critical role of microstructure in these two entirely different damage modes. Experimentally the test is very simple and requires only a hard ball bearing as an indenter. Yet it is uniquely powerful in the insights it provides into the damage modes. Moreover, because it samples damage in the short-crack region, it bears closely on microstructure-sensitive properties like strength and wear resistance. Most importantly the test simulates oral-loading conditions more compellingly than conventional mechanical tests and is characterized by variables that have direct clinical relevance, notably contact (occlusal) load and indenter (cuspal) radius.

Peterson and co-workers (Peterson *et al*., 1998a,b) study, where they presented data on the role of microstructure on Hertzian contact damage, responses of selected generic ceramic systems for dental applications. The ceramic systems studied included mica glass-ceramics, glass-infiltrated aluminum oxide, feldspathic porcelain, and transformable Zirconia. Even though their goal was to make a point on the importance of the test and not on the materials differences they found that aluminum oxide and Zirconia are much more damage tolerant. They concluded that in order to develop superior materials a more thorough understanding of both the deformation and fracture modes, especially in the context of microstructure is required. Ultimately a proper understanding of microstructure properties relationships will require appropriate modeling of the damage processes. Modeling has begun on contact quasi-plasticity at the macroscopic level by finite element modeling and at the microscopic level in terms of shear fault micro-mechanics. Together with such modeling, and along with relevant clinical data, the Hertzian contact test presents itself as a powerful methodology for the development of the next generation of dental ceramics.

In monolithic ceramics, tensile stresses generate macroscopic cone-like cracks around the contact; shear stresses generate diffuse "quasi-plastic" damage zones, consisting of distributed grain-localized failures, beneath the contact (Lawn *et al*., 1994). The dominant damage mode in any given material is dictated by the microstructure. Fine microstructures with minimal internal weakness tend to exhibit macroscopic cracks; coarse microstructures with enhanced internal weakness tend to exhibit quasi-plastic zones. Both cracks and quasi-plasticity can lead to degradation of properties, and ultimately compromise the useful lifetimes of restorative structures, in different ways (Lawn *et al*., 1998; Peterson *et al*., 1998b). The two modes may be interactive-plasticity can enhance or inhibit fractures by redistributing tensile stresses. Similar studies were conducted to analyze contact damage in Zirconia (Y-TZP) (Zhou *et al*., 2007). The results suggested that the cyclic contact loading induce both plastic damage and tetragonal to-monoclinic phase transformation in the Zirconia (Y-TZP) that can lead to significant degradation in long-term strength.

Several studies have been reported on Hertzian contact stress fields in bilayered structures with a brittle outer layer on either a hard or soft substrate material. Systems studied include glass/glass-ceramics (Wuttiphan *et al*., 1996), alumina-based bilayer (An *et al*., 1996), silicon nitride bilayer (Lee *et al*., 1998), ceramic/metal systems (Wuttiphan *et al*., 1997), alumina/Zirconia based bilayer (Guazzato *et al*., 2004), Zirconia Y-TZP (Zhang et al., 2004). Generally, coating fracture is the dominant source of failure. Such fracture is driven by tensile stresses which concentrate at the surface outside the contact and at the internal interface immediately below the contact, with relative intensities dependent on the interlayer elastic-plastic mismatch (as measured by differences in elastic modulus and hardness) and coating thickness. Debate continues as to which of these two locations of tensile concentration dominates clinical failures of ceramic-based restorations. In systems where the mismatch is small (glass-glass-ceramic bilayer) (Wuttiphan *et al*., 1996), first crack initiation tends to occur at the top surface; in systems where the mismatch is large and the underlayer is soft (ceramic-metal/Zirconia systems) (Pajares *et al*., 1996a, b), first initiation tends to occur at the internal interface. Plasticity in the substrate can exacerbate this latter kind of interior fracture. As the load is increased above the cracking threshold, the fracture patterns can become complex, with a multiplicity of co-existent cracks extending both downward from the top surface and upward from the internal interface (and even some initiated within the coating interior) (Lee *et al*., 1998). Such multiple cracks tend to be highly stable, consistent with the clinical experience of sustainable hairline cracks in tooth enamel, especially in older patients. Such cracks may not always be clearly visible by surface inspection prior to failure.

Fractography is the analysis of fracture surfaces. It refers to the quantitative fracture surface analysis (FSA) in the context of applying the principles of fracture mechanisms to the topography observed on the fracture surface of brittle materials. The application of FSA is based on the principle that encoded on the fracture surface of brittle materials is the entire history of the fracture process (Mecholsky, 1995).

In glasses, glass ceramics, polycrystalline ceramics and polymers, many time-dependent phenomena have been observed. Slow crack growth due to stress corrosion processes have been observed both at room temperature and at elevated temperatures in brittle and ductile materials. On the other hand, creep and viscoelastic processes can be observed in all other materials under the FSA. Cyclic fatigue processes can lead to an apparent time dependence failure. These slow crack growth phenomena can be detected using the principles of FSA.

Polycrystalline materials often have a change in the fracture surface topography as the crack transitions from slow growth to rapid growth. In many brittle materials this change is characterized by transition from inter-granular propagation in the slow crack regime to trans-granular in the fast crack regime. However, other paths have been observed, most notably a transition from trans-granular in slow crack growth to inter-granular for fast crack growth in ferrites. Situations arise where the transition from slow crack growth to fast crack growth is not marked by a change in morphology, e.g. in aluminum oxide and aluminum oxide-based materials at room temperature. At these times the crack propagation proceeds primarily inter-granularly throughout the specimen.

Complex loading on pre-existing cracks can be resolved into three modes. If the loading is tensile, it is referred to as Mode I; if it is in-plane shear, Mode II; and out-of-plane shear (or torsion), is referred to as Mode III. Although most of the fractures that occur in brittle materials are primarily Mode I fractures, there are cases in which shear loading, i.e., Mode II and/or Mode III, are important contributors to failure (Jayatilaka, 1979). These cases include bi-material applications, torsional loading, and machining damage, which results in cracks that are at an angle to the applied stressing direction and composites. Usually when shear contributes to failure, the initial propagation is affected, i.e., the load which leads to failure are higher than expected for that size crack and the angle of propagation is affected. However, in most cases, soon after the crack starts propagation, Mode I loading will dominate.

Dental ceramics testing methods

The determination of the true tensile strength of ceramic materials has always presented experimental difficulties in part because most ceramics are brittle and have significantly lower tensile vs. compressive strengths. Large differences between tensile and compressive strengths results from ceramics inability to dissipate stress concentrations. The localize stress build-ups in brittle ceramic material may result in crack initiation at the defect and crack propagation through the material (Griffith, 1920). Such stress concentrations usually occur at the point of load application in the test fixture and result in premature fracture. Stress concentrations that may occur under tensile loading produce problems in accurately measuring the true tensile properties of ceramics.

The testing of dental porcelain has local stress concentration problem. Ideally the tensile strength of ceramics would be determined from a direct tension test but stress concentrations in the grips that holding the specimen have produced premature fractures and ceramics lack any appreciable plastic deformation. It is virtually impossible to apply a tensile load uniformly over the end of a rod, making this approach unrealistic in practice. In addition, alignment problems of the specimen usually cause bending that went undetected unless strain gauges were place on the specimens. So the tensile testing of ceramics is difficult and expensive. However, since the tensile strength is the most desired design property for assessing mechanical failure, considerable research has focused on improving the tensile test methods for ceramics, with the result being that tensile testing is becoming more common. For these reasons, in dentistry most tensile strengths were obtained indirectly using the bending test or diametral compression test. The bending test is including the three-point and four-point tests. The diametral compression test is including the piston-on-three ball, ring-on-ring test, etc.

The bend test is a simple, reliable, and sensitive method for testing the relative strength of dental ceramic material. No special grip is required and simple sample shapes may be used. The bending test is independent of test geometry for a range of specimen shapes and test configurations. Furthermore, the International Standards Organization (ISO) supports the use of the simple three-point-bend test as a means of evaluating the strength of dental porcelain. However, one criticism of three-point bending test is that the bending creates a stress gradient in the specimen, and only a relatively small volume is exposed to high tensile stress. Also, the specimens are very sensitive to flaws along the sample edge or surface machining damage. It is impossible to eliminate all flaws and, because fracture often initiates at the edges, large variations strength data have been recorded. Due to those factors the test is deceptive in that it appears easy to set up and conduct, but too often the strength-controlling flaws in the bend test are not the same as for a component in service.

On the other hand biaxial bend test is recommended since the edges of the sample are not stressed and biaxial loading is commonly encountered in service. The biaxial flexure test method involves supporting a specimen on three or more points near its periphery and equidistance from its center and loading a more central portion. The area of maximum tensile stress falls at the center of the lower face of the plate and the strength should be independent of the condition of the edges of the plate. Ring on ring involves supporting a circular plate on a ring and loading with a small concentric ring. Wachtman and co-workers (Wachtman *et al*., 1972) evaluated the biaxial flexure tests of eight types of aluminum oxide substrate by eleven testing laboratories using five testing methods (ring on ring, piston on ring, ball on ring, piston on 3 ball, ball on 3 ball). The results showed that the piston on 3-ball biaxial test method is useful for quality control purposes with the 7% variation being sufficiently small. However, as with any bend test, the maximum stress is on the surface, and volume flaws are not generally strength controlling (Ritter, 1995).

There are two elastic solutions that exist for the biaxial flexure test, each using unique corrections for stiffening by the annulus of material outside the support radius, and each behaving differently with changes in disc and loading piston dimensions. One of the biaxial stress equations is based on an analysis by Kirstein and Woolley of the elastic solution of Bassali (Kirstein *et al.*, 1965; Bassali, 1957). Kirstein and Woolley observed that Bassali solution accounted for the stiffening effect of the annular portion of the circular disc overhanging the support circle and that the stiffening effect decreased with increasing numbers of support points but never vanished completely. The second approach to the axial stress equation comes from general formulae given by either Roark or Timoshenko and Woinowsky-Krieger for a center-loaded disc uniformly supported along its periphery (Roark, 1965; Timoshenko *et al.*, 1959). These equations are used to correct the effects of the testing methods in order to find the relative flexural strength of the material.

In biaxial stress equations based on these general solutions, three-point support is assumed to be equivalent to uniform support, and material extending beyond the periphery support, receives no special consideration (Kelly, 1995). The piston-on three-ball test has the disadvantage that the load distribution under the piston is uncertain and difficult to model; ultimately the load is not uniformly distributed (Zeng *et al.*, 1996)

A valid test method should permit the determination of the strength of specimens having the same surface condition which they have in use and so, should not require any machining of a type not actually occurring in the normal use of the material. Most substrates are used with as fired of surfaces, so a test method capable of measuring strength on specimens in this condition is needed. Although many investigations have been carried out to assess the strength of the dental porcelain, a comparison of these results is sometimes confusing. Zeng and co-workers evaluated the failure stress in flexural tests of the aluminum oxide coping material by using the three-point bending, ring-on-ring, and piston-on-three-ball tests. These authors reported substantial differences in the failure stresses (up to 50%) with the different testing methods. Conclusions of this study emphasized the importance of knowing the test method being used and the method of calculation when data are reported or compared (Zeng *et al*., 1996)

The specimen size and method of fabrication should be as close as possible to normal practice. For the strength test to accurately reflect the variability and time-dependency of a ceramic component in service, the test environment must be the same as the service environment, and the strength-controlling flaw population must be the same as that responsible for failure in service. For this reason, it is generally recommended that the test sample and mode of loading be chosen to closely simulate the actual component in service (Ritter, 1995).

Before testing two factors must be considered; first the glass always fails in tension and second, the surface characteristics of glass is the most important factor influencing its strength. All ordinary cracks in glass start from tensions produced by pressures, scratches, or abrasions; sources of these tensions are surface and internal imperfections such as gas bubbles. The place and distribution and the time when these tensions develop are factors that vary with testing conditions.

CHAPTER 2

OBJECTIVES OF THE STUDY

The purpose of this study was to evaluate the influence of different surface finish and heat treatments namely grinding, polishing and glazing on the fracture resistance, measured in terms of biaxial flexural strength, of different feldspathic veneering ceramics used to stratified Zirconia ceramic frameworks and assess the reliability, strength and mode of fracture of feldspathic veneering ceramics/Zirconia ceramic frameworks used in the fabrication of prosthodontic fixed restorations.

To reach this purpose, quantitative measurements were made of the fracture resistance of the feldspathic veneering ceramics and the feldspathic veneering ceramics/Zirconia ceramic frameworks and, qualitative evaluations of the ultramorphology and mode of fracture of the veneering ceramic/Zirconia ceramic interfaces.

From the referred objectives it resulted the formulation of the following experimental hypotheses:

$H_1.0$: There are no significant differences in the load to fracture, measured in terms of biaxial flexural strength, among different ceramic surface treatments in the feldspathic veneering ceramics.

$H_1.1$: There are significant differences in the load to fracture, measured in terms of biaxial flexural strength, among different ceramic surface treatments in the feldspathic veneering ceramics.

H_2.0: There are no significant differences in the load to fracture, measured in terms of biaxial flexural strength, among different ceramic surface treatments in each feldspathic veneering ceramic.

H_2.1: There are significant differences in the load to fracture, measured in terms of biaxial flexural strength, among different ceramic surface treatments in each feldspathic veneering ceramic.

H_3.0: There are no significant differences in the load to fracture, measured in terms of biaxial flexural strength, among the feldspathic veneering ceramics/Zirconia ceramic frameworks independently of the side tested under tensile stress.

H_3.1: There are significant differences in the load to fracture, measured in terms of biaxial flexural strength, among the feldspathic veneering ceramics/Zirconia ceramic frameworks independently of the side tested under tensile stress.

H_4.0: There are no significant differences in the load to fracture, measured in terms of biaxial flexural strength, among each feldspathic veneering ceramic/Zirconia ceramic frameworks independently of the side tested under tensile stress.

H_4.1: There are significant differences in the load to fracture, measured in terms of biaxial flexural strength, among each feldspathic veneering ceramic/Zirconia ceramic frameworks independently of the side tested under tensile stress.

H_5.0: There are no significant differences in the mode of fracture among the feldspathic veneering ceramics/Zirconia ceramic frameworks independently of the side tested under tensile stress.

H_5.1: There are significant differences in the mode of fracture among the feldspathic veneering ceramics/Zirconia ceramic frameworks independently of the side tested under tensile stress.

CHAPTER 2

MATERIALS AND METHODS

1- Surface finish and heat treatment study

Type of research

This was an experimental *in vitro* study using feldspathic dental veneering ceramic discs used to layer Zirconia frameworks in the fabrication of prosthodontic fixed restorations.

Dependent variable

Load to fracture was measured in terms of biaxial flexural strength of the material.

Independent variable

Surface finish and heat treatment of 3 feldspathic dental veneering ceramics using the following different surface treatments: 1) manufacturer's instructions, and mimicking some possible deviations of the clinical application; 2) grinding of the ceramic with diamond instrument, 3) grinding of the ceramic with diamond instrument followed by glazing procedure, 4) grinding of the ceramic with diamond instrument followed by polishing procedure, 5) grinding of the ceramic with diamond instrument followed by polishing and glazing procedures, and 6) polishing of the ceramic surface followed by glazing procedure.

Constant independent

Apparatus used to measure the biaxial flexural strength – Universal Instron testing machine determined by the piston-on three-ball test method (See section – biaxial flexural strength test).

Sample size calculations

Sample sizes were chosen based on data from a similar experiment (White *et al*., 2005; White *et al*., 1996). However, a pilot study was undertaken to evaluate the sample size necessary that would yield an estimated pooled standard deviation of 12.05. Power was set at 80%, and an overall level of Type I error equal to 0.05 was desired. Since all pairwise comparisons among the eighteen (18) experimental groups were be examined, the level of Type I error for a given pairwise comparison was set at alpha=0.05/15 =0.0034, based on Bonferroni adjustment for multiple comparisons. This maintains a minimum level of 0.05 for the experiment-wise Type I error. Based upon a desire to be able to detect a 15% difference between mean percentage surface treatment reductions between a pair of groups, 10 specimens per treatment group were required. Given that multiple comparisons adjustment was actually carried out using the t-test method, power would be expected to be slightly greater.

Design of the study

A convenient sample of hundred and eighty (180) disc-shaped specimens 2.2(± 0.1) mm thick and 12.7(± 0.1) mm in diameter were fabricated and used in this study. The disc specimens were fabricated using feldspathic veneering dental ceramic used to stratified Zirconia 3Y-TZP frameworks of three (3) commercially brands: 1) NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), 2) Lava™ Ceram veneer ceramic (3M™, ESPE™, Germany), and 3) Vita® VM®9 veneer ceramic (Vita®, Zahnfabrick, Germany). The feldspathic veneering porcelains used for the study were the NobelRondo™ Zirconia veneer ceramic Dentine A2 (Lot No. 0709); the Lava™ Ceram veneer ceramic Dentine A2 (Lot No. 189943); and the Vita® VM®9 veneer ceramic Dentine A2 (Lot No. 7565) (Figure 1). All disc specimens were fabricated according to the specific manufacturer's recommendations for each ceramic.

Figure 1. Feldspathic veneering ceramics

Preparation of the specimens

Specimens were prepared according to ISO/DIS 6872: 1995 (three-point and biaxial flexural strength) (ISO/DIS 6872: 1995). Sixty (60) monolithic specimens of each ceramic were fabricated using a separable steel mold. The mixing liquid and the ceramic powders were combined in proportions recommend by the manufacturers to form a sticky slurry, which was vibrated and packed into the mold. Excess liquid was sucked off with absorbent tissue paper. The firing of the specimens was performed in a programmable and calibrated vacuum ceramic furnace (Programat P500, Ivoclar Vivadent AG, Liechtenstein) according to the recommendations of the manufacturers (Figure 2).

Figure 2. Ceramic furnace Programat P500

After the first firing, more porcelain was added and fired to compensate for the shrinkage resulting from the first sintering. The fired porcelain discs were examined with a stereomicroscope (Nikon SMZ-U, Tokyo, Japan) at original magnification X75 to evaluate the specimens for small cracks and flaws (Figure 3).

Figure 3. Stereomicroscope Nikon SMZ-U

Specimens displaying visible defects were replaced. All specimen surfaces were then polished with SiC discs (grit-silicon-carbide P220, P500, P1200 - Ultra-Prep, Buehler Ltd., Lake Bluff, IL, USA) in a mechanical grinder (Ecomet® 3, Buehler Ltd., Lake Buff, IL, USA) according to ISO 6344-1: 1998 (ISO/DIS 6344-1: 1998) (Figure 4).

Figure 4. Ecomet 3, mechanical grinder

This procedure was carried until specimens with a thickness and diameter of approximately 2.2 (± 0.1) mm by 12.7(± 0.1) mm respectively were obtained. A special stainless steel holder was used to ensure accuracy of thickness and parallelism of the surfaces during grinding and polishing. As required by the ISO Standards the two faces of the specimens did not differ more than 0.05 mm in parallelism. The specimens' dimensions were measured with a digital micrometer (Digimatic Caliper Series 500, Mitutoyo America Corporation, Dawn, IL, USA) to ensure the exact thickness and diameter.

Finally, all specimens were cleaned in an ultrasonic bath (Eurosonic® 4D, Euronda, Vicenza, Italy) with distilled water for 15 minutes before the auto glaze firing was performed in the ceramic furnace according to the recommendations of the manufacturers (Figure 5). After the glazing procedure, all specimens were again measured with the same digital caliper. The diameter and thickness of the ceramic discs were measured by the micrometer to the nearest 1/100 mm at five randomly selected locations prior to the test procedures.

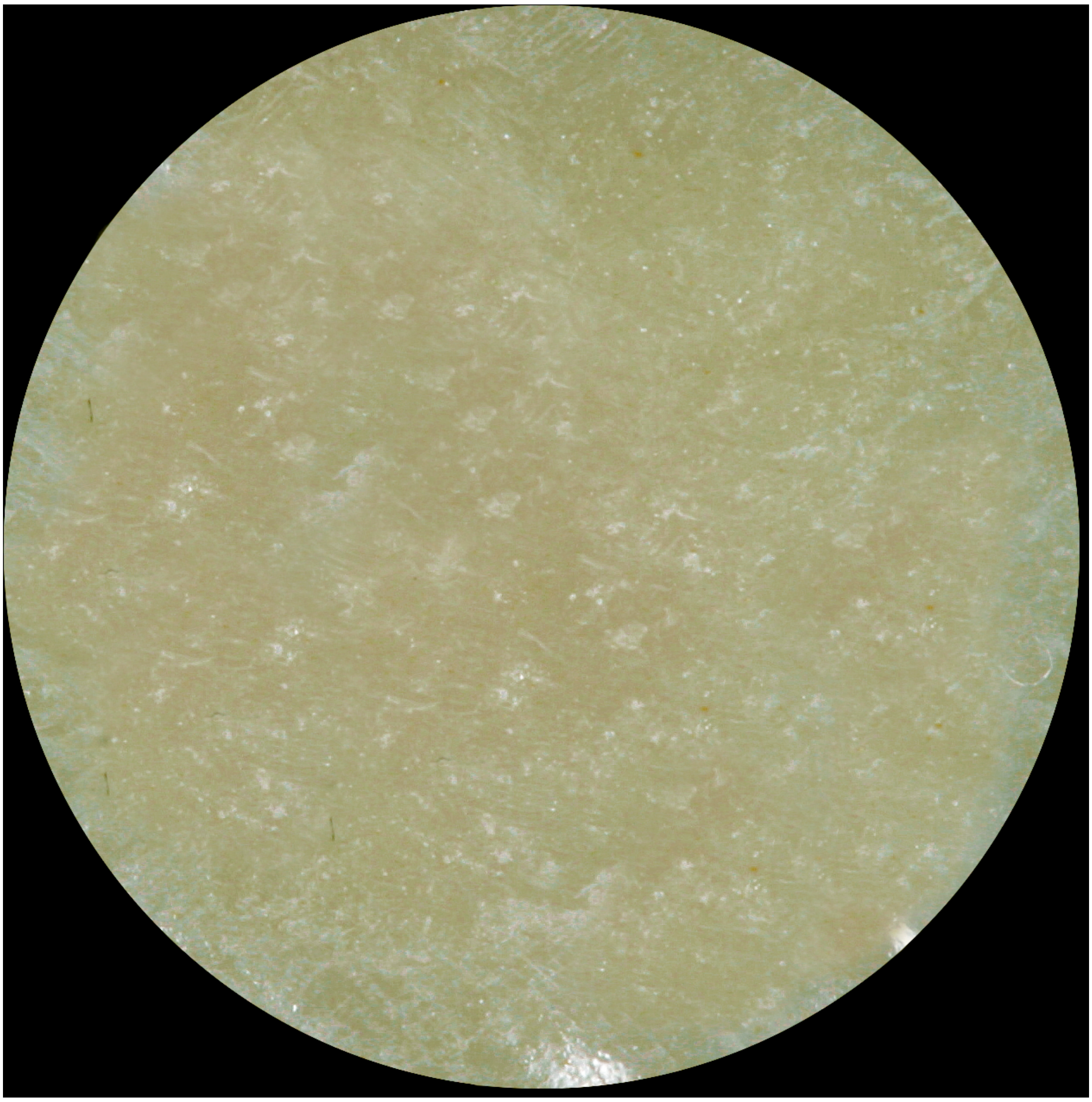

Figure 5. Test specimen before testing procedure

Distribution and treatment of the specimens

The hundred and eighty (180) specimens were randomly assigned to the eighteen groups, six groups for each ceramic, each group consisting of ten specimens (Table 1). The order in which the specimens were treated was random, to avoid a possible bias due to any particular sequence of treatment.

Table 1 Composition of each group and respective material and surface treatment

Groups	**Material**	**Surface Treatment**
Group 1	NobelRondo Zr (Dentine A2)	Manufacturer's instructions
Group 2	NobelRondo Zr (Dentine A2)	Grinding
Group 3	NobelRondo Zr (Dentine A2)	Grinding + Glazing
Group 4	NobelRondo Zr (Dentine A2)	Grinding + Polishing
Group 5	NobelRondo Zr (Dentine A2)	Grinding + Polishing + Glazing
Group 6	NobelRondo Zr (Dentine A2)	Polishing + Glazing
Group 7	3M Lava Ceram (Dentine A2)	Manufacturer's instructions
Group 8	3M Lava Ceram (Dentine A2)	Grinding
Group 9	3M Lava Ceram (Dentine A2)	Grinding + Glazing
Group 10	3M Lava Ceram (Dentine A2)	Grinding + Polishing
Group 11	3M Lava Ceram (Dentine A2)	Grinding + Polishing + Glazing
Group 12	3M Lava Ceram (Dentine A2)	Polishing + Glazing
Group 13	Vita VM9 (Dentine A2)	Manufacturer's instructions
Group 14	Vita VM9 (Dentine A2)	Grinding
Group 15	Vita VM9 (Dentine A2)	Grinding + Glazing
Group 16	Vita VM9 (Dentine A2)	Grinding + Polishing
Group 17	Vita VM9 (Dentine A2)	Grinding + Polishing + Glazing
Group 18	Vita VM9 (Dentine A2)	Polishing + Glazing

The six experimental groups, for each ceramic, were prepared as described previously and submitted to the treatment surfaces in the following manner:

Groups 1/7/13: Control group (CN)

The preparation of the specimens was performed according to manufacturer's instructions for each ceramic through the method previously described. No surface treatment was made to any group CN specimens. These groups were used as control group.

Groups 2/8/14: Grinding procedure (G)

After preparation of the specimens according to manufacturer's instructions for each group of ceramic, the samples were ground under water coolant with a 91 μm grit size diamond cup wheel (100 mm diameter, 291 6ª Struers diamond cup wheel, Copenhagen, Denmark) mounted on a cutting/grinding machine (Struers Accutom 50, Copenhagen, Denmark). Grinding conditions were: 3300 rpm, 2 cm/s horizontal speed, 5 μm depth of cut. A special stainless steel holder was used to ensure accuracy of thickness and parallelism of the surfaces during grinding and oriented perpendicular the length of the specimen (perpendicular to the tensile stresses applied during fracture strength test).

Groups 3/9/15: Grinding followed by glazing (GG)

After preparation of the specimens according to manufacturer's instructions for each group of ceramic, the samples were ground under water coolant with a 91 μm grit size diamond cup wheel (100 mm diameter, 291 6ª Struers diamond cup wheel, Copenhagen, Denmark) mounted on a cutting/grinding machine (Struers Accutom 50, Copenhagen, Denmark). Grinding conditions were: 3300 rpm, 2 cm/s horizontal speed, 5 μm depth of cut. A special stainless steel holder was used to ensure accuracy of thickness and parallelism of the surfaces during grinding and oriented perpendicular the length of the specimen (perpendicular to the tensile stresses applied during fracture strength test). All specimens were then auto-glazed in a programmable and calibrated vacuum ceramic furnace (Programat P500, Ivoclar Vivadent AG, Lichenstein) according to the recommendations of the manufacturers for each ceramic group.

Groups 4/10/16: Grinding followed by polishing (GP)

After preparation of the specimens according to manufacturer's instructions for each group of ceramic, the samples were ground under water coolant with a 91 µm grit size diamond cup wheel (100 mm diameter, 291 6ª Struers diamond cup wheel, Copenhagen, Denmark) mounted on a cutting/grinding machine (Struers Accutom 50, Copenhagen, Denmark). Grinding conditions were: 3300 rpm, 2 cm/s horizontal speed, 5 µm depth of cut. A special stainless steel holder was used to ensure accuracy of thickness and parallelism of the surfaces during grinding and oriented perpendicular the length of the specimen (perpendicular to the tensile stresses applied during fracture strength test). All specimen surfaces were then polished with SiC discs (grit-silicon-carbide P220, P320, P500, P800, P1200 - Ultra-Prep, Buehler Ltd., Lake Bluff, IL, USA) in a mechanical grinder (Ecomet 3, Buehler Ltd., Lake Buff, IL, USA) according to ISO 6344-1: 1998 followed by 4, 2, and 1 µm diamond paste (diamond polishing paste, Henry Schein Inc., NY, USA) mounted on a laboratory handpiece machine (KaVo EWL K4, KaVo Dental Gmbh, Germany).

Groups 5/11/17: Grinding followed by polishing and glazing (GPG)

After preparation of the specimens according to manufacturer's instructions for each group of ceramic, the samples were ground under water coolant with a 91 µm grit size diamond cup wheel (100 mm diameter, 291 6ª Struers diamond cup wheel, Copenhagen, Denmark) mounted on a cutting/grinding machine (Struers Accutom 50, Copenhagen, Denmark). Grinding conditions were: 3300 rpm, 2 cm/s horizontal speed, 5 µm depth of cut. A special stainless steel holder was used to ensure accuracy of thickness and parallelism of the surfaces during grinding and oriented perpendicular the length of the specimen (perpendicular to the tensile stresses applied during fracture strength test). All specimen surfaces were then polished with SiC discs (grit-silicon-carbide P220, P320, P500, P800, P1200 - Ultra-Prep, Buehler Ltd., Lake Bluff, IL, USA) in a mechanical grinder (Ecomet 3, Buehler Ltd., Lake Buff, IL, USA) according to ISO 6344-1: 1998 followed by 4, 2, and 1 µm diamond paste (diamond polishing paste, Henry Schein Inc., NY, USA) mounted on a laboratory handpiece machine (KaVo EWL K4, KaVo Dental Gmbh, Germany). All specimens were then auto-glazed in a programmable and calibrated vacuum ceramic furnace (Programat P500, Ivoclar Vivadent AG, Lichenstein) according to the recommendations of the manufacturers for each ceramic group.

Groups 6/12/18: Polishing followed by glazing (PG)

After preparation of the specimens according to manufacturer's instructions for each group of ceramic, all specimen surfaces were then polished with SiC discs (grit-silicon-carbide P220, P320, P500, P800, P1200 - Ultra-Prep, Buehler Ltd., Lake Bluff, IL, USA) in a mechanical grinder (Ecomet 3, Buehler Ltd., Lake Buff, IL, USA) according to ISO 6344-1: 1998 followed by 4, 2, and 1 µm diamond paste (diamond polishing paste, Henry Schein Inc., NY, USA) mounted on a laboratory handpiece machine (KaVo EWL K4, KaVo Dental Gmbh, Germany). All specimens were then auto-glazed in a programmable and calibrated vacuum ceramic furnace (Programat P500, Ivoclar Vivadent AG, Lichenstein) according to the recommendations of the manufacturers for each ceramic group.

Before testing all specimens were examined with a stereomicroscope (Nikon SMZ-U, Tokyo, Japan) at original magnification X75 to evaluate the specimens for small cracks and flaws. Specimens displaying visible defects were replaced.

Biaxial flexural strength test

The standard for testing the strength of dental ceramics has been the three-point flexural test, but one problem has been the sensitivity of the test to flaws along the sample edges. It is impossible to eliminate all flaws and, because fracture often initiates at the edges, large variations strength data have been recorded (Ban *et al*., 1990). The biaxial flexural test eliminates the effect of edges because they are not directly loaded. Therefore the biaxial test should produce less variation in data for strength determination and this was the reason way it was used in this study. With the biaxial flexure test, a disc-shaped specimen is supported from below by either a ring or several ball bearings distributed in a circular pattern. The load is applied from above by use of a piston in a position concentric with the supporting ring or ball bearings (Figure 6).

Figure 6. Disc-shaped test specimen positioned over testing apparatus before fracture strength measurement. The specimen is supported by 3 ball bearings distributed in a circular pattern. The load is applied from above by use of a piston in a position concentric with the supporting ball bearings.

The load to fracture was measured with the piston-on-three-ball test method by using a Universal Instron testing machine (Model TT-BM Instron Corp., Canton, MA) according with the Standard ISO/DIS 6872 for dental ceramics (ISO/DIS 6872: 1995) (Figure 7).

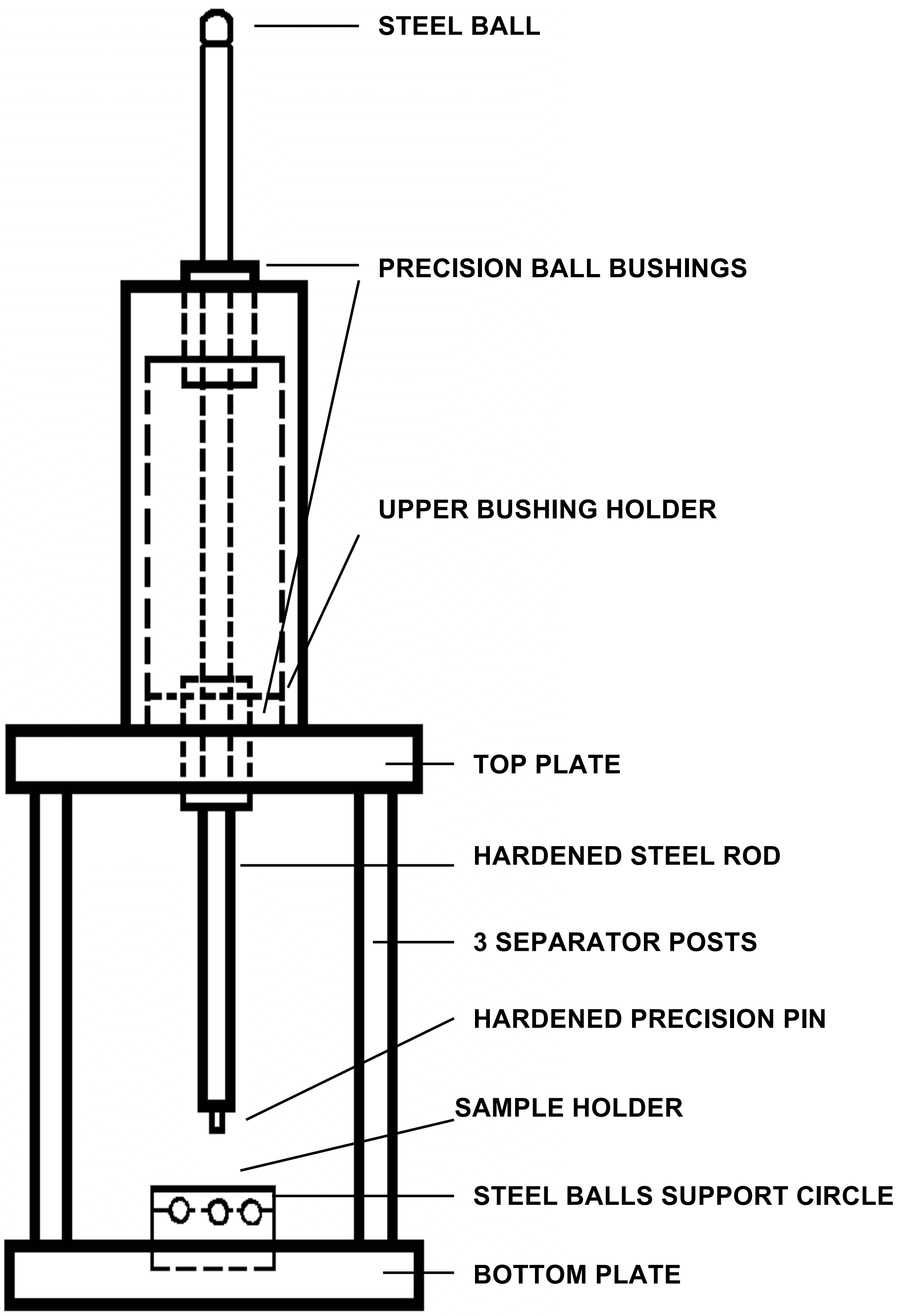

Figure 7. Schematic diagram of the apparatus used for the biaxial flexure strength measurements.

The disc specimens were first positioned in the sample holder on top of the supporting balls, with the treated surface in tension on three symmetrically spaced balls. The three balls, of 1.5 mm diameter, were equidistant around a circle of radius 6 mm. The load was applied at the center of the top surface through the flat tip of a cylindrical piston (diameter 0.7 mm) at a cross-head speed of 0.5 mm/min until fractured had occurred (Figure 8). The test was conducted at room conditions (22 ± 1°C, and 60% ± 5% relative humidity).

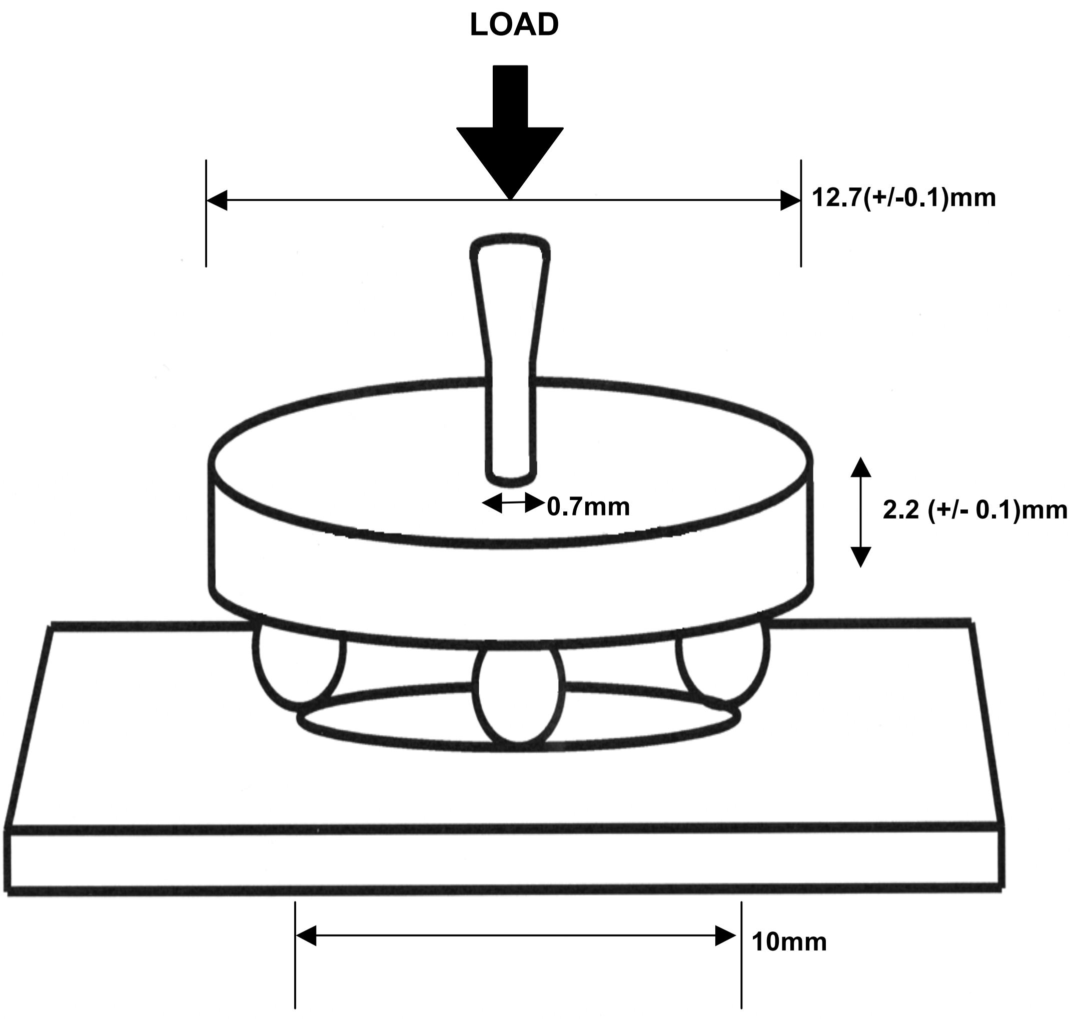

Figure 8. Schematic diagram of biaxial flexural strength test arrangement

Calculations to determine biaxial flexural strength

The recorded maximum resistance to fracture (N) was used in conjunction with the following formula (ASTM F 394-78, 1996), which resulted in the biaxial flexural strength for each specimen.

$$S = - 0.2387 P (X - Y)/d^2$$

Where S represents the maximum center tensile stress/flexural strength (MPa), P is the maximum resistance to fracture (N) and *d* is the specimen thickness at fracture origin (mm). X and Y where determined as follows:

$$X = (1+n) ln(B/C)^2 + [(1- n)/2](B/C)^2$$

$$Y = (1+n) [1+ln (A/C)^2] + (1- n)(A/C)^2$$

Where *n* represents the Poisson's ratio, A is the radius of the supporting circle (mm), B is the radius of the tip of the piston or radius of the loaded area (mm) and C is the radius of the specimen (mm). In this study, *n* = 0.25, A = 5mm, and B = 0.7mm.

Statistical analysis

The statistical analysis of the results was performed through descriptive and inference methods. The descriptive statistic of the maximum resistance to fracture of all group specimens was performed according to common methods determining the mean, median, standard deviation, standard error, variance, and the minimum and maximum values for all studied group specimens.

The influence of the surface/heat treatments and ceramic material on the biaxial flexural strength was analyzed by using two-way ANOVA followed by Fisher's PLSD (protected least significant difference) test to identify the source of differences with an overall 5% level of error. In the present study, the Fisher's (PLSD) test was chosen because it is usually less conservative than other post hoc all-pairwise tests. Therefore, if a significant ANOVA result is found, then Fisher's (PLSD) is more likely than some other tests to identify a significant pairwise comparison.

For each feldspathic veneering ceramic one-way ANOVA ceramic was used to analyze if there was significant difference between the six groups tested. Pairwise comparisons among the six groups for each ceramic were made using Fisher's PLSD (protected least significant difference) test to identify the source of differences with an overall 5% level of error. An ANOVA test is used to find out if there is a significant difference between three or more group means. However, the ANOVA analysis simply indicates there is a difference between two or more group means, but it does not tell what means there is a significant difference between. In order to find out what means there is a significant difference between, a post hoc test needs to be done. Fisher's PLSD (probable least significant difference) test is a post hoc test designed to perform a pairwise comparison of the means to see where the significant difference is; this was the reason why it was used.

In addition, the Weibull moduli were calculated for biaxial flexural strength data. Weibull moduli are a statistical property, based on the Weibull distribution to characterize variability of the strength of ceramic materials. High Weibull moduli imply low variability in strength. Weibull moduli were calculated by plotting *ln ln* (1/1 – F) versus *ln*(s).

Where F = (i – 0.5)/n, i = rank of a sample in terms of strength (i = 1 for the lowest-strength sample); n = total number of samples; and s = strength of sample i. The slope of the line was then determined by use of linear regression. The slope of the line was calculated as the Weibull modulus.

The Weibull equation describes the probability of failure as a function of a characteristic stress and a shape parameter, the Weibull modulus. The modulus can be found as the slope of a standard Weibull plot, i.e., *ln* (1/1- F) versus *ln* (failure load), where F = probability of failure. The Weibull coefficient m is a measure of variability in the strength of the material. Large values of the Weibull parameter m indicate a small variability in strength.

The failure stresses of the ceramic materials from each test were analyzed using the Weibull formulation, which is given by:

$F1- \exp [-\int_v (\sigma - \sigma_u / \sigma_0)m \ dv]$

This is the three-parameter Weibull function where σ_u, σ_0, and m are three parameters. σ_u is the threshold stress (i.e., the stress below which the probability of failure is zero). σ_0 is a normalizing parameter (often selected as the characteristic stress, at which the probability of failure is 0.632), m is the Weibull modulus, which describes the flaw size distribution (and thus the data scatter), and F is the failure probability that can be estimated as described above. Higher m values indicate a narrower flaw size distribution, and therefore lower scatter in the data; low m values indicate a larger flaw size distribution and therefore a larger spread in the data.

2- Strength and reliability study

Type of research

This was an experimental *in vitro* study using Zirconia ceramic discs stratified with feldspathic dental veneering ceramic used to layer Zirconia frameworks in the fabrication of prosthodontic fixed restorations.

Dependent variable

Load to fracture was measured in terms of biaxial flexural strength of the material.

Independent variable

Feldspathic dental veneering ceramic employed to stratified Zirconia 3Y-TZP subtracts of three (3) commercially brands: 1) NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), 2) Lava™ Ceram veneer ceramic (3M™ ESPE™, Germany), and 3) Vita® VM®9 veneer ceramic (Vita® Zahnfabrick, Germany).

Constant independent

Apparatus used to measured the biaxial flexural strength – Universal Instron testing machine determined by the piston-on three-ball test method (See section – biaxial flexural strength test)

Sample size calculations

Sample sizes were chosen based on data from a similar experiments (Guazzato *et al*., 2004; White *et al*., 2005). However, a pilot study was undertaken to evaluate the sample size necessary which would yield an estimated pooled standard deviation of 13.45. Power was set at 80% and an overall level of Type I error equal to 0.05 was desired. Since all pairwise comparisons among the six (6) experimental groups were to be examined, the level of Type I error for a given pairwise comparison was set at alpha=0.05/15=0.0029 based on Bonferroni adjustment for multiple comparisons. This maintains a minimum level of 0.05 for the experiment-wise Type I error. Based upon a desire to be able to detect a 15% difference between mean percentages between a pair of groups, 10 specimens per treatment group were required. Given that multiple comparisons adjustment was actually carried out using the t-test method, power would be expected to be slightly greater.

Design of the study

A group of sixty (60) disc-shaped specimens 2.2(± 0.1) mm thick and 12.7(± 0.1) mm in diameter were fabricated and used in this study. The disc-shaped specimens were composed of a framework of 3Y-TZP produced by a CAD-CAM technique and a feldspathic veneering dental ceramic. The porcelains used to stratify the Zirconia 3Y-TZP frameworks were from 3 commercially available brands: 1) NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), 2) Lava™ Ceram veneer ceramic (3M™ ESPE™, Germany), and 3) Vita® VM®9 veneer ceramic (Vita® Zahnfabrick, Germany). The feldspathic veneering dental ceramics used for the study were the NobelRondo™ Zirconia veneer ceramic Base Liner (Lot No. 0508) and NobelRondo™ Zirconia veneer ceramic Dentine A2 (Lot No. 0709); the Lava™ Ceram veneer ceramic Frame-Work Modifier (Lot No. 167786) and Lava™ Ceram veneer ceramic Dentine A2 (Lot No 189943); and the Vita VM®9 veneer ceramic Base Dentine (Lot No. 7933) and Vita VM®9 veneer ceramic Dentine A2 (Lot No. 7565) (Figure 9). All disc specimens were fabricated accordingly to the specific manufacturer's recommendations for each ceramic.

Figure 9. Feldspathic veneering ceramics dentin and opaque

Preparation of the specimens

Specimens were prepared according to ISO/DIS 6872: 1995 (three-point and biaxial flexural strength) (ISO/DIS 6872: 1995). Sixty (60) 3Y-TZP discs with 1.1 (± 0.1) mm thick and 12.7 (± 0.1) mm in diameter were produced by CAD/CAM technology (Nobel Biocare™ AB, Sweden) and used to veneer the 3 brands of feldspathic veneering dental ceramics. The discs were obtained from the manufacturer without any surface treatment and in the same condition that 3Y-TZP substructures are delivered to the commercial laboratory. Twenty bilayered specimens of each ceramic were fabricated using a separable steel mold. The mixing liquid and the opaque ceramic were combined in proportions recommend by the manufacturers to form a sticky slurry, which was applied over the 3Y-TZP discs and then vibrated. The firing of the specimens was performed in a programmable and calibrated vacuum ceramic furnace (Programat P500, Ivoclar Vivadent AG, Lichenstein) according to the recommendations of the manufacturers. After the first firing the bilayered specimens were prepared by veneering the core materials with the feldspathic ceramics. The discs were put in a steel mold and the ceramic powders were combined in proportions recommend by the manufacturers to form a sticky slurry, which was vibrated and packed. Excess liquid was wicked off of the surface with absorbent tissue paper. The firing of the specimens was performed in the same programmable and calibrated vacuum ceramic furnace according to the recommendations of the manufacturers. After the first firing, additional porcelain was added and fired to compensate for the shrinkage resulting from the first sintering. The fired porcelain discs were examined with a stereomicroscope (Nikon SMZ-U, Tokyo, Japan) at a magnification X75 to evaluate the specimens for small cracks or flaws. Specimens displaying defects were replaced. All specimen surfaces were then polished with SiC discs (grit-silicon-carbide P220, P500, P1200 - Ultra-Prep, Buehler Ltd., Lake Bluff, IL, USA) in a mechanical grinder (Ecomet® 3, Buehler Ltd., Lake Buff, IL, USA) according to ISO 6344-1: 1998 (ISO/DIS 6344-1: 1998). This procedure was repeated until samples with a thickness and diameter of approximately 2.2(± 0.1) mm x 12.7(± 0.1) mm respectively were obtained. A special stainless steel holder was used to ensure accuracy of thickness and parallelism of the surfaces during grinding and polishing. As required by the ISO Standard the two faces of the specimens did not differ more than 0,05 mm in parallelism. The specimens' dimensions were measured with a digital micrometer (Digimatic Caliper Seriates 500, Mitutoyo America Corporation, Dawn, IL, USA) to ensure the exact thickness and diameter. Finally, all specimens were cleaned in an ultrasonic bath (Eurosonic® 4D, Euronda, Vicenza, Italy) with distilled water for 15 minutes before auto glazing according to the recommendations of the manufacturers (Figure 10). After the glazing procedure, all specimens were again measured with the same digital caliper. The diameter and thickness of the ceramic discs were measured by the micrometer to the nearest 1/100 mm at five randomly selected locations prior to the test locations.

Figure 10. Test specimen before testing procedure. Top and cross-section view

Distribution of the specimens

The sixty (60) specimens were assigned to the six groups, two groups for each ceramic, each group consisting of ten specimens (Table 2). The order in which the specimens were treated was random, to avoid a possible bias due to any particular sequence of treatment.

Table 2 Composition of each group and respective material and surface treatment

Groups	Material	Treatment
Group 1	NobelRondo Zr (top) Zirconia (bottom)	Manufacturer's instructions
Group 2	Zirconia (top) NobelRondo Zr (bottom)	Manufacturer's instructions
Group 3	3M Lava Ceram (top) Zirconia (bottom)	Manufacturer's instructions
Group 4	Zirconia (top) 3M Lava Ceram (bottom)	Manufacturer's instructions
Group 5	Vita VM9 (top) Zirconia (bottom)	Manufacturer's instructions
Group 6	Zirconia (top) Vita VM9 (bottom)	Manufacturer's instructions

The two experimental groups, for each ceramic, were prepared as described previously and distributed in the way that is followed described:

Groups 1 and 2: Preparation according to manufacturer's instructions

The preparation of the specimens was performed according to manufacturer's instructions for NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), through the method previously described. No surface treatment was made in any of Group 1 or 2 specimens (Figure 11).

Groups 3 and 4: Preparation according to manufacturer's instructions

The preparation of the specimens was performed according to manufacturer's instructions for the ceramic Lava™ Ceram veneer ceramic (3M™ ESPE™, Germany), through the method previously described. No surface treatment was made in any of Group 3 or 4 specimens.

Groups 5 and 6: Preparation according to manufacturer's instructions

The preparation of the specimens was performed according to manufacturer's instructions for the ceramic Vita® VM®9 veneer ceramic (Vita® Zahnfabrick, Germany), through the method previously described. No surface treatment was made in any of Group 5 or 6 specimens.

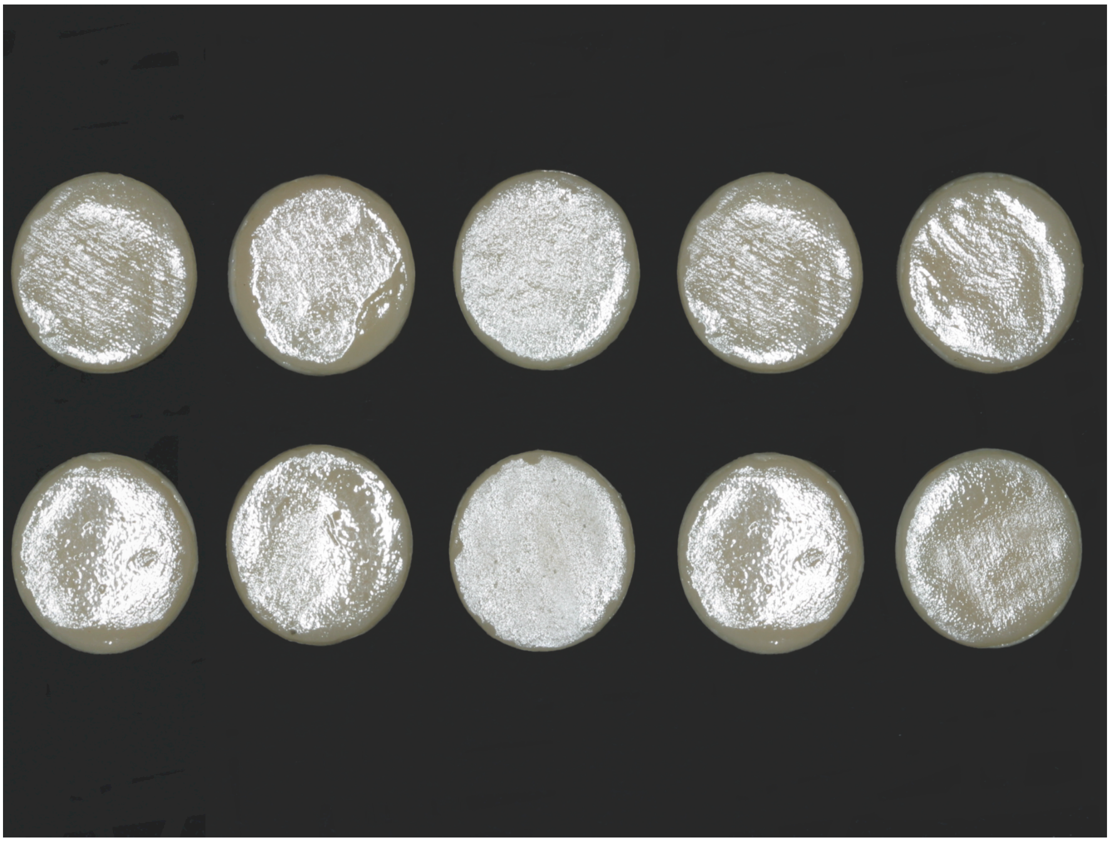

Figure 11. Test specimens of group 1 consisting of NobelRondo Zirconia veneer ceramic and Zirconia ceramic framework

Biaxial flexural strength test

The load to fracture was measured with the piston-on-three-ball test method by using a Universal Instron testing machine (Model TT-BM Instron Corp., Canton, MA) according with the standard ISO/DIS 6872 for dental ceramics (ISO/DIS 6872: 1995). The disc specimens were first positioned in the sample holder on top of the three supporting balls symmetrically spaced. The three balls, of 1.5 mm diameter were equidistant around a circle with a radius of 6mm. In groups 1, 3 and 5 the load was applied over the feldspathic veneer dental porcelain while the 3Y-TZP ceramic core rested on the three supporting balls. In groups 2, 4 and 6 the position of the feldspathic veneer dental porcelain and the 3Y-TZP ceramic core was reversed. The purpose was to determine the influence of the feldspathic veneer ceramic on the internal or external origin of the fracture.

The load was applied at the center of the top surface through the flat tip of a cylindrical piston (diameter 0.7mm) at a cross-head speed of 0.5 mm/min until fractured had occurred. The test was conducted at room conditions (22 ± 1°C, and 60% ± 5% relative humidity).

Figure 12. Disc-shaped test specimen positioned over testing apparatus before fracture strength measurement. The load was applied at the center of the top surface through the flat tip of a cylindrical piston (diameter 0.7 mm) at a cross-head speed of 0.5 mm/min until fracture.

Calculations to determine biaxial flexural strength

The recorded maximum resistance to fracture (N) was used in conjunction with the following formula (ASTM F 394-78, 1996), which resulted in the biaxial flexural strength for each specimen.

$$S = - 0.2387\, P(X - Y)/d^2$$

Where S represents the maximum center tensile stress/flexural strength (MPa), P is the maximum resistance to fracture (N) and *d* is the specimen thickness at fracture origin (mm). X and Y where determined as follows:

$$X = (1+n)\,ln(B/C)^2 + [(1- n)/2](B/C)^2$$

$$Y = (1+n)\,[1+ln\,(A/C)^2] + (1- n)(A/C)^2$$

where *n* represents the Poisson´s ratio, A is the radius of the supporting circle (mm), B is the radius of the tip of the piston or radius of the loaded area (mm) and C is the radius of the specimen (mm). In this study, $n = 0.25\text{-}0.31$, A = 5 mm, and B = 0.7 mm. It needs to be stated that the Poisson´s ratio used in these calculations was based on an adjustment between the Poisson´s ratio of the feldspathic veneer dental ceramics (0.25) and the Poisson´s ratio of the 3Y-TZP ceramic (0.31) having in attention their thickness; which already been established by different authors (White *et al*., 2005).

Statistical analysis

The statistical analysis of the results was performed through descriptive and inference methods. The descriptive statistic of the maximum resistance to fracture of all group specimens was performed according to common methods determining the mean, median, standard deviation, standard error, variance, and the minimum and maximum values for all studied group specimens.

The influence of the feldspathic veneering ceramic material on the biaxial flexural strength was analyzed by using two-way ANOVA followed by

Fisher's PLSD (protected least significant difference) test to identify the source of differences with an overall 5% level of error. In the present study the Fisher's (PLSD) test was chosen because it is usually less conservative than other post hoc all-pairwise tests. Therefore, if a significant ANOVA result is found, then Fisher's (PLSD) is more likely than some other tests to identify a significant pairwise comparison.

Pairwise comparisons among each group of ceramic and between ceramic groups were made using the Student’s t-test for multiple comparisons in conjunction with an overall 5% level of Type I error.

In addition, the Weibull moduli were calculated for biaxial flexural strength data in the same method previously described. Weibull moduli are calculated by plotting ln (1/1 – F) versus ln (s), where:

$Pf=(i - 0.5)/N$

Where i = rank of a samples in terms of strength (i = 1 for the lowest strength sample), N = total number of samples, and s = strength of sample i.

The slope of the line is then determined by use of linear regression. The resulting slope is the Weibull modulus.

3- Mode of fracture study

Type of research

This was an experimental *in vitro* study using Zirconia ceramic discs stratified with feldspathic dental veneering ceramic used to layer Zirconia frameworks in the fabrication of prosthodontic fixed restorations.

Dependent variable

Mode of fracture of the bilayered feldspathic dental veneering ceramic/Zirconia ceramic framework discs.

Independent variable

Feldspathic dental veneering ceramic employed to stratify Zirconia 3Y-TZP subtracts of three (3) commercially brands: 1) NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), 2) Lava™ Ceram veneer ceramic (3M™ ESPE™, Germany), and 3) Vita® VM®9 veneer ceramic (Vita® Zahnfabrick, Germany).

Design of the study

A group of sixty (60) disc-shaped specimens 2.2 (± 0.1) mm thick and 12.7(± 0.1) mm in diameter were fabricated and used in this study. The disc specimens were composed of a framework of 3Y-TZP produced by a CAD-CAM technique. Feldspathic veneering dental ceramic used to stratify Zirconia 3Y-TZP frameworks were from 3 commercially available brands: 1) NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), 2) Lava™ Ceram veneer ceramic (3M™ ESPE™, Germany), and 3) Vita® VM®9 veneer ceramic (Vita® Zahnfabrick, Germany). The feldspathic veneering dental ceramics used in the study were the NobelRondo™ Zirconia veneer ceramic Base Liner (Lot No. 0508) and NobelRondo™ Zirconia veneer ceramic Dentine A2 (Lot No. 0709); the Lava™ Ceram veneer ceramic Frame-Work Modifier (Lot No. 167786) and Lava™ Ceram veneer ceramic Dentine A2 (Lot No 189943); and the Vita VM®9 veneer ceramic Base Dentine (Lot No. 7933) and Vita VM®9 veneer ceramic Dentine A2 (Lot No. 7565). All disc specimens were fabricated accordingly to the specific manufacturer's recommendations for each ceramic.

Preparation of the specimens

Specimens were prepared according to ISO/DIS 6872: 1995 (three-point and biaxial flexural strength) (ISO/DIS 6872: 1995). Sixty (60) 3Y-TZP discs with 1.1 (± 0.1) mm thick and 12.7 (± 0.1) mm in diameter were produced by CAD/CAM technology (Nobel Biocare™ AB, Sweden) and used to veneer the 3 brands of feldspathic veneering dental ceramics. The discs were obtained from the manufacturer without any surface treatment and in the same condition that 3Y-TZP substructures are delivered to the commercial laboratory. Twenty bilayered specimens of each ceramic were fabricated using a separable steel mold. The mixing liquid and the opaque ceramic were combined in proportions recommend by the manufacturers to form a sticky slurry, which was applied over the 3Y-TZP discs then vibrated. The firing of the specimens was performed in a programmable and calibrated vacuum ceramic furnace (Programat P500, Ivoclar Vivadent AG, Lichenstein) according to the recommendations of the manufacturers. After the first firing the bilayered specimens were prepared by veneering the core materials with the feldspathic ceramics. The discs were put in a steel mold and the ceramic powders were combined in proportions recommend by the manufacturers to form a sticky slurry, which was vibrated and packed. Excess liquid was wicked off of the surface with absorbent tissue paper. The firing of the specimens was performed in the same programmable and calibrated vacuum ceramic furnace according to the recommendations of the manufacturers. After the first firing, additional porcelain was added and fired to compensate for the shrinkage resulting from the first sintering. The fired porcelain discs were examined with a stereomicroscope (Nikon SMZ-U, Tokyo, Japan) at a magnification X75 to evaluate the specimens for small cracks or flaws. Specimens displaying visible defects were replaced. All specimen surfaces were then polished with SiC disks (grit-silicon-carbide P220, P500, P1200 - Ultra-Prep, Buehler Ltd., Lake Bluff, IL, USA) in a mechanical grinder (Ecomet® 3, Buehler Ltd., Lake Buff, IL, USA) according to ISO 6344-1: 1998 (ISO/DIS 6344-1: 1998). This procedure was repeated until samples with a thickness and diameter of approximately 2.2 (± 0.1) mm e 12.7 (± 0.1) mm respectively were obtained. A special stainless steel holder was used to ensure accuracy of thickness and parallelism of the surfaces during grinding and polishing. As required by the ISO Standard the two faces of the specimens did not differ more than 0,05mm in parallelism. The specimens' dimensions were measured with a digital micrometer (Digimatic Caliper Seriates 500, Mitutoyo America Corporation, Dawn, IL, USA) to ensure the exact thickness and diameter.

Finally, all specimens were cleaned in an ultrasonic bath (Eurosonic® 4D, Euronda, Vicenza, Italy) with distilled water for 15 minutes before auto glazing according to the recommendations of the manufacturers in the ceramic furnace. After the glazing procedure, all specimens were again measured with the same digital caliper. The diameter and thickness of the ceramic discs were measured by the micrometer to the nearest 1/100 mm at five randomly selected locations prior to the test locations.

Distribution of the specimens

The sixty (60) specimens were randomly assigned to the six groups, two groups for each ceramic, each group consisting of ten specimens. The order in which the specimens were treated was random, to avoid a possible bias due to any particular sequence of treatment.

The two experimental groups, for each ceramic, were prepared as described previously and distributed in the way that is followed described:

Groups 1 and 2: Preparation according to manufacturer's instructions

The preparation of the specimens was performed according to manufacturer's instructions for the ceramic NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), through the method previously described. No surface treatment was made in any of Group 1 or 2 specimens.

Groups 3 and 4: Preparation according to manufacturer's instructions

The preparation of the specimens was performed according to manufacturer's instructions for the ceramic Lava™ Ceram veneer ceramic (3M™ ESPE™, Germany), through the method previously described. No surface treatment was made in any of Group 3 or 4 specimens.

Groups 5 and 6: Preparation according to manufacturer's instructions

The preparation of the specimens was performed according to manufacturer's instructions for the ceramic Vita® VM®9 veneer ceramic (Vita® Zahnfabrick, Germany), through the method previously described. No surface treatment was made in any of Group 5 or 6 specimens.

Mode of fracture evaluation

The maximum load at failure was measured with the piston-on-three-ball test method by using a Universal Instron testing machine (Model TT-BM Instron Corp., Canton, MA) according with the Standard ISO/DIS 6872 for dental ceramics (ISO/DIS 6872: 1995). The disc specimens were first positioned in the sample holder on top of the three supporting balls symmetrically spaced. The three balls, of 1.5 mm diameter, were equidistant around a circle with a radius of 6 mm. In groups 1, 3 and 5 the load was applied over the feldspathic veneer dental porcelain while the 3Y-TZP ceramic core rested on the three supporting balls. In groups 2, 4 and 6 the position of the feldspathic veneer dental porcelain and the 3Y-TZP ceramic core was reversed. The purpose was to understand the influence of the feldspathic veneer ceramic on the internal or external origin of the fracture and respective mode of fracture.

The load was applied at the center of the top surface through the flat tip of a cylindrical piston (diameter 0.7 mm) at a cross-head speed of 0.5mm/min until fractured had occurred. The test was conducted at room conditions (22 ± 1°C, and 60% ± 5% relative humidity).

After fracture testing procedure, all specimens were analysed with a stereomicroscope (Nikon SMZ-U, Tokyo, Japan) under X75 magnification using a halogen lamp (Nikon 26120, Tokyo, Japan) as external illumination source without any special surface coatings. This observation was performed for all specimens of the six experimental groups with the purpose of characterizing the origin and mode of fracture. Subsequently two representative specimens and several fractured surfaces from each group were selected and micrographs were taken at the same X75 magnification, using a copulated camera (Nikon FX-35DX, Tokyo, Japan) to the stereomicroscope.

Morphologic characterization of the different types of fractures registered at the Zirconia core/feldspathic veneering ceramic was performed using scanning electron microscopy. Six representative specimens, two from each group, were randomly selected and mounted on aluminium stubs and coated with gold. The specimens were examined with a scanning electron microscope (SEM) (Amray 1820, Bedford, MA, USA) (Figure 13). Photographs at different magnifications were made of representative areas for each specimen.

Figure 13. Amray 1820, scanning electron microscope

CHAPTER 4

RESULTS

1- Surface finish and heat treatment study

In this investigation the influence of the surface finish and heat treatment on the biaxial flexural strength of three different feldspathic veneering ceramics were studied. The mean biaxial flexural strength values and standard deviations for each group of specimens are shown in Table 3 and Figure 14.

Examination of the standard deviations revealed considerable heterogeneity in variability of the outcome among the eighteen groups, with the largest group variance being over thirty-fold that of the group with the least variation.

A two-way ANOVA was carried out to determine if there was any statistical significant difference between the two major effects; the "ceramic" factor" and the "surface treatment" factor on the load to fracture of the test specimens. The power values for analyzing the major factor "ceramic" and "surface treatment" were high enough, (1 and 0.998) respectively, so that the project design was considered valid. The two-way ANOVA results demonstrated that both factors had statistically significant effect on load to fracture of the test specimens ($p<0.0001$).

When analyzing the source of differences between the surface treatments Fisher's protected least significant difference (PLSD) test showed statistically significant differences between the grinding groups and all other groups. Strong evidence was found that the variance of the groups corresponding to G groups was significantly smaller than that of the variance of the CN groups ($p<0.0001$). There was also evidence of differences in variance between the G groups and the GG groups ($p=0.0007$), the G groups and the GP groups ($p<0.0001$), the G groups and the GPG groups ($p=0.0007$) and finally the G groups and the PG groups ($p=0.0027$). The results obtained in the CN group differ significantly from the G group ($p<0.0001$), has previously mentioned and from the PG group ($p=0.0261$). However the results obtained in the CN group could not be said to differ significantly from those obtained in the GG group ($p=0.0712$) nor did the results differ for the two groups corresponding to the GP ($p=0.3443$) and GPG ($p=0.0702$). No other comparisons among the four remaining groups were significant; i.e., variability appeared to be similar in the GP, GG, GPG and GP groups.

When analyzing the source of differences between the ceramics, Fisher's protected least significant difference (PLSD) between the Lava™ Ceram ceramic and the other two ceramics; NobelRondo™ ceramic ($p<0.0001$) and Vita® VM®9 ($p<0.0001$). Fisher's test indicated also that there was no strong evidence of significant heterogeneity of variances ($p=0.6665$) among the NobelRondo™ and Vita® VM®9 groups.

Table 3 Descriptive statistic by group for load fracture MPa

Load Fracture MPa		Mean	STD	N	Min	Max	Range	Median
Total		91,61	15,71	180	59,46	148,25	88,79	90,54
Nobel Rondo	CN	105,60	22,24	10	75,21	148,25	73,04	103,90
	G	73,49	15,17	10	59,46	103,24	43,78	67,12
	GG	80,44	14,81	10	60,42	104,50	44,08	80,19
	GP	98,75	13,81	10	80,76	122,77	42,01	98,18
	GPG	88,19	15,96	10	63,44	109,91	46,47	87,98
	PG	82,65	14,50	10	62,74	108,61	45,87	78,80
Lava Ceram	CN	98,79	14,22	10	75,50	128,08	52,58	100,23
	G	88,95	13,29	10	62,69	105,09	42,40	89,87
	GG	106,76	19,18	10	73,83	140,76	66,93	106,74
	GP	100,20	16,41	10	76,32	123,63	47,31	102,21
	GPG	104,08	9,62	10	87,39	118,22	30,83	105,75
	PG	98,20	13,88	10	78,76	118,94	40,18	100,51
Vita VM9	CN	91,12	5,45	10	81,10	96,09	14,99	93,74
	G	78,90	8,08	10	68,10	94,01	25,91	77,74
	GG	89,72	6,55	10	78,66	98,69	20,03	90,84
	GP	86,85	5,83	10	78,40	96,34	17,94	86,48
	GPG	84,59	5,74	10	76,73	93,47	16,74	85,17
	PG	91,69	6,01	10	83,89	101,09	17,20	93,07

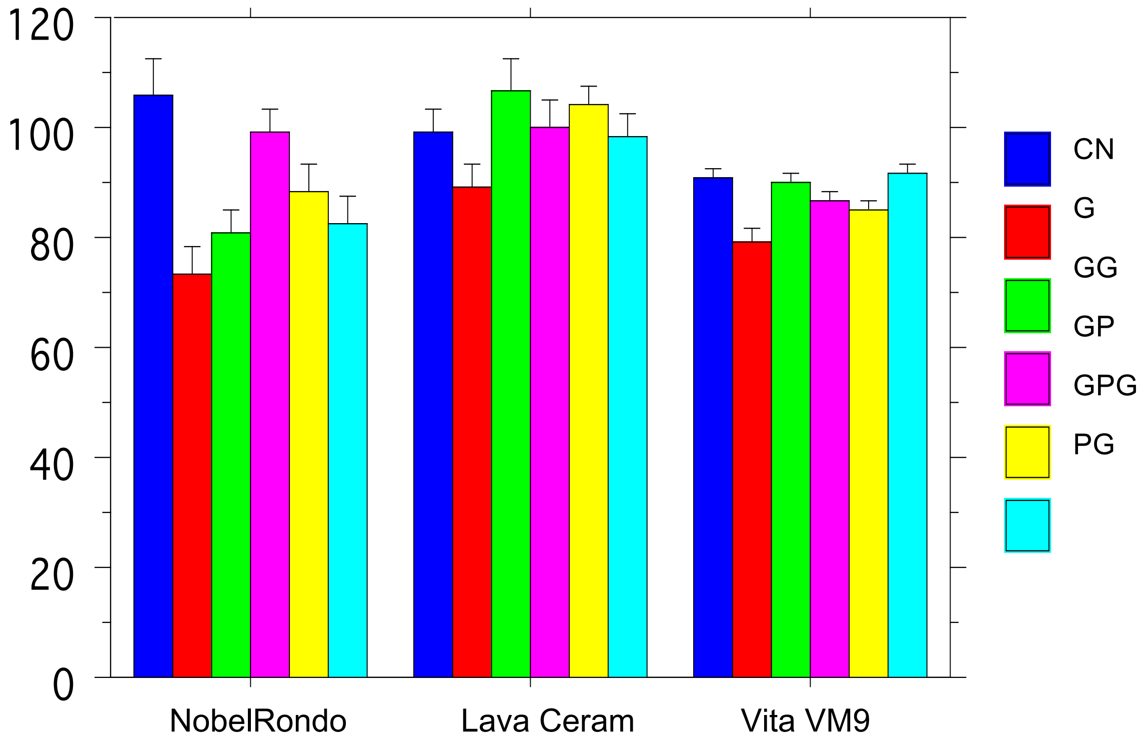

Figure 14. Biaxial flexural strength mean values (MPa) of feldspathic veneering ceramics specimens versus surface treatments

A one-way ANOVA test was run for each ceramic group to determine if there was any statistical difference between the surface treatments of each ceramic on the fracture load of the test specimens. The results for variance homogeneity were highly significant (p=0.0004) for the NobelRondo™ ceramic and for the Vita® VM®9 ceramic (p=0.0002). There were no significant differences for variance homogeneity for the Lava™ Ceram ceramic groups (p=0.1429). The power values for analyzing the surface treatment factor were high enough, (0.987 NobelRondo™, 0.993 Vita® VM®9 and 0.548 Lava™ Ceram), so that the project design was valid.

The results of all possible pairwise comparisons of the treatment groups for the NobelRondo™ ceramic after Fisher's protected least significant difference (PLSD) adjustment for multiple comparisons are summarized in Figure 15. As seen in Figure 15, the distribution of fracture loads could not be said to differ among the three surface treatment groups with the highest outcomes: CN, GP and GPG. The CN and GP groups clearly differed in distribution of percentage load fracture from the three groups with the lowest distribution of outcome the PG, the GG and the G groups. In addition, the data provided evidence that the fracture load in the highest group (that associated with the control group) was significantly greater than that obtained by the GPG, and that the results obtained by this group were significantly better than those obtained under grinding conditions. No other pairwise comparisons were statistically significant after adjustment for multiple comparisons; the results obtained in G group could not be said to differ significantly from those obtained in the GG and PG groups, nor did the results differ between this two groups. The two groups with the highest fracture loads the CN and the GP group were not statistically different.

The results of all possible pairwise comparisons of the treatment groups for the Vita Lava™ Ceram ceramic after Fisher's protected least significant difference (PLSD) adjustment for multiple comparisons are summarized in Figure 16. As seen in Figure 16 the distribution of fracture loads could not be said to differ among the five surface treatment groups with the lowest outcomes: G, PG, CN, GP and GPG. In addition the data provided evidence that the fracture load in the two highest groups (that associated with the GG group and the GPG group) was significantly greater than that obtained by the grind group. No other pairwise comparisons were statistically significant after adjustment for multiple comparisons; the results obtained in the two groups with the highest fracture loads the GG and the GPG groups were not statistically different, nor did the results differ between these two groups and the GP, the CN and the PG groups.

The results of all possible pairwise comparisons of the treatment groups for the Vita® VM®9 ceramic after Fisher's protected least significant difference (PLSD) adjustment for multiple comparisons are summarized in Figure 17. As seen in Figure 17 the G group clearly differed in distribution of percentage load fracture from all other groups. In addition the data provided evidence that the fracture load in the two highest groups (that associated with the PG group and the CN group) was significantly greater than that obtained by the GPG group. No other pairwise comparisons were statistically significant after adjustment for multiple comparisons; the results obtained in the PG group could not be said to differ significantly from those obtained in the CN, GG and GP groups, nor did the results differ between the GG and the GP and GPG groups. The two groups with the highest fracture loads the CN group and the PG group were not statistically different.

The analysis of the Weibull modulus results (Table 4) indicated that, for the three types of ceramics tested, leaving the surface in a grinding condition influence the flaw distribution, since the values for the three grinding groups are smaller than for all other groups of specimens in each ceramic. This is especially evident for the Vita® VM®9 ceramic. For the NobelRondo™ ceramic the Weibull modulus values varied with different surface treatments, showing higher values for GP group and similar values for the GG, GPG and PG groups and the CN group. The Weibull modulus values for the Lava™ Ceram ceramic showed that the surface treatment groups with simultaneous polishing and glazing had the highest results (GPG and PG groups) and the groups that had been grinded had lower values (G, GG and GP). The CN group revealed intermediate Weibull modulus values. The Vita® VM®9 ceramic groups recorded similar values when compared to the other ceramic groups. The CN group showed the highest value. All other surface treatments showed similar results except, like previously stated, the G group.

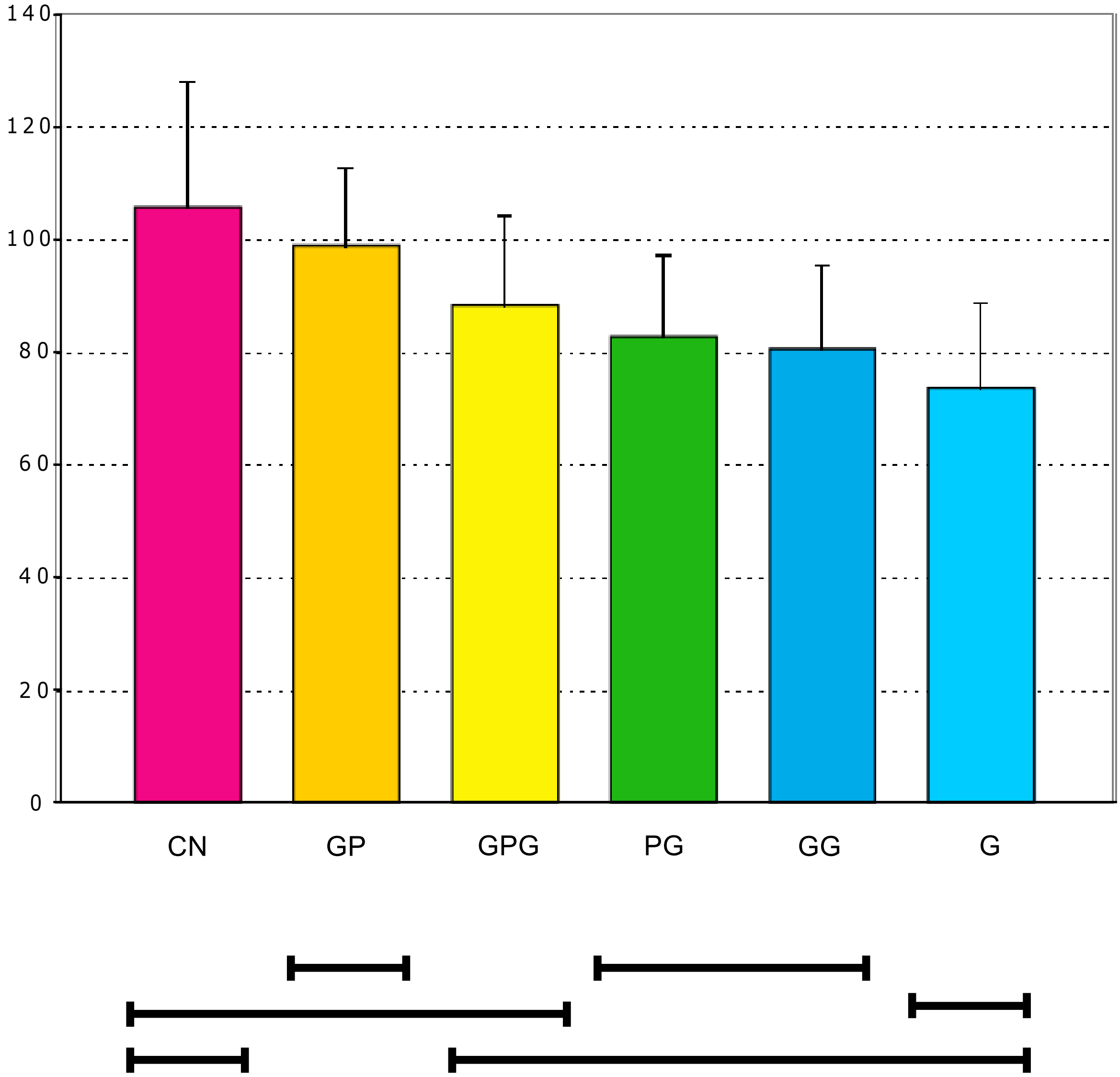

Figure 15. Biaxial flexural strength mean values (MPa) of NobelRondo feldspathic veneering ceramic specimens versus surface treatments with Fisher's grouping indicated.

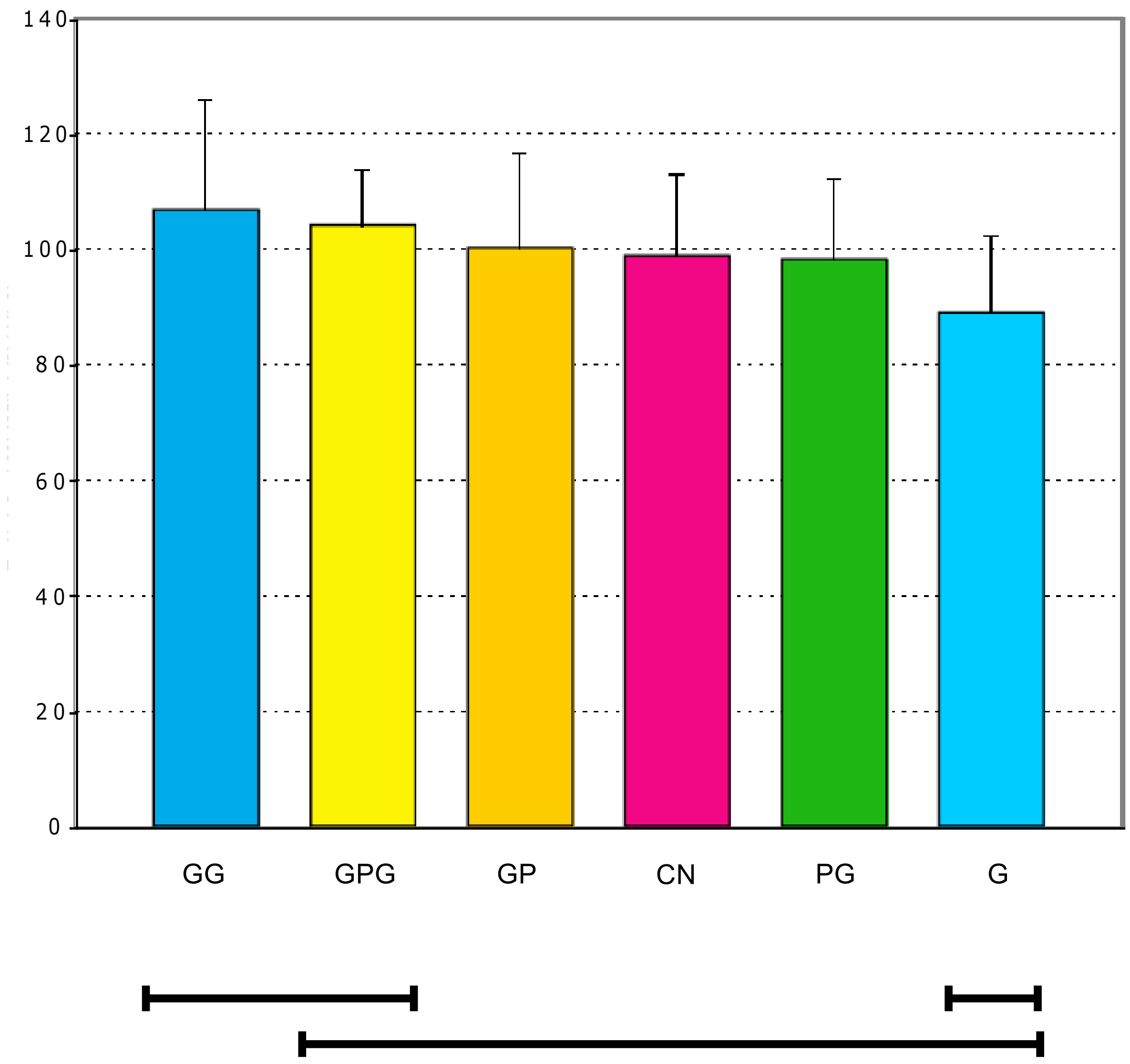

Figure 16. Biaxial flexural strength mean values (MPa) of Lava Ceram feldspathic veneering ceramic specimens versus surface treatments with Fisher's grouping indicated.

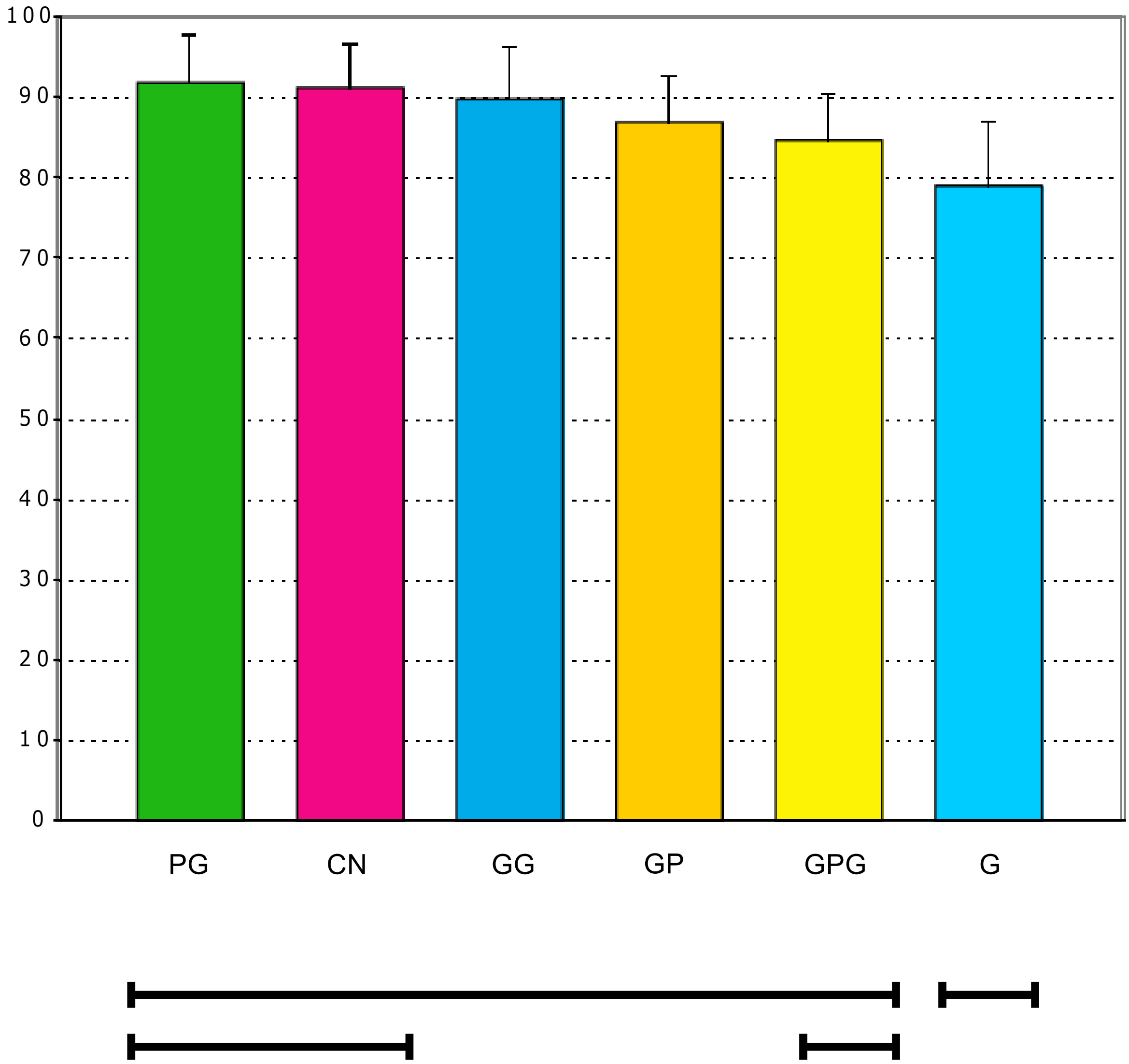

Figure 17. Biaxial flexural strength mean values (MPa) of Vita VM9 feldspathic veneering ceramic specimens versus surface treatments with Fisher's grouping indicated.

Table 4 Weibull modulus values for the feldspathic veneering ceramics versus surface treatments

Ceramic Material	Surface Treatment	Weibull modulus
Nobel NobelRondo	CN	6,688
Nobel NobelRondo	G	4,522
Nobel NobelRondo	GG	6,398
Nobel NobelRondo	GP	8,406
Nobel NobelRondo	GPG	6,447
Nobel NobelRondo	PG	6,809
3M ESPE Lava Ceram	CN	8,165
3M ESPE Lava Ceram	G	5,589
3M ESPE Lava Ceram	GG	7,329
3M ESPE Lava Ceram	GP	7,092
3M ESPE Lava Ceram	GPG	11,585
3M ESPE Lava Ceram	PG	9,187
Vita VitaVM9	CN	8,257
Vita VitaVM9	G	4,473
Vita VitaVM9	GG	7,242
Vita VitaVM9	GP	6,338
Vita VitaVM9	GPG	6,133
Vita VitaVM9	PG	6,427

2- Mode of fracture and biaxial flexural strength of bilayered feldspathic dental veneering ceramics/Zirconia core ceramic.

In this investigation the influence of the side tested under tensile stress (the Zirconia core or the feldspathic veneering ceramics) on the biaxial flexural strength of different feldspathic veneering ceramics/Zirconia core ceramic was studied. In addition the reliability and mode of fracture of different feldspathic veneering ceramics/Zirconia core ceramic was investigated. The mean biaxial flexural strength values and standard deviations for each group of specimens are shown in Table 5 and Figure 18. Examination of the standard deviations reveals considerable heterogeneity in variability of the outcome among the six groups, with the largest group variance being over hundred-fold that of the group with the least variation.

A two-way ANOVA was carried out to determine if there was any statistical significant difference between the two major effects; the “ceramic” factor and the “Zirconia location” factor on the load to fracture of the test specimens. The power values for analyzing the major factor “ceramic” and “Zirconia location” were high enough (1), so that the project design was valid. The two-way ANOVA results demonstrated that both factors had statistically significant effect on load to fracture of the test specimens ($p<0.0001$).

When analyzing the source of differences between the ceramics tested, Fisher’s protected least significant difference (PLSD) test showed statistically significant differences between the NobelRondo™ ceramic and the other two ceramics; Lava™ Ceram ceramic ($p<0.0001$) and Vita® VM®9 ($p<0.0001$). Fisher’s test indicated also that there was no strong evidence of significant heterogeneity of variances ($p=0.3148$) among the Lava™ Ceram and Vita® VM®9 groups. When analyzing the difference between which surface was placed under tensile stress the results of Fisher’s test showed that there was a statistical significant difference between both positions ($p<0.0001$).

An unpaired t-test was used to identify in each ceramic group if there was a significant difference between the locations of the surface that was placed under tensile stress (Zirconia core or veneering feldspathic ceramic) (Figures 19, 20 and 21). Strong evidence was found that the variance between groups corresponding to the Lava^{TM} Ceram and Vita$^{®}$ VM$^{®}$9 was significant ($p<0.0001$). However, the results obtained in the NobelRondo^{TM} groups could not be said to differ significantly ($p=0.4073$).

The analysis of the Weibull modulus results (Table 6) indicated that, for the three types of ceramics tested, when the Zirconia core ceramic was placed under tension, with the feldspathic veneering ceramics facing the loading piston, higher values were obtained demonstrating narrower flaw size distribution on the Zirconia core ceramic. The results of the Weibull modulus values for the NobelRondo^{TM} groups showed smaller values than those for the the Lava^{TM} Ceram and Vita$^{®}$ VM$^{®}$9 groups indicating a different distribution of flaws and homogeneity of the material, which result in different strength reliability.

Table 5 Descriptive statistic by group for load fracture MPa

Load Fracture MPa		Mean	STD	N	Min	Max	Range	Median
Total		404,26	139,76	60	215,86	689,31	473,45	437,85
NobelRondo	ZR Bottom	518,66	93,78	10	376,92	689,31	312,39	520,92
	ZR Top	476,93	124,03	10	221,62	580,78	359,16	514,79
Lava Ceram	ZR Bottom	494,89	66,04	10	406,56	575,35	168,79	488,51
	ZR Top	246,03	16,39	10	221,30	272,07	50,77	247,56
Vita VM9	ZR Bottom	447,44	100,84	10	215,86	542,83	326,97	455,25
	ZR Top	241,59	15,62	10	223,53	264,35	40,82	239,91

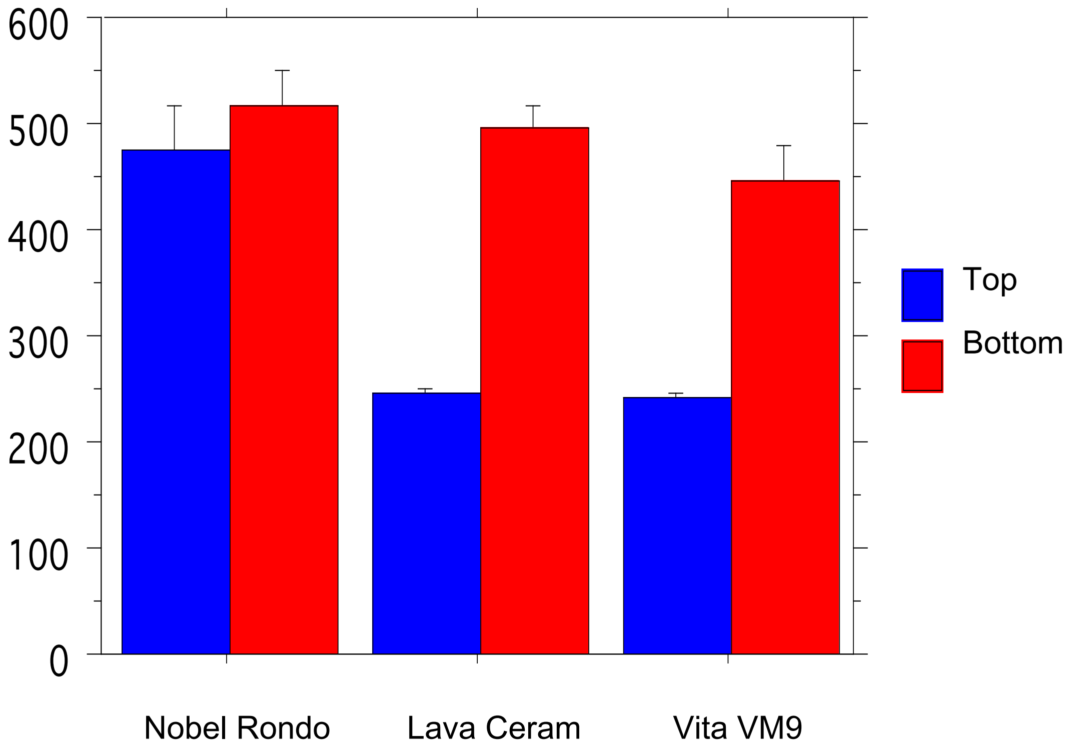

Figure 18. Biaxial flexural strength mean values (MPa) of feldspathic veneering ceramics/Zirconia core ceramic specimens versus side placed under tension.

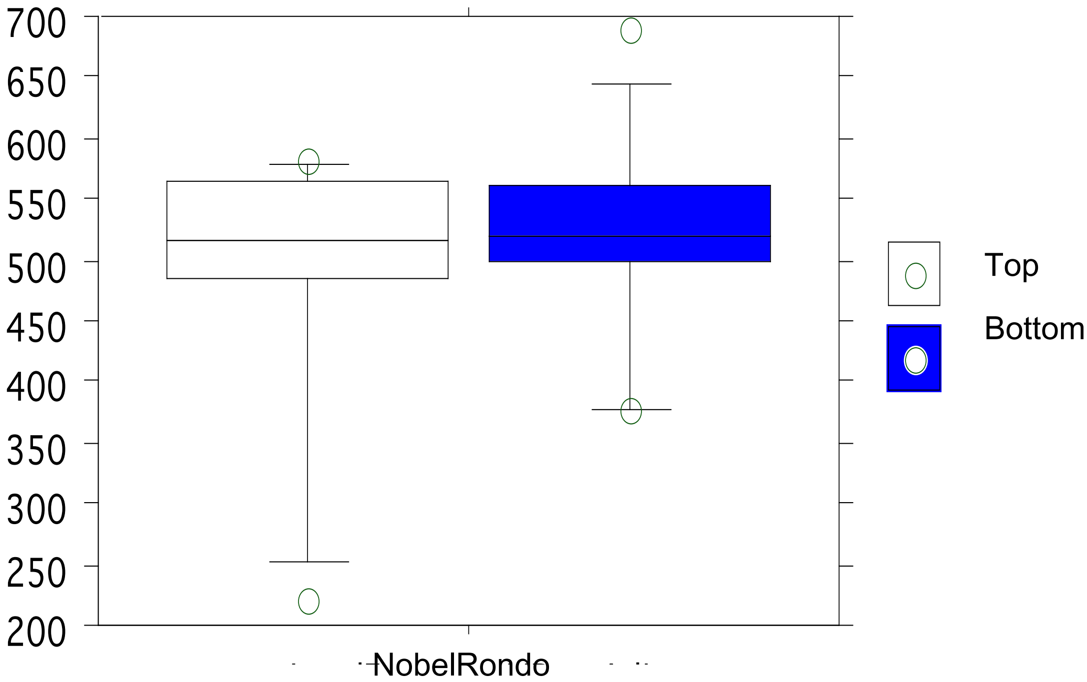

Figure 19. Box-and-whisker plots of biaxial flexural strength for the NobelRondo feldspathic veneering ceramic/Zirconia core ceramic specimens versus side placed under tension. The white color corresponds to the Zirconia core when placed on the top. The blue color when the Zirconia core is placed on the bottom.

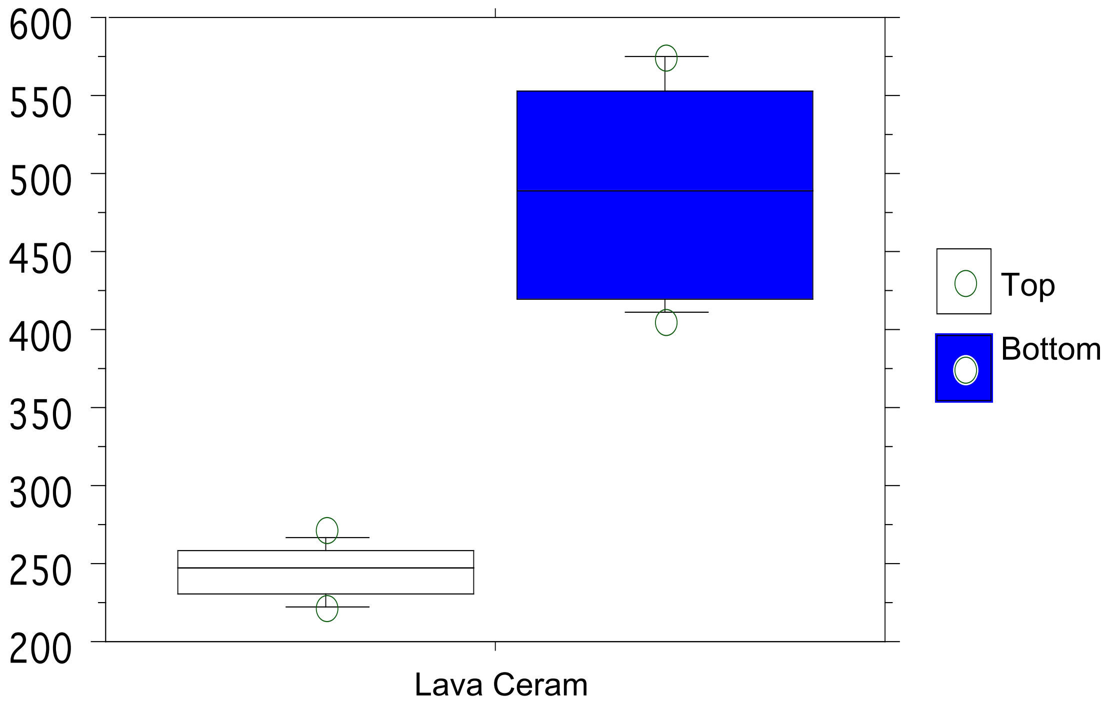

Figure 20. Box-and-whisker plots of biaxial flexural strength for the Lava Ceram feldspathic veneering ceramic/Zirconia core ceramic specimens versus side placed under tension. The white color corresponds to the Zirconia core when placed on the top. The blue color when the Zirconia core is placed on the bottom.

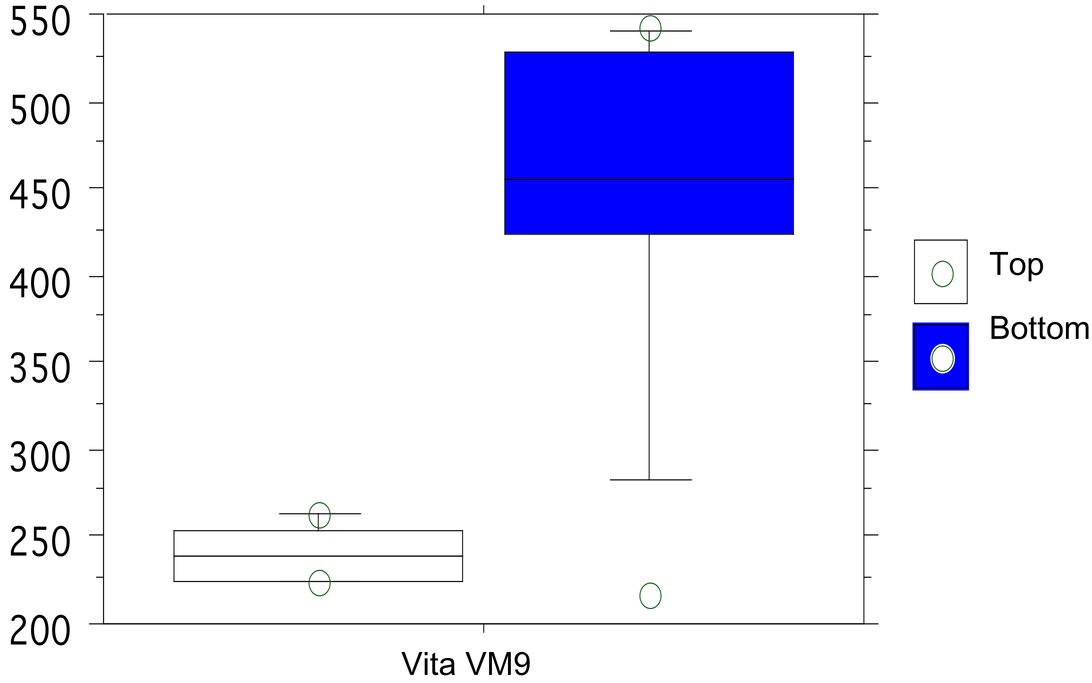

Figure 21. Box-and-whisker plots of biaxial flexural strength for the Vita VM9 feldspathic veneering ceramic/Zirconia core ceramic specimens versus side placed under tension. The white color corresponds to the Zirconia core when placed on the top. The blue color when the Zirconia core is placed on the bottom.

Table 6 Weibull modulus values for the feldspathic veneering ceramics/Zirconia core ceramic versus Zirconia location

Ceramic Material	Zirconia Location	Weibull modulus
Nobel NobelRondo	Bottom	15,378
Nobel NobelRondo	Top	5,227
3M ESPE Lava Ceram	Bottom	17,475
3M ESPE Lava Ceram	Top	8,819
Vita VitaVM9	Bottom	17,100
Vita VitaVM9	Top	7,615

Different modes of fracture were observed in the six groups of bilayered disc specimens according to which material was on the surface contacting the loading force. The fracture patterns and failure modes of the materials were classified in adhesive, cohesive and combined adhesive/cohesive failure.

In groups 1, 3 and 5 when the Zirconia core material was on the surface away from the load and the NobelRondo™, Lava™ Ceram and Vita® VM®9 feldspathic veneering ceramics were on the surface facing the loading piston, fracture tended to initiate at the top surface although the maximum peak of tensile stress was located on the bottom surface. Despite of the fact that the maximum peak of tensile stress was located on the surface of the core material, a Hertzian cone crack was consistently observed on the surface of the veneering ceramic facing the loading piston. This mode of fracture was observed in eight of the ten specimens of each group (Figures 22 and 23). In these specimens partial delamination of the veneering ceramic was present surrounding the Hertzian cone crack but there was no fracture of the Zirconia ceramic core that was maintained intact. In these specimens, two distinct failure loads were noted on the chart recorder. The first failure load occurred upon initial tensile failure; the second occurred on crack deflection and the initiation of delamination when the loading piston reached the Zirconia core. Cohesive failure was the prevalent type of failure in these specimens.

In the other six remaining specimens the extension of the cone crack to the interface caused deflection of the cracks laterally when the stronger Zirconia core was reached. In these specimens, two distinct failure loads were noted on the chart recorder. The first failure load occurred upon initial tensile failure; the second occurred on lateral crack deflection and the initiation of delamination. Catastrophic failure following initial tensile crack progression also involved veneering ceramic/Zirconia core interface as well as fracture of the core material as a result of crack progression within the Zirconia ceramic. Partial or complete delamination of the feldspathic veneering ceramic and opaque material was also present (Figures 24, 25, 26 and 27). A combination of adhesive/cohesive failure was the prevalent type of failure in these specimens.

Conversely, when the core material was on top (facing the loading piston) and the feldspathic veneering ceramic was on bottom, fracture tended to initiate at the bottom surface, accompanied by apparent delamination at the interface, followed by catastrophic failure with fracture of the Zirconia core material. In a few specimens, the fracture origin was localized at the veneering ceramic/Zirconia core material interface. In all specimens tested, two distinct failure loads were noted on the chart recorder as previously described. The first failure load occurred upon initial tensile failure and lateral crack deflection and the initiation of delamination and the second occurred on rupture of the Zirconia ceramic core. In all specimens a combination of adhesive/cohesive failure was the prevalent type of failure.

In all specimens of group 2 corresponding to the NobelRondo™ ceramic, the failure mode involved initial tensile crack progression with lateral and radial crack deflection involving the veneering ceramic/Zirconia core interface as well as partial delamination of the feldspathic veneering ceramic and opaque ceramic material. In all specimens catastrophic failure occurred with fracture of the Zirconia material in different areas as a result of crack propagation through the Zirconia ceramic core (Figures 28 and 29).

In groups 4 and 6 corresponding to the Lava™ Ceram and Vita® VM®9 ceramics 90% of specimens fractured in the middle as a result of crack progression within the Zirconia ceramic core. The initial tensile crack progressed with one or more lateral crack deflections involving the veneering ceramic/Zirconia core interface with partial delamination of the feldspathic veneering ceramic and opaque ceramic material. This was followed by rupture of the Zirconia core material (Figures 30, 31, 32 and 33). In 2 specimens, one in each group the fracture pattern was similar to that present in the specimens of group 2.

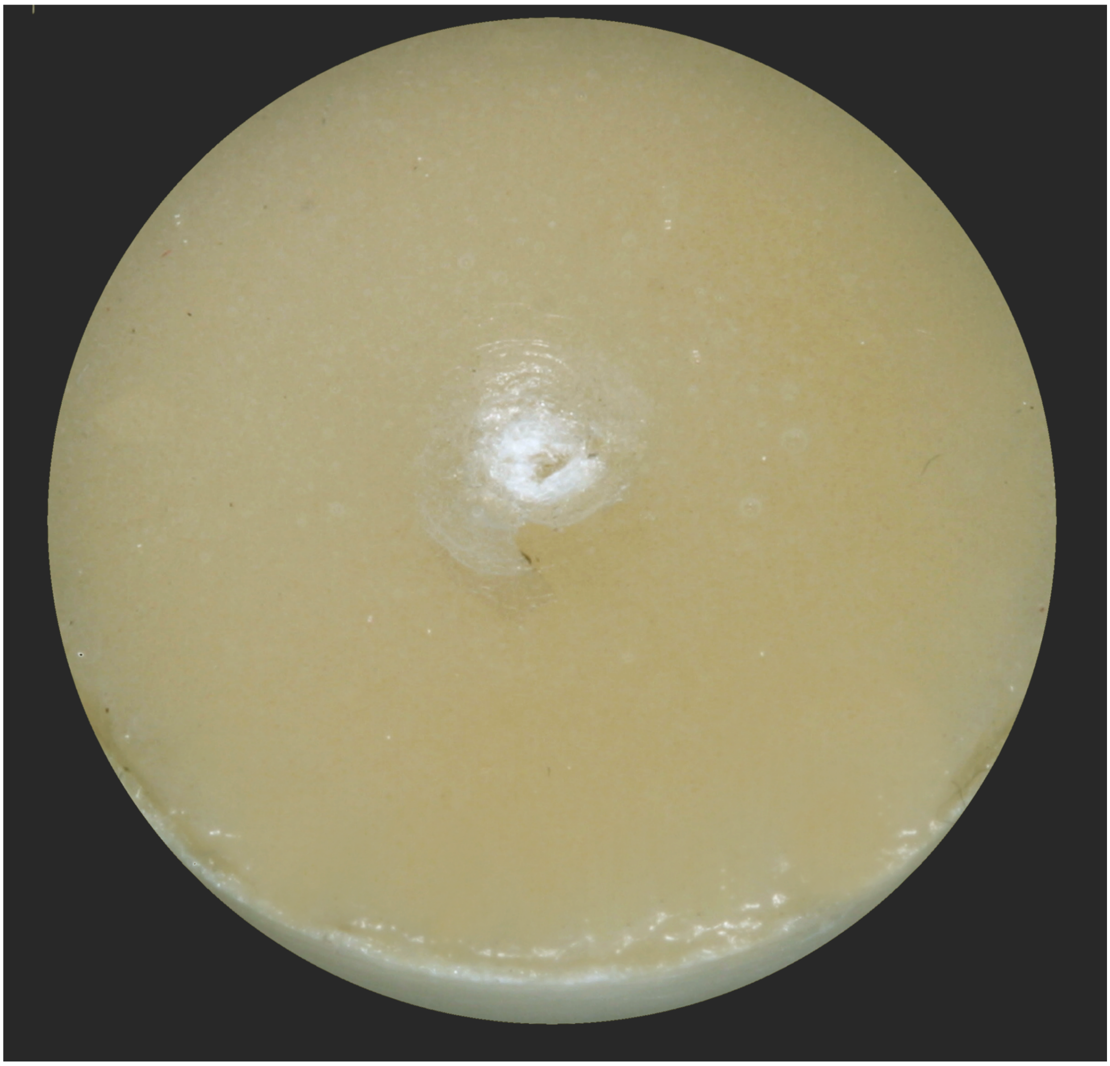

Figure 22. Most common representative fracture mode of bilayered feldspathic veneering ceramic/Zirconia core ceramic specimens of groups 1, 3 and 5. Hertzian cone crack and feldspathic veneering ceramic delamination with exposure of the Zirconia core are present in the loaded area.

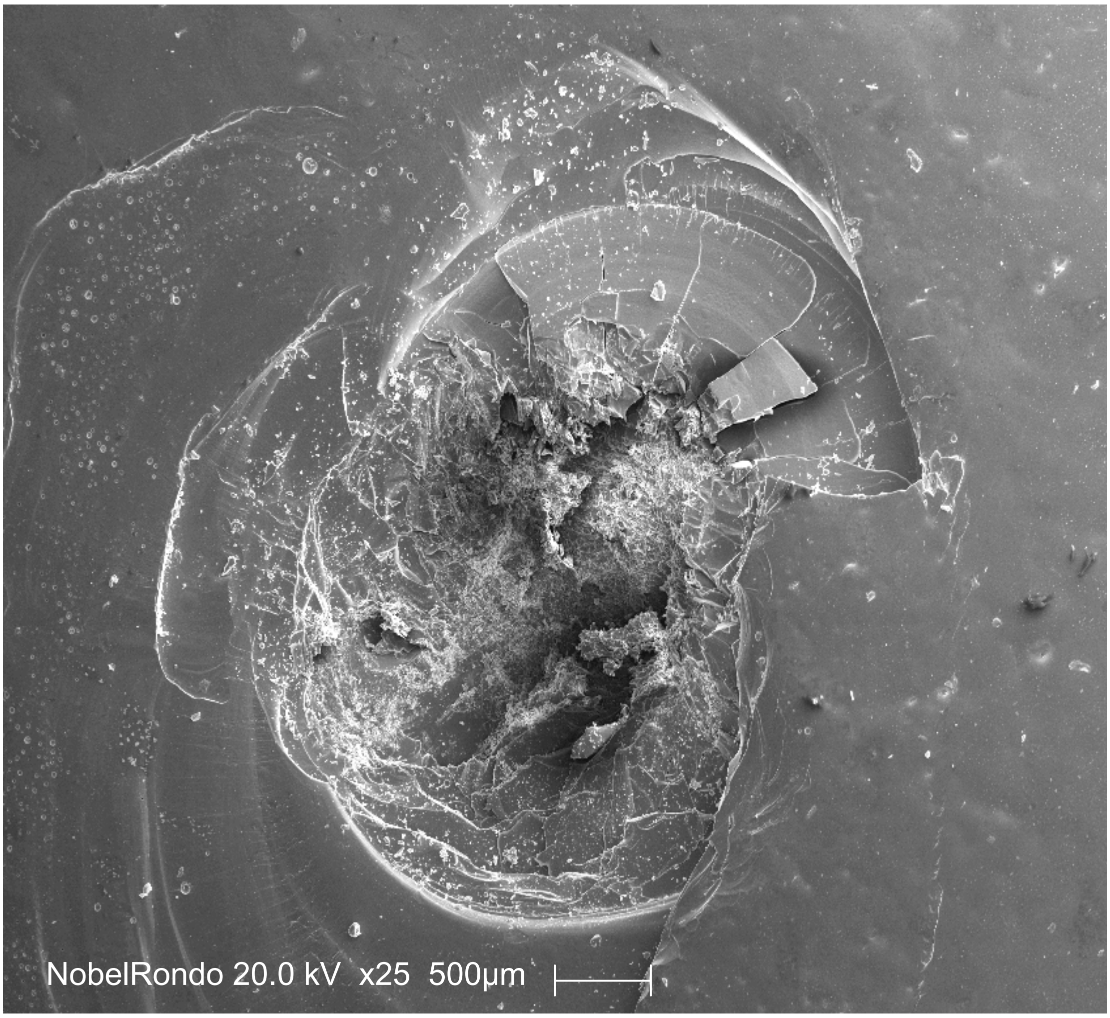

Figure 23. SEM micrograph of representative Hertzian cone crack observed on the surface of the bilayered feldspathic veneering ceramic/core Zirconia ceramic specimen of group 1.

Figure 24. Catastrophic failure of specimen of group 3 following initial tensile crack progression with lateral crack deflection involving veneering ceramic/Zirconia core interface as well as fracture of the Zirconia core and partial delamination of the feldspathic veneering ceramic.

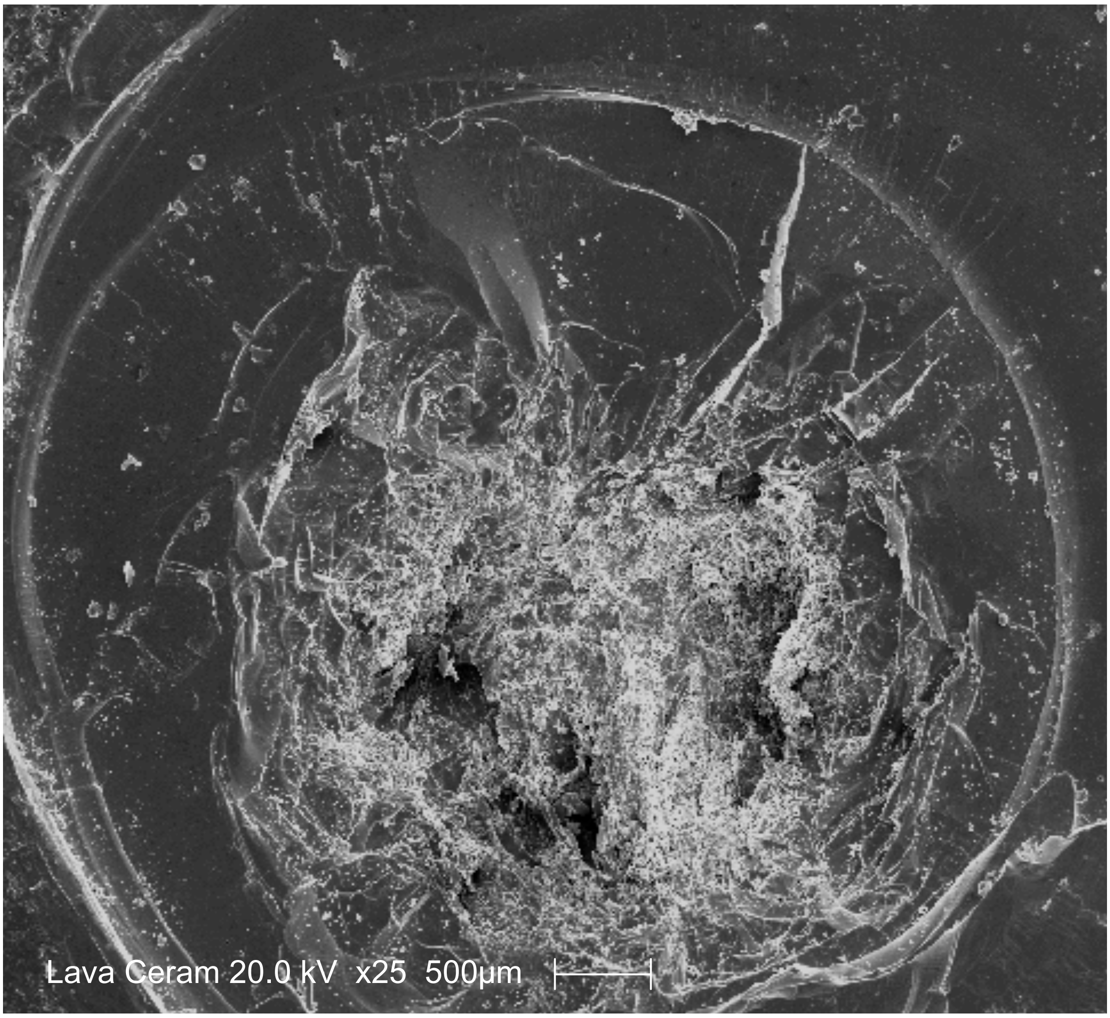

Figure 25. SEM micrograph of representative Hertzian cone crack and feldspathic veneering ceramic delamination following initial tensile crack progression with lateral and radial crack deflection involving veneering ceramic/Zirconia core interface observed on the surface of specimen of group 3.

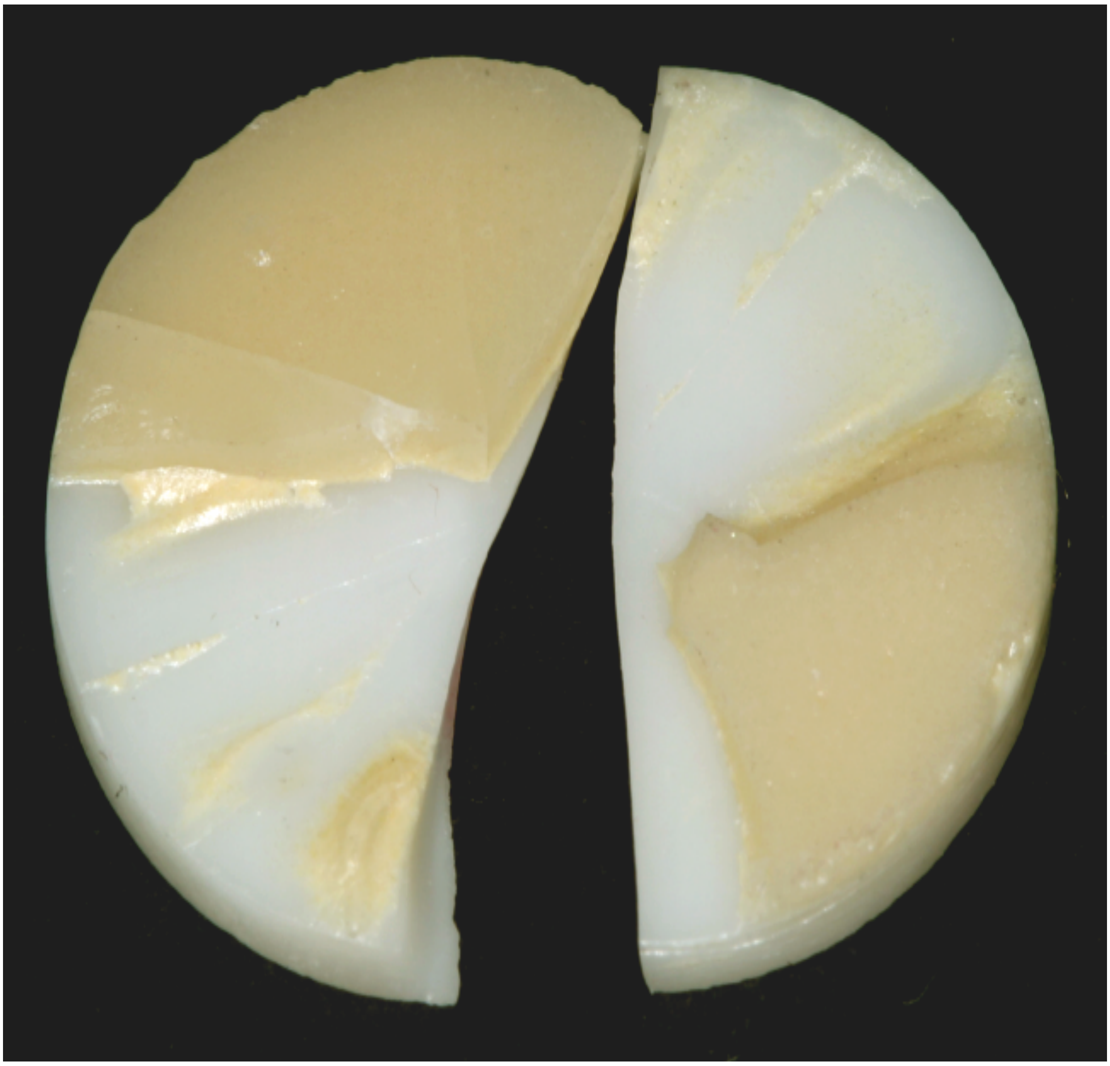

Figure 26. Catastrophic failure of specimen of group 5 following initial tensile crack progression involving veneering ceramic/Zirconia core interface as well as fracture of the Zirconia core with complete delamination of the feldspathic veneering ceramic and opaque material in some areas.

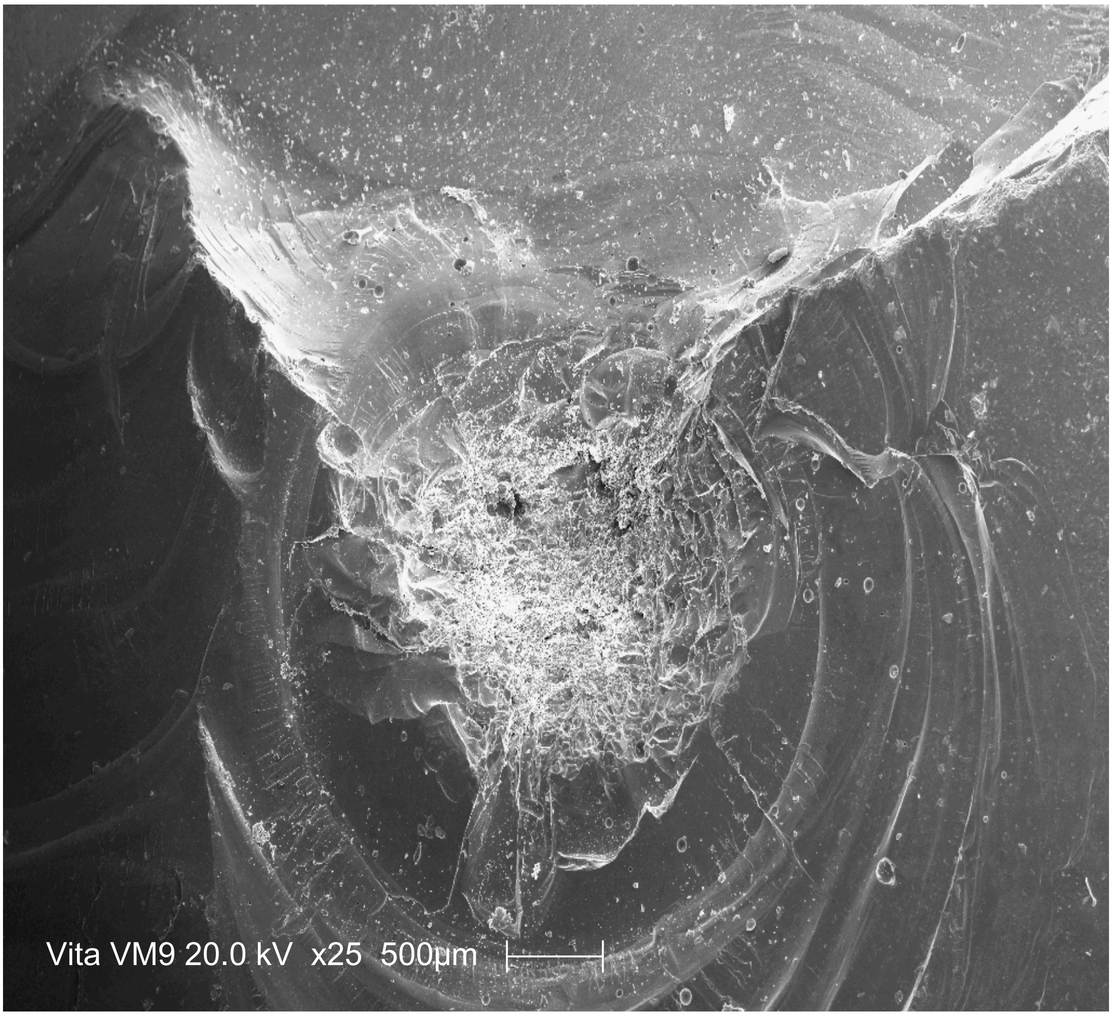

Figure 27. SEM micrograph of representative Hertzian cone crack and feldspathic veneering ceramic delamination with Zirconia core exposure observed on the surface of specimen of group 5.

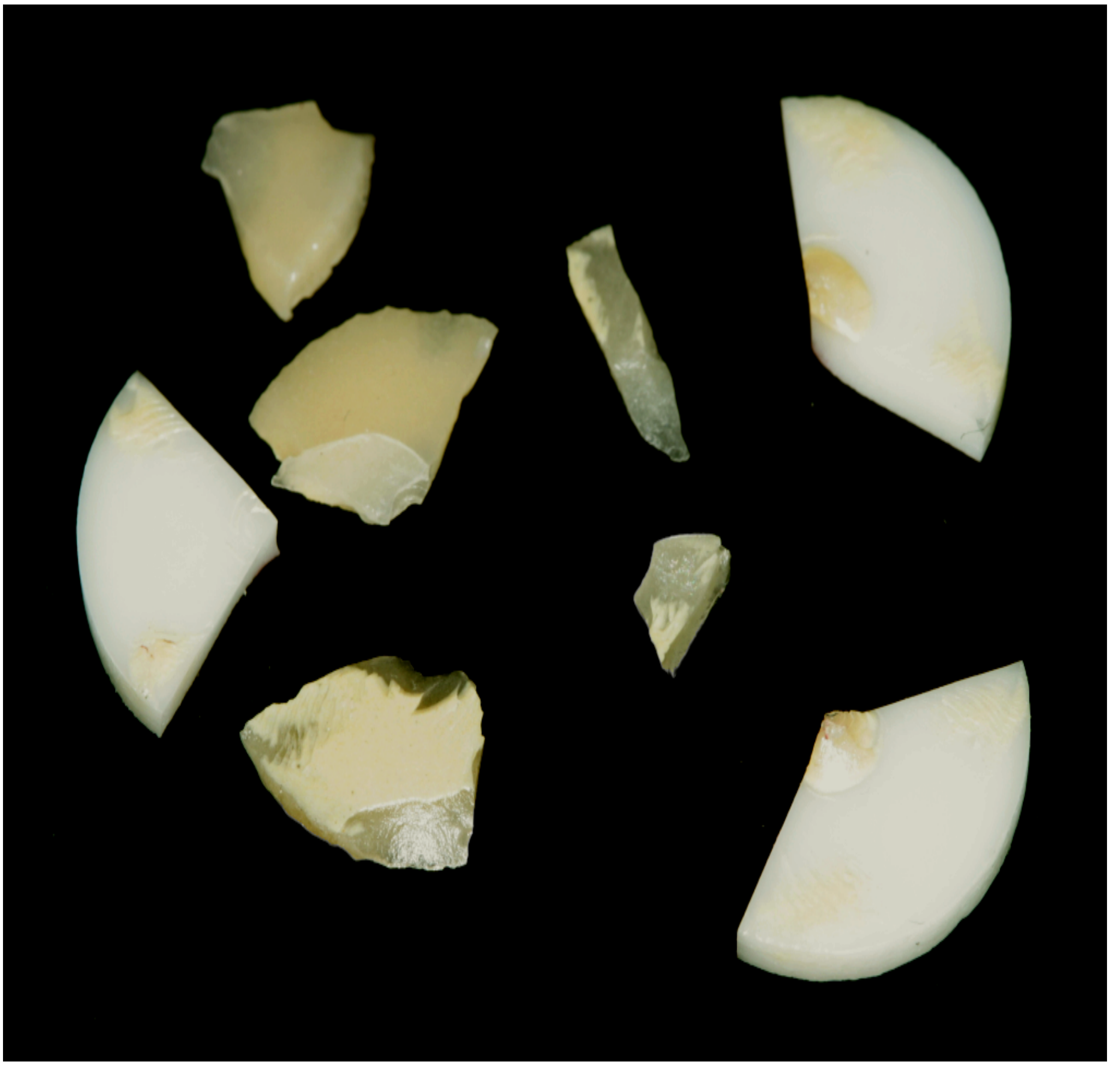

Figure 28. Representative fracture mode of specimens of group 2 with partial delamination of the feldspathic veneering ceramic and opaque ceramic material and catastrophic failure of the Zirconia ceramic core as a result of crack propagation.

Figure 29. SEM micrograph of representative tensile crack progression with feldspathic veneering ceramic delamination and Zirconia core exposure observed on the surface of specimen of group 2.

Figure 30. Catastrophic adhesive-cohesive failure after fracture of specimen of group 4 as a result of crack progression within the Zirconia ceramic core. The initial tensile crack progressed with lateral crack deflections involving the veneering ceramic/Zirconia core interface with partial delamination of the feldspathic veneering ceramic and opaque ceramic material.

Figure 31. SEM micrograph of representative tensile crack progression with feldspathic veneering ceramic delamination and Zirconia core exposure with adhesive-cohesive failure of specimen of group 4.

Figure 32. Catastrophic failure of specimen of group 6 as a result of crack progression within the Zirconia ceramic core. The initial tensile crack progressed with lateral crack deflections involving the veneering ceramic/Zirconia core interface with partial delamination of the feldspathic veneering ceramic and opaque ceramic material.

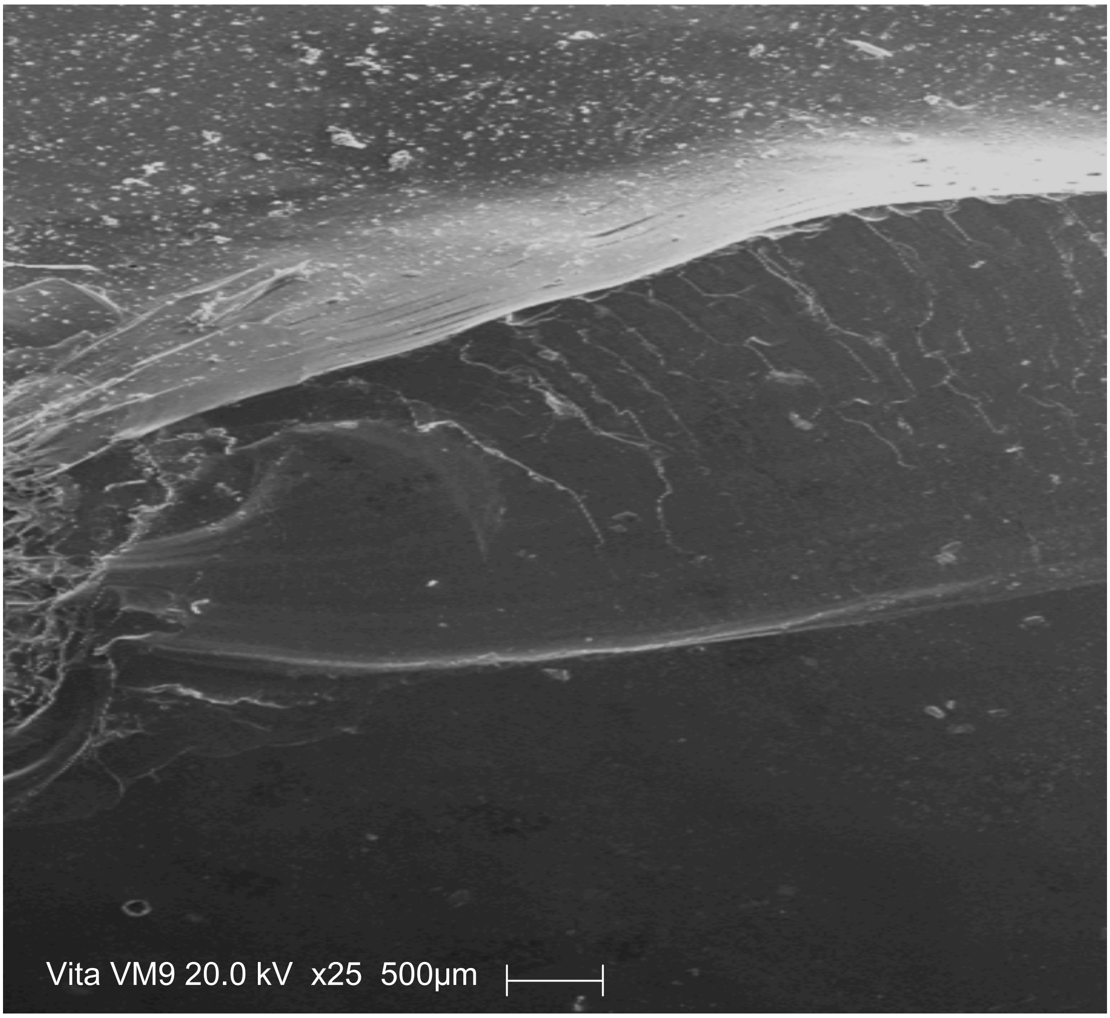

Figure 33. SEM micrograph of representative adhesive-cohesive failure after fracturing a specimen of group 6. In this specimen the type of fracture was mainly cohesive.

CHAPTER 5

DISCUSSION

The massive increase in demand for more esthetic restorative materials in dentistry has led to the use of dental ceramics as an alternative for both anterior and posterior restorations. Any material used for these purposes must possess certain physical requirements such as high strength, good marginal integrity, and good esthetic properties to be acceptable for clinical usage. Metal-ceramic crowns have been widely accepted as the treatment of choice for single or fixed partial denture restorations because of the advantages of high strength, reasonable esthetics, and long-term predictability (Hondrum, 1994; Denry *et al*., 2008).

However, the increased requirements for optimal esthetics have made metal-ceramic restorations no longer adequate to meet the expectations of both clinicians and patients. In this respect, the all-ceramic crown has set a standard for esthetics that is difficult to match by the metal-ceramic crown. This is due mainly because of the absence of underlying metal and increased light transmission through the restoration. While recognizing the improved esthetics clinicians and scientists have questioned the use of all-ceramic crowns because of their lack of strength. Their counterparts, the metal-ceramic crowns, have been used successfully with a failure rate of only 1 to 3% over 5 years (Anusavice, 1993; Fisher *et al*., 2008).

Metal-ceramic systems have come under scrutiny, however, because of (1) potential alloy corrosion leading to toxicity and allergy concerns; (2) esthetic problems such as lack of translucency, discoloration of some ceramics from silver in the alloy, and excessive value in the cervical third; (3) the amount of tooth reduction necessary, and associated tendency to overcontour the restoration; and (4) incompatibility between metal and ceramic, and the difficulty in establishing standard tests for bond strength and thermal compatibility (Denry *et al*., 2008).

Research has emphasized the development of stronger all-ceramic restorations, largely because of increasing esthetic demands (Fradeani, 2003). For this reason, since the early 1990s, researchers and clinicians have been seeking news ways of fabricating all-ceramic restorations that possess the needed qualities of strength, color stability, favorable wear characteristics, and precision of fit so that they may be placed in all regions of the oral cavity. Additionally, these techniques must produce crowns that consistently meet these qualities in a manner that is cost-effective for the patient, dentist, and laboratory. The development of present day all-ceramic systems has offered many improvements over the metal-ceramic crown such as increase translucency, adaptability and biocompatibility.

However, the biggest advantage of all-ceramic crown is its natural tooth-like appearance. High strength ceramic copings now available, mimic the light transmission properties of natural tooth by improving the translucency of light through the restoration and the underlying tooth structure. Dental ceramics allow regular and diffuse transmission, as well as diffuse and specular reflectance of light, and therefore have the potential to reproduce the depth of color, and texture of natural teeth (O'Brien 2002).

Several all-ceramic systems with various compositions are now available, demonstrating technologies that are reported as satisfying the demands of the dental profession (Luthardt *et al*., 1999). These ceramic systems are widely used as restorative materials in dentistry and present similar success rates of metal-ceramic crowns (Zitzmann *et al*., 2007; Fradeani *et al*., 2002; Odman *et al*., 2001). These new all-ceramic crowns with high strength still remain susceptible to the high mechanical loading during function. The absence of reinforcement by a metal substructure results in relatively weak flexural strength and low fracture resistance. Therefore, available all-ceramic restorations have limited clinical application in areas of high stress, such as posterior single crowns and fixed partial dentures. The major disadvantage of dental ceramics is their susceptibility to fracture during placement, mastication, and trauma. Different reports suggest that all-ceramic restorations demonstrate acceptable longevity compared with conventional restorations (e.g., metal-ceramic crowns) (Odman *et al*., 2001; Fradeani *et al*., 2002). For single-rooted anterior teeth, broad support was found for the premise that clinicians may select from any all-ceramic systems for laminate veneers, intracoronal restorations such as inlays and onlays, and for full-coverage restorations. Single crowns composed of different materials (lithium disilicate, leucite, aluminum oxide) have been successfully placed for 10 to 20 years. They have been shown to achieve good clinical survival rates and have thus become the standard of care for single crowns, especially in the anterior region. For restoration of molar teeth, the reviews suggest that relatively few all-ceramic systems will provide predictable long-term success (Land *et al*., 2010; Della Bona *et al*., 2008)

In order to overcome the inherent mechanical weakness of all-ceramic crown systems, several manufacturers have recently investigated and introduced a high strength ceramic material composed of Y_2O_3-partially-stabilized zirconium oxide for the substructures of all-ceramic crowns and fixed partial dentures. Its' superior mechanical behavior over alumina ceramic substructures has been reported in the dental literature (Piconi *et al*., 1999). The indication that Zirconia containing ceramics exhibit durability in a highly loaded environment makes them attractive for use in dentistry and increases the clinical indications to posterior molars and posterior fixed partial dentures. Zirconia is chemically an oxide and technologically a ceramic material, not soluble in water, that was proved not to be cytotoxic, and not to enhance bacterial adhesion, and exhibits a favorable radio-opacity and a low corrosion potential (Piconi *et al*., 1999)

While much research has been conducted to assess the strength of traditional dental porcelain materials very little information has been reported concerning the relative strength of many of the ceramic materials already in clinical applications. The precision of fit, strength and biocompatibility of Zirconia have been studied and found to be excellent for multiple dental applications (Piconi *et al*., 1999, Luthard *et al*., 1999). However, little data exists regarding the strength, fracture toughness, fracture mode and hardness of veneering dental ceramics designed for Zirconia structures (Denry *et al*., 2008; Fischer *et al*., 2008).

Some clinical studies with data up to 5 years reported a high prevalence of chipping of the ceramic veneering material for Zirconia-supported restorations (Sailer *et al*., 2006; Raigrodski *et al*., 2009; Christensen *et al*., 2009; Schley *et al*., 2010). Fracture of the framework, however has been rarely reported to date. The prevalence of veneer chipping seems to be higher when compared to that for metal-supported restorations (Heintze *et al*., 2010). The data available indicate also fewer mechanical problems for single crowns compared to Zirconia-supported fixed partial dentures (Ortorp *et al*., 2009). Whether the higher frequency of veneer chipping could be attributed to the material or to the technique sensitivity of the veneer porcelain processing, or whether there might be other unknown influencing factors remains unclear. Another important conclusion drawn form these reviews was that the frequency of chipping varied greatly across studies. Some studies did not report any veneer chipping, and in others, more than 20% or 30% of all Zirconia restorations showed veneer chipping. An explanation as to why the results of these studies were so different could be the clinical examination criteria. In some studies chipping was evaluated by direct observation and in others was performed using scanning electron microscopy. Therefore, small chippings that would otherwise not have seen during clinical examination were recorded. Another possible reason for the variation could be that studies were performed in different environments. Some were carried out in private practice by general dentists and others at universities. General practitioners might be less careful with operational procedures (e.g., seating of restorations, occlusal adjustment, polishing) than clinicians at universities. It may be speculated that suboptimal parameters during the fabrication of the Zirconia restorations or during the incorporation of the Zirconia restorations in the oral cavity could account for the variation in the researchers findings. In this case, Zirconia restorations have a higher technique sensitivity compared with metal-ceramic restorations, the later showing significantly less veneer chipping, even in fabrication and evaluation performed in the same environment and by the same technicians and operators (Heintze *et al*., 2010).

The question of interest for the practitioner is how Zirconia-based materials may be improved to reduce the risk of veneer chipping. When Zirconia materials were introduced, it was thought that they could be handled similar to metal-ceramic materials. Since clinical studies have shown a high frequency of chipping at the veneer material, dental manufacturers began to address this issue.

Several factors must be taken into account during the fabrication of a Zirconia based restoration. The coefficient of thermal expansion of the veneer and Zirconia material must be adjusted. Generally, the veneer material has a higher coefficient than the core, which puts the veneer under tensile stress and helps it to adapt well to the core. The difference in the coefficient, however, should not be too great. If there is a strong misfit, technical failures occur with high frequency (Fischer *et al*., 2009). The low thermoconductivity of Zirconia leads to unfavorable temperature distributions and the development of internal stresses in the veneer material during firing and cooling of the restoration (Fischer *et al*., 2009). Prolonged cooling until the glass ceramic has reached the critical glass transition point results in less stress distribution (Swain, 2009). If the thickness of the veneer exceeds that of the core twofold or more, the risk of veneer chipping is increased considerably (Hsheh *et al*., 2008). If the veneer is not supported by the core, which means that the cusps that are built up with veneer material do not have an anatomical counterpart on the core side, the risk of veneer chipping is increased (Rosentritt *et al*., 2009). When the first computer-aided design/computer-assisted manufacturing systems were brought to the market, the software of some systems did not allow the core to be designed anatomically. Subsequently, manufacturers have made it possible to anatomically alter the shape of the Zirconia substructure. There is wide spread agreement that veneer materials with higher strength should be developed. These materials should withstand the occlusal and articulation forces better than the current porcelains. Additionally, these materials should be able to be adjusted by the dental technician and/or the dentist and maintain not only the strength but also clinical long-term performance.

Although the last two points did not yield a significant correlation and analyses with the frequency of veneer chipping in these studies, in can be expected that these configuration parameters are important in reducing the risk of chipping. For these reasons and because there is no study available to characterize the strength alterations that can occur in the veneer ceramics, used in conjunction with Zirconia-based restorations, after surface adjustments or treatments the present investigation was carried out.

Strength and fracture toughness characterize the responses of materials, like brittle dental ceramics, to loading forces and crack propagation, respectively. Strength is a very important property of all-ceramic materials. However since many variables, such as testing design, specimen geometry, polishing procedures and testing environments affect strength measurements, it is not considered an intrinsic property. Conversely, fracture toughness is a more fundamental property then strength, since it characterizes bulk structure without involving the flaw size.

The standard for testing the strength of dental ceramics has been the three-point flexural test, but one problem has been the sensitivity of the test to flaws along the sample edges. It is impossible to eliminate all flaws and, because fracture often initiates at the edges, large variations strength data have been recorded (Ban *et al*., 1990). The strength measured in a uniaxial tension test is sensitive to flaws that exist throughout the material, because the tensile stress is uniform. In a flexure test, the stress is expected to vary from pure compression on one surface to pure tension on the other. The flexure strength therefore depends primarily on the size of the flaws at and near the surface, where the tensile stresses are the highest. Four-point and biaxial flexure tests develop lower levels of shear in the test section as compared to three-point flexure tests. The stress state in four-point and biaxial tests is therefore closer to pure bending. Furthermore, biaxial tests are less sensitive to edge effects than three- or four-point flexure tests and less sensitive to surface imperfections resulting from specimen preparation. In addition, the biaxial test probes for the largest flaws oriented over a wider range of angles, while the three- and four-point bend tests are most sensitive to flaws nearly perpendicular to the beam axis of the sample. For all these reasons the methods used for determining strength are important factors since the methods used can profoundly affect results and test interpretations.

The difference in results of the three different test designs may be explained as follows. Flexural strength obtained with the four-point flexure test is generally lower because the probability to have a surface crack between the two loading pistons is higher than in the more limited area beneath the loading piston of a three-point flexure test. In the biaxial flexure test, the force is applied in the center of the specimen. Defects at the edges, which most probably lead to an early failure, are less effective. Nevertheless, the probability of a crack in the vicinity of the loading piston is higher than in the three-point flexure test because the loaded area is larger (Anusavice *et al*., 2007). It can be concluded that for screening tests, for instances, during the development of ceramics, the biaxial flexure test is most appropriate because preparation of the samples is easy, compared to the three- and four-point flexure test. However, according to some studies results (Ban *et al*., 1990; Fischer *et al*., 2007), when scientific approach is intended, the four-point flexure test should be preferred.

Although the four-point flexure test might give a more systematic and logical approach, the present investigation used the biaxial flexural test as the method of choice since this test is less sensitive to surface imperfections resulting from specimen preparation and less sensitive to flaws perpendicular to the beam axis and on the edge of the specimens. This is especially important in this study since the specimens tested in the first part of this investigation were subjected to surface preparation. Moreover, the International Standard for Dental Ceramic defines the biaxial flexural strength test method as suitable for assessing the effect of different surface preparations (for example air-borne-particle abrasion, as-fired, ground, and overglazed) on the strength of ceramic materials.

The mean biaxial flexural strengths of the control groups for the three ceramics tested in the first part of this investigation are in good agreement with previous results found in the literature using the same ceramic materials (Fischer *et al*., 2008). Although the results are slightly higher than those reported, this may be attributed to the fact that in this study the tip piston diameter of 0.7mm used, when compared to the 1.5mm piston tip diameter used in the Fischer's study, gave rise to mean biaxial strength, as a smaller area of the disc specimen was subjected to the maximum tensile stresses; subsequently there was less chance of the specimen having a critical flaw in that area, which led to the improved, mean biaxial strength. This clearly shows that mean biaxial flexural strength is affected by testing design. Care is therefore required, as mean strength comparisons under different conditions or testing parameters may become invalid (Guazzato *et al*., 2002; Albakry *et al*., 2004).

The present study also showed that grinding, polishing and heat treatment have remarkable effects on the strength of the feldspathic veneering ceramics tested. This confirm the first two hypotheses formulated originally in this investigation that there are significant differences in the load fracture resistance, measured in terms of biaxial flexural strength, among different ceramic surface treatments in the feldspathic veneering ceramics tested in conjunction or individually. The effects of surface treatments analysis showed that grinding reduced the strength of the tested specimens, and this was statistically significant when all the ceramics were considered together.

The tentative conclusion is that the stress concentration due to roughness of the surface caused by the grinding procedure is responsible for the differences between the biaxial flexural strength of the different specimen groups. In the literature (Bazant, 1986; Jager *et al*., 2000), the failure of many materials, including ceramics, has been attributed to the propagation of a large system of densely distributed cracks, rather than to a single precisely defined fracture. The number of cracks and microcracks is extremely large and, according to the literature, their location and orientation are random. Irwin (Irwin, 1957) demonstrated that stress intensity is related to a crack shape in a particular location with respect to the loading geometry. The finishing procedures influence the existence of microcracks and residual stress. For example, polishing and glazing could round the crack tip of possible microcracks. The change in crack length and crack tip radius would change the strength of the material. The finishing procedures, however, also produce a certain surface roughness. Surface roughness will lead to a non-uniform stress distribution and concentrate locally an applied stress due to the shape differences in the surface layer. The distributed cracks may not develop or propagate randomly, but occur or propagate at points with the higher stress as a result of the surface roughness. This hypothesis is also supported by the work of Mecholsky and co-workers, who loaded samples with grinding grooves and gouges both perpendicular and parallel to the loading direction (Mecholsky *et al*., 1977).

In the case of the specimens with the tensile axis perpendicular to the grinding direction, like in this investigation, this resulted in a lower fracture strength and flaws resulting in failure generally being situated parallel to the grinding direction. Grinding grooves or gouges parallel to the tensile axis will not cause stress concentration, while those perpendicular to the tensile axis will do so. This stress concentration will result in lower fracture strength and in failure-causing flaws being situated on the points with the highest stress parallel to the grinding direction. The height difference of a rough spot over a distance of 50 µm will dominate the stress concentration (Jager *et al*., 2000). The roughness proved to be an indication for the height difference of a characteristic rough spot.

In the dental ceramics evaluated in this study the relation between the biaxial flexural strength and the roughness of the specimens supports the hypothesis that surface roughness will concentrate an applied stress, and resulting in a lower biaxial flexural strength. Stress concentration caused by either roughness, surface defects, or internal stresses are believed to be the main reasons for the mean biaxial strength variations among all tested groups of the feldspathic veneering ceramics. Ground groups in the three feldspathic veneering ceramics tested recorded the lower strength values when compared to the control, polished and glazed groups. During grinding, heat, cracking, chipping and residual stresses may be generated, from which strength reducing flaws are initiated. Like previously mentioned, Mecholsky and co-workers reported that two sets of flaws grow in directions both perpendicular and parallel to the grinding direction, but the tensile orientation during testing determines which set initiates fracture (Mecholsky *et al*., 1977). In the case of biaxial test, the direction of grinding will not be as critical, as stresses develop uniformly within the maximum tensile area, at the centre of a disc (Albakry *et al*., 2004), and the cracks developed in this area are more likely to dictate strength. In addition, during these treatments, if a flaw size, sharpness, orientation or distribution changed, the final strength of a ceramic material will be changed (Kelly, 1995). This possibly explains why the reduction in strength in this study is not as much pronounced as it is in other studies, where the strength degradation caused by grinding can reach 80% in relation to untreated surfaces (Giordano *et al*., 1995).

When each feldspathic veneering ceramic was examined separately, only in the Vita® VM®9 veneering ceramic the grinding group was statistical significant different from all the others surface treatment groups. The statistical insignificant difference in strength values between the grinding group and some other groups in the NobelRondo™ and Lava™ Ceram was somewhat surprising. This is because lower strength values are expected after more severe procedures such as grinding. This also indicates that strength reduction of a ceramic material is a consequence of the finishing tools, coarseness and feed rate, which determines the associated defects, size and the amount of compressive stresses developed. It may also depend upon the characteristics of different ceramics microstructure. The size of this strength-reducing flaws originated by grinding is also dictated by the fracture toughness of the treated material. Although grinding involve mechanical removable and a damage of varying degrees, they may be accompanied, as previously explained, by development of residual stresses, which can be compressive, depending upon the parameters of the treatment procedure. The compressive layer, formed by surface plastic deformation, may act to prevent the extension of microscopic defects. Consequently, provided no significant median or lateral cracks formed, strength might by improved by grinding or machining (Albraky *et al*., 2004).

Grinding is commonly involved during machining of an all-ceramic framework and adjustments by ceramist and dentist to improve occlusion and proper fitting. The effect of grinding on the surface of ceramic and its mechanical properties can be contradictory. The influence of grinding on the ultimate strength of a ceramic can be explained by tacking into account factors which may alter the combined effect of the surface flaws and the residual stress layers. Some of these factors are: the magnitude of the residual stress (which is also related to the composition and microstructure of the ceramic); the ratio of the crack length to surface compressive layer depth (Lange *et al*., 1983); the effective size of grinding particles (Johnson-Walls *et al*., 1986); the dimensions of the pre-existing flaws (Tuan *et al*., 1998); the orientation of grinding (Mecholsky *et al*., 1977); and ceramics microstructure (Albakry *et al*., 2004). When comparing to the control groups the reduction of strength in the grinding groups was almost 30% for the NobelRondo™ veneering ceramic 17% for the Lava™ Ceram veneering ceramic and 15% for the Vita® VM®9 veneering ceramic. This clearly demonstrates that careful must be taken when adjusting ceramic Zirconia based restorations since a considerable reduction in strength can compromise the long-term success and contribute for the failure of the rehabilitation.

It has been well established that crack propagation brought about by tensile stress, can cause a brittle ceramic to fracture (Anusavice *et al*., 1991). However, if the strength of the material, affected by internal microstructure and surface quality is improved, then the performance of all-ceramic dental restorations should improve with respect to fracture resistance (Anusavice, 1996). The glazing surface treatment is a routine procedure in a dental laboratory. It produces crowns with smooth and shiny surfaces and has a positive effect on the biaxial flexural strength of ceramic materials (McLean *et al*., 1979). The term “overglazing” defines the firing of a low-fusing colorless glass on the ceramic core or veneering ceramic. This thin layer of about 4 µm of glass, produced after 60 seconds of hold time at the final temperature reduces the size of flaws present on the surface (probably introduced during the fabrication of the restoration), thus increasing the strength of the materials (Brackett *et al*., 1989; Isgro *et al*., 2003). Furthermore, it is used to provide large surface compression, which strengthens the ceramic body.

In addition to the application of a low fusing glass overcoat, glazing can be also be performed on firing for a certain time, held at the maximum temperature and is termed auto-glazing or self-glazing. As far as chemical durability is concerned, self-glazing ceramics are preferred over glazing masses. A higher concentration of glass modifiers reduces the resistance of the applied surface glaze compared to the normal surface glaze of the ceramic. During glazing a thin outer layer is formed. A certain temperature and treatment time leads to the formation of a softer glass phase and the formation of crystalline particles within the surface region.

Auto-glazing and/or the application of glazing material after grinding is believed to increase the strength of ceramic materials by reducing the depth and/or sharpness of critical flaws as previously stated (Baharav *et al*., 1999). However, this effect is still uncertain (Fairhurst *et al*., 1992). Several reports in the literature also showed that auto-glazing had no effect on strength (Anusavice, 1989; Fairhurst *et al*., 1992; Griggs *et al*., 1996; Denry *et al*., 1999; Albakry *et al*., 2003). It should also be considered that heat treatment after polishing or grinding may degrade strength, which is though to be a result of releasing compressive stresses that normally develop during polishing or grinding. The present investigation found that submitting the feldspathic veneering ceramic materials to heat treatment that is typically encountered during dental laboratory processing, revealed contradictory results in terms of improving the strength of ceramic materials and did not clarified the contradictory results present in the literature.

When analyzing the results of all three feldspathic veneering ceramics together glazed specimens demonstrated a statistically significant increase in strength when compared to grinded specimens. This increase in strength was almost 20% for the glazed groups (GG, GPG) that were previously submitted to grinding procedure. Characteristics rough spots were found in the glazed specimens and such spots did not appear in the untreated control specimens. Subsurface pores in the untreated samples and/or produced by grinding probably caused these defects. The considerable stress concentration caused by these rough spots, especially if they were presented near the loading piston, may help to explain the lower increase in biaxial strength verified in glazed specimens compared to the grinding specimens. The conclusion that the stress concentration caused by higher or lowers surface roughness after glazing is the dominant factor may be supported by the findings of Griggs and co-workers (Griggs *et al*., 1996). In their study flaws were created by means of diamond cup wheel. The researchers found statistically significant improvement in biaxial flexure strength after glazing, as Giordano and co-workers did, and in contrast with different other studies (Chen *et al*., 1999; Jager *et al*., 2000). In these studies, roughness produced on the surface was created my means of a Vickers indenter which creates a much more severe surface roughness that may by present even after glazing.

The glazing of grinded NobelRondo™ specimens did not resulted in a statistical significant increase in strength, even though the percentage of increase was almost 10%. However, in the specimens subjected to grinding that were polished before glazing significant difference was encountered. This may be explained by the fact that the polishing may have reduced the flaws present at the surface of the specimens produced by grinding and increased the compressive surface layer of the ceramic. If we could extrapolate clinically these results, they indicate that polishing should always be performed before glazing if this ceramic needs to be adjusted. On the other hand, specimens that were subjected to just polishing and glazing were not statistically significant difference from those that were just grinded. The fact that the strength of NobelRondo™ ceramic was not altered by these different surface treatments may be due to the size of the microstructure particles and the differences in thermal expansion between the different particles and the surrounding glass matrix. During cooling, the particles contract more than the surrounding glass, and above critical particle size the stress created during cooling can induce microcracks circumferential to the particles. Also as mentioned above grinding may introduce some surface compressive strength especially if it is followed by polishing and glazing that reduces flaws and pores in the surface.

On the Lava™ Ceram veneering ceramic specimens, the effects of glazing, in contrast with the results of the NobelRondo™ ceramic specimens, were more evident. The two groups with higher biaxial flexural strength were the GG and GPG groups. The percentage of increase in the glazing groups when compared to the grinding group was almost 20%. This increase was in this particular ceramic group statistically significant. However the capability of the heat treatment to significantly enhancing the strength is dependent of the preceding surface treatment. This is especially apparent since there was no statistically significant difference between the grinding group and the polishing and glazing group. It seems that in this ceramic glazing or polishing after grinding has a beneficial effect. This may be explained, as previously, by the recognized capability of grinding to generate a shallow surface layer of compressive stresses which tends to close flaws and thus strengthens the material (Lange *et al*., 1983). At the conditions applied in the present study, less severe damage is expected by grinding compared to other studies (Chen *et al*., 1999; Jager *et al*., 2000) were Vickers indenter were used. Furthermore, the diamond grains of the grinding wheel used in this study are unevenly distributed and relatively blunt and extensive heat (up to 700°C) can be generated during grinding, despite the water coolant. In this case, such temperature may favor healing of the crack and hence reduce the depth of the damage zone. It become apparent also, that the size of the microstructure particles and the differences in thermal expansion between the different particles and the surrounding glass matrix may play an important role in the alterations of the strength of this material, since just polishing and glazing were not capable of enhancing the material strength.

On the Vita® VM®9 veneering porcelain, glazing significantly increase the strength of the ceramic specimens. All the groups that where glazed revealed higher strength values when compared with the grinding group. This increase was almost 20% for the PG group. Probably in this ceramic the damage caused by grinding was not compensated by the creation of a compressive layer or the microstructure of the ceramic is severely affected by the procedure and a large number of surface defects and flaws remained at the surface after grinding. Stress concentration can be initiated not only from surface roughness, but also, from other factors, such as internal stresses (within the microstructure), porosity, inherently developed cracks and thin sectional areas close to tensile stress. Thus, surface roughness can dictate strength if no larger stress concentration greater than that of surface roughness occurs (Jager *et al*., 2000). Kitazaki and co-workers reported that surface roughness is not the only factor that determines strength (Kitazaki et al., 2001). Furthermore, some ceramic materials exhibit different crystalline concentrations between their surfaces and the internal portions, which may occur as a result of different surface treatment and may act to strengthen materials significantly. This is further substantiated by the fact that in this ceramic the PG group was the one that demonstrated the higher biaxial flexure strength.

Polishing is a procedure often used to minimize the flaw size and therefore improve the strength of a given ceramic (Giordano *et al*., 1995; Giordano *et al*., 1995; Williamson *et al*., 1996). Polishing with SiC disks and diamond paste material produces the smoothest surface and the highest strength compared to all other surface treatments (Albraky *et al*. 2004).

In this study when considering the effect of all three veneering ceramics tested together polished specimens demonstrated a statistically significant increase in strength when compared to grinded specimens. This increase was almost 20% for the GP group when compared with the G group. The ability of polishing to eliminate various defects and flaws from the treated surface is considered responsible for such strength increment. Moreover, the compression layer formed during polishing is also relevant (Giordano *et al*., 1995). The strengthening due to compressive surface stresses is a result of residual stresses, which oppose the applied tensile stresses. However, if a flaw size exceeded the depth of this layer, compressive stresses may not contribute to the strengthening effect of polishing (Kosmac *et al*., 1999). It has been speculated that more than 50% strength increase can be achieved after fine polishing (Albakry *et al*., 2003).

The polishing of grinded NobelRondo™ and Vita® VM®9 specimens resulted in a statistical significant increase in strength, with a percentage of increase of about 25% and 10% respectively. For the Lava™ Ceram the polishing of grinded specimens did not resulted in a statistical significant increase in strength, even though the percentage of increase was almost 12%.

For testing mechanical strength on brittle materials like ceramics, considerable variation among data is often reported (Seghi *et al*., 1990). The traditional method for presenting the results of such tests is to report the number of specimens, the mean strength, and the standard deviation. Except for standard deviation, little or no regard is given to the distribution of the individual data. Alternatively, the “strength distribution” of a material can be considered as the outcome of individual specimens deviating from the population mean. The Weibull modulus in the equation characterizing the scatter in strength is an excellent indicator of the variability of strength for brittle materials. A high Weibull modulus value indicates a close grouping of fracture stress values, while a low value suggests a wide distribution with a long tail at low stress levels (Chandrasekhar, 1997).

The nature of flaws in most ceramics is statistical in nature. As such, the strength of ceramics is not one specific value, but a distribution of strengths. The Weibull modulus is a measure of the distribution of flaws, usually for a brittle material. The modulus is a dimensionless number corresponding to the variability in measured strength and reflects the distribution of flaws in the material.

For brittle materials, the maximum strength (stress that a sample can withstand) varies unpredictably from specimen to specimen -- even under identical testing conditions. The strength of a brittle material is thus more completely described with a statistical measure of this variability, the Weibull modulus. For example, consider strength measurements made on many small samples of a brittle material such as ceramic. If the measurements show little variation from sample to sample, the Weibull modulus will be high and the average strength of the material would be a good representation of the potential sample-to-sample performance of the material. The material is consistent and flaws, due to the material itself and/or the manufacturing process, are distributed uniformly and finely throughout the material. A low Weibull modulus reflects a high variation in measured strengths and an increase in the likelihood that flaws will tend to congregate and produce a weaker material. A material with a low Weibull modulus will more likely produce products where the strength is substantially below the average and show greater inconsistency of strength. Such products will exhibit greater variation in strength performance and will probably be less reliable.

The Weibull modulus results for the three ceramic types tested are in good agreement with values obtained in other studies found in the literature, and correspond to the Weibull modulus range reported for most ceramics, which is between 5 and 15 (Albakry *et al*., 2004; Fischer *et al*., 2008). Weibull modulus values of the control (untreated), polished, and glazed groups for the three veneering ceramics tested showed higher values than the ground groups. This indicates less variation in the strength and similar distribution of flaws among samples. The damage and mechanical removal of particles during grinding normally introduce wide distribution and different shapes of defects and flaws. Consequently, a wider range of strength values is expected. During testing, some specimens recorded very low load at fracture, and consequently demonstrated lower strength value compared to other specimens. These specimens are expected to be either defected or suffering from detrimental pores that were located within the maximum tension area. Weibull modulus results tend to significantly escalate if these specimens are discarded. More reliable Weibull modulus can be achieved when large number of specimens is tested. Typically 30 or more are recommended (Cattel *et al*., 1997)

Based on the results of the first part of this study, the hypothesis that the strength is affected by grinding and improved by polishing and glazing, and the roughness determines the strength is partly accepted. Stress concentration can be initiated not only from surface roughness, but also from other factors, such as internal stress (within the microstructure), porosity, inherently developed cracks and thin sectional areas close to tensile stress. Thus, surface roughness can dictate strength if no larger stress concentration greater than that of surface roughness occurs. The results of this study suggest that surface roughness determines the strength of a ceramic material, except where the material has an inner structure which causes an even larger stress concentration than that caused by the combination of surface roughness and flaws. Flaws can be introduced into a ceramic during powder compaction, forming drying, firing, and later shaping, or they can be inherent in the microstructure. Processing flaws in dental ceramics might include grinding damage, pullout during polishing, subsurface microporosity, or large pores introduced by the dental technician or dentist during adjustments. Inherent flaws might include cracking around large grains with unmatched thermal expansion properties and pores developed during firing.

No previous studies that appraised the effect of surface treatments on the strength of veneering ceramics for Zirconia restorations were found in the literature. The first part of present study showed that grinding did decrease the strength of veneering ceramics and in some cases significantly. For this reason, grinding should always be avoided if any other procedure is to be done, as this will either create or change the developed crack dimensions or increase the volume loss. In addition grinding, when required, should be used at low speed and in a wet environment to reduce the potential of microcrack formation. Although some materials did not show a significant decrease in strength after grinding, probably do to their microstructural nature and the creation of a compressive layer, grinding is not advisable in areas where layering ceramic may be added or be unsupported. By grinding in these areas the risk is that as the bond created between the core and the layering ceramic may deteriorate and cause fracture at lower forces. Polishing and glazing have improved the strength of all materials and in some cases significantly, which is thought to be related to the ability of the procedures to improve the condition of the ceramic’s surface and free it from various defects and flaws. Polishing and glazing are recommended to counteract the detrimental effects of grinding. It is therefore suggested that polishing, whenever possible, should follow clinical adjustment or surface modifications in order to minimize the effect of possible harmful defects and flaws. Further research is needed to find a material for the outer layer of the restoration that in combination with surface treatment produces a surface that remains smooth. Given proper surface treatment, such a material would not require crack-stopping properties, and in vivo, the surface of the material would remain smooth, hopefully resulting in long-lasting restorations.

The results of this study revealed that the biaxial flexural strength values of veneering ceramics for Zirconia are in the same range as those of veneering ceramics for metal-ceramic systems (Fischer *et al*., 2008). The fact that the strength of veneering ceramics for Zirconia is in the same order as that of veneering materials for metal-ceramics may be interpreted in the sense that the strength of the veneering ceramics are not the limiting factor for the clinical long-term success of Zirconia restorations. Nevertheless, compared to metal-ceramics excessive chipping has been observed in clinical studies with Zirconia restorations (Vult von Steyern *et al*., 2005; Sailer *et al*., 2006; Sailer *et al*., 2007). Although Zirconia based systems offer the advantage of favorable material characteristics for substructures, the clinical problem of chipping of the weaker esthetic veneer persists (Zarone *et al*., 2011).

To explain this effect, two aspects must be considered. The first is the stress, built during cooling after firing of the veneering ceramic. In metal-ceramic systems, this stress may be at least partially relaxed by an elastic or plastic deformation of the substructure (Anusavice *et al*., 1987). Especially, high gold alloys show a low sag-resistance (Fischer *et al*., 1999). A Zirconia substructure in contrast is rigid, which leads to higher stress formation. Hence, compared to metal-ceramics a higher flexural strength of the veneering ceramic is favorable to provide a high reliability of the veneer. The present investigation has shown that, depending on the test method and the brand, the flexural strength of veneering ceramics for Zirconia is rather similar than that of veneering ceramics for the metal-ceramic technique. Therefore, the effort to improve the veneering ceramics for Zirconia should be directed to the optimal adjustment of the thermal expansion and the increases of mechanical strength, which is in accordance with the appraisal of other authors (Fischer *et al*., 2008). A second point is that in the oral cavity water exposure may cause hydrolysis of the Si-O-Si bonds, thus affecting the mechanical properties of the ceramic. Flexural strength values are obtained at ambient laboratory conditions. The increased failure rate of veneering ceramics for Zirconia under humid conditions in the oral cavity may be attributed to a different chemical composition compared to ceramics for the metal-ceramic technique, resulting in a higher susceptibility for hydrolytic attack. Further investigations are needed to test this hypothesis.

Another focus of attention must be given to the production criteria and design methods used in the fabrication of all-ceramic crowns. Layered all-ceramic crowns have become widely used since the introduction of Alumina and Zirconia cores and the availability of CAD/CAM milling techniques. However, some questions and problems, besides those mentioned above and related to brittle material fracture and to the esthetics of core materials remain (McLean, 2001; McLaren *et al*., 2000). Design practices have been based more upon empirical guidelines than upon clinically relevant scientific data. (Kelly, 1999; McLaren *et al*., 1999). Remarkably little scientific data on optimal design of all-ceramic crowns has been published. Ceramic copings are often generically milled to arbitrary thickness of 0.4 or 0.7mm. This may not provide uniform and appropriate thickness for veneering porcelain. Future studies of failure mechanisms and clinical outcomes may guide clinical practice (Kelly, 1999; Anusavice *et al*., 2000; Denry *et al*., 2008).

Fracture appears to be the most common clinical failure mechanism of all-ceramic crowns. (Denry *et al*., 2008). Overall crown thickness may be of primary importance in resisting fracture; a minimum overall thickness of

1.5mm has been recommended. (Lawn *et al*., 2004). However, relative layer thickness is also important (White *et al*. 1994; Lawn *et al*., 2002; White *et al*., 2005). Relative layer thickness influences strength, stress distribution, and failure mode in Zirconia restorations (Fleming *et al*., 2005). It has been suggested that a 1 to 1 ratio of core to veneering porcelain thickness may provide reasonable strength, esthetics, and fabrication tolerance (Lawn *et al*., 2004). Most authors agree that the importance of adequate core thickness may be paramount to clinical success (Wakabayashi *et al*., 2000; Fleming *et al*., 2005). The stiffness, or elastic modulus, of the core material is also influential. A stiffer core may better resist flexure under load. This is important because ceramics have low critical strains and are, in general, poorly supported by flexible dentin (Wakabayashi *et al*., 2000; Lawn *et al*., 2002). However, stiffer core materials may also be more vulnerable to radial cracks originating from their internal surfaces (Wakabayashi *et al*., 2000). Pertinently, Zirconia is stronger, tougher, and more flexible than alumina (White *et al*., 1994). Thus, Zirconia-based crowns might be expected to differ from alumina-based crowns in clinical failure mode and in overall clinical performance, but such comparative data is absent in the dental literature.

The advantages of customizing coping design are that: core and porcelain thickness can be controlled; marginal areas can be optimized for strength with a high shoulder or for esthetics with a porcelain labial margin; and butt joints between the porcelain and the core can be facilitated. Until more is known about clinical failure modes and clinical performance parameters, precise recommendations cannot be made with confidence. The disadvantage of using a customizing technique is primarily the dental laboratory technician time involved in full-contour waxing and cut back, as well as in completing a second scan in some all-ceramic systems. This may be offset by greater ease in porcelain application and in a potential, but yet unknown, improvement clinical service. Over the past years, only one clinical study has reported the utilization of this technique (Marchack *et al*., 2008). Although the authors have not yet encountered any instances of cohesive porcelain fracture or core fracture, conclusions cannot be drawn from such a small sample size in so short time. Clearly, more research is needed to relate material properties, crown design geometry, and tooth preparation parameters to the clinical failure mechanisms and clinical performance of all-ceramic crowns.

The present investigation, as well as previous studies conducted on clinically failed all-ceramic crowns and FPDs (Kelly *et al*., 1995; Marchack *et al*., 2008) and in-vitro-tested bilayered samples (Zeng *et al*., 1998; Lawn *et al*., 2001; Chong *et al*., 2002; Guazzato *et al*., 2004), indicated that the strength, reliability, and mode of fracture of bilayered ceramic composite are mainly dictated by the material on the surface undergoing biaxial tensile stress (bottom surface). The biaxial flexural strength of the groups with the core material at the bottom surface was much greater than that of the groups with the veneering ceramic on the bottom when the ceramics were analyzed together or individually. Only with the NobelRondo™ veneering ceramic groups did this situation not occur; independently of the material that was placed in the bottom surface, and therefore undergoing biaxial tension, the biaxial flexural strength of the bilayered specimens was not statistically significant. This can probably be related with the development of residual stresses due to mismatch of the coefficient of thermal expansion, fabrication procedures or surface damage. Since the fabrication procedures were the same for all three types of ceramic and no surface treatment was made in these specimens, the explanation for this outcome may be associated with the fact that the Zirconia discs used in this study were obtained from the same manufacturer of the NobelRondo™ veneering ceramic. It is normally anticipated that the coefficient of thermal expansion of the veneering ceramic will be slightly lower than that of the core materials, so as to induce a slight residual compressive stress. This should have resulted in higher applied stresses for the Lava™ Ceram and Vita® VM®9 ceramic discs since the Zirconia used by these manufacturers is not the same. Furthermore, the firing schedules for the three ceramics are different and this phenomen may have contributed for the outcome result. Another concern may be the bond between the veneering ceramics and the Zirconia core. It has been well established that bonding between veneering ceramics and Zirconia is based on chemical bonds (Fischer *et al*., 2008). If the Zirconia used in this study has better bond to the NobelRondo™ ceramic than to the other two veneering ceramics is impossible to determine without further studies. However, this clearly demonstrates that Zirconia production and correct choice of the respective veneering ceramic may have an important role in the long-term sucess of the final all-ceramic restoration.

The concept regarding the compatibility between core and overlay porcelain constitutes a very important dilemma. It is stated that matched physical properties between core and overlay porcelain reduces permanent stress in finished restorations, minimizing crack propagation and allowing flexibility in thickness of the overlay porcelain and design of the restoration (Anusavice *et al*., 1994). Although sufficient toughness and hardness of the core material is important, and should never be neglected, it is equally, if not more, important that the working property of the core being compatible with that of overlay porcelain to achieve harmony of the ceramic restoration. In addition, excessive hardness and toughness may be potentially problematic. In case that the porcelain overlay of a fixed prosthesis fracture, it will become extremely difficult to remove the core that is fabricated from materials with excessive hardness, while by using core materials with a relative high compressive and fracture resistance, it will provide sufficient hardness and support, while at the same time allow removal within reasonable amount of time in cases of porcelain fracture.

Previous studies conducted by Zeng and co-workers and Isgro and co-workers observed that the veneering porcelain had a negative impact on the strength of some ceramic core materials (Zeng *et al*., 1998; Isgro *et al*., 2003). However, Zeng did not indicate which side of the double-layered specimens was under tensile stress, whereas Isgro did not test the 2-layer specimen with the veneering porcelain under tension. In this study, the decline in strength of the 2-layer specimens was recorded for all specimens when comparing these results with the flexural strength reported for Zirconia which is around 900-1200 MPa. One criticism has to be made on the analysis of the biaxial strength values for this part of the study. The biaxial flexural strength was achieved by adjusting the mean between the Zirconia and the veneering ceramics Poisson's ratio presented in the literature (White *et al*., 2005) and not by the quantification of the modulus of rupture or elastic modulus for this specific study. Though, the impact of such process can easily be neglected given the difference of the values encountered.

The present study suggests that the material that undergoes tensile stress dictates the ultimate strength of the all-ceramic restoration. The contribution of stronger and tougher core materials to the performance of all-ceramic Zirconia-based restorations may be offset by the weaker vennering ceramic if the design of the restorations does not take into account the actual distribution of the tensile stresses. As far as crowns are concerned, the improvement of the mechnical properties of the core material should provide better clinical performance of the restoration, provided measured are taken to avoid the creation of spurious flaws during laboratory processing. Because the discs with the strong core material on their tensile surfaces recorded larger strength values than when the weak veneering ceramic was placed on the tensile surfaces, it is strongly recommended that the undersurfaces of fixed partial denture connectors and other areas of high tensile stress not be veneered with porcelain at all.

Another important evidance from these results is the fact that, in contrast to traditional all-ceramic crowns that tended to fracture from the internal surface, in Zirconia-based restorations the veneering ceramic can be considered the weakest factor in terms of material resistance. This is in accordance with the results present in the literature that revealed that clinical failed Zirconia restorations caused by Zirconia core fractures are rare (Zarone *et al*., 2011). Therefore, it is recommended that prostheses be designed with as thick a core and as thin a ceramic veneer as posible. Although crowns and fixed partial dentures may be primarilly loaded in a vertical occlusal-to-gingival direction, their complex shapes and human masticatory habits may cause prostheses to be loaded in many different ways (White *et al*., 2005). Therefore, the maximum amount of core material in all potential areas of high stress is recommended.

The reliability of strength has been discussed in terms of Weibull modulus, where a higher Weibull modulus corresponds to a more homogeneous flaw distribution, less scatter of values and therefore greater reliability. The opposite result is expected with a lower Weibull modulus as previously exposed. The Weibull modulus is a statistical analysis which is influenced by the flaw distribution as well as by the number of the specimens used for the analysis. The flaw distribution is in turn affected by processing of the material and fabrication of the specimens. During this phase of the study and the preparation of the specimens, an effort was made to ensure that the surface treatment of each specimen was uniform and all specimens were equally treated following the specific recommendations of each manufacturer.

As seen for the strength values, the Weibull modulus was mainly related to the material on the bottom surface. When Zirconia was on the bottom surface undergoing biaxial tensile stress the reliability of the material was greater demonstrating a surface that is more homogeneous with similar flaws distribution among specimens and less variation in the strength. The production of the Zirconia discs industrially using a CAD/CAM technique normally introduces narrow distribution and different shapes of defects and flaws. Consequently, a smaller range of strength is expected.

The different variability in strength between all the ceramics tested when the veneering ceramic was placed under tension may be the reason to suspect that specimen’s preparation or type of Zirconia used in this part of the study was a factor affecting the strength values of the NobelRondo ceramic and the other two veneering ceramics. As far as the reliability of strength is concerned, it should be considered that the Weibull modulus is a statistical analysis which does not take into account some other factors which influence the long-term clinical performance of all-ceramic Zirconia-based restorations. For instance, a greater ratio of Zirconia/veneer ceramic or presence of unsupported veneering ceramic areas may be capable of altering the resistance of the restoration even when the preparation of the restorations is the same.

The fracture mode of bilayered specimens was substantially different according to which material underwent biaxial tensile stresses. When the core material was on top and the veneering ceramic on the bottom, the fracture tended to originate from a major defect at the bottom surface of the ceramic and propagate toward the interface. The failure mode of the veneering ceramic consisted of a star-like crack configuration radiating across the bottom tensile surface. Inevitable failure from such cracks, opposite each other, extended through the entire specimen. Those cracks generally met the core near to the interface and were not deflected at the interface. Some of the other star cracks initiated at the bottom surface did not approach the core near to the interface and were further deflected to run along the veneering ceramic/core interface. This behavior might be expected because of the elastic modulus and fracture toughness mismatch between the core and veneering ceramic. The-higher modulus core creates a greater mode II, or shear crack, loading as the crack approaches the interface, resulting in a more inclined crack. The interlaminar crack deflection could also indicate a relatively poor Zirconia-to-veneering ceramic bond. The clinical implication of this finding is that this system could have a tendency to produce porcelain “pop-off” rather than catastrophic failure. Of course, any type of damage is unwelcome, but “pop-off” might be considered a lesser evil.

Microscopy showed that when delamination occurred, the crack normally propagated through the lowest-toughness phase, the veneering ceramic. In only a small number of specimens did the fracture originate at the interface, where there was (within the stronger core material) the greatest peak of tensile stress. In the majority of the specimens the continuing load applied by the Instron machine resulted in catastrophic failure with fracture of the Zirconia ceramic in different areas as a result of crack propagation through the Zirconia core. Other investigators have shown that the fracture origin and fracture mode are greatly influenced by the test methodology and ct/vt ratio (White *et al*., 1994; Zeng *et al*., 1998; Thompson, 2000; Wakabayashi *et al*., 2000; Guazzato *et al*., 2004). In this study the biaxial flexure test was chosen because it is unaffected by edge failure and better resembles clinical conditions compared to the uniaxial flexure test. In fact, the biaxial flexure test more realistically replicates the in vivo situation, as it generates the greatest number of interfacial failures. Furthermore, the disc-shaped specimens have an area similar to dental restorations (Thompson, 2000; White *et al*., 2005). The site of crack initiation shifts from the veneering porcelain to the inner core as the core/porcelain thickness ratio increases (Wakabayashi *et al*., 2000). A ct/vt ratio of 1:1 was chosen in the present study as an acceptable tradeoff between the situation of a crown and a fixed partial denture.

When the core material was on the bottom and subjected to biaxial tension, a Hertzian crack was seen in the veneering ceramic of the majority of the test specimens. Cone cracking was significantly extended and reached the Zirconia core in all these samples, with partial delamination of the veneering ceramic in the region of the cone crack but without causing fracture of the core material. This was consistent in all the veneering ceramics tested demonstrated like failure modes. Therefore, it is not apparent that either residual stress remained from the ceramic firing, or finishing and polishing procedures or that the interfacial bond was relatively poor, or that both effects were present. Since this fracture mode was independent of the veneering ceramic tested in this study it does not seem, as previously discussed, that the different strength values obtained in the veneering ceramics tested are related with the bond between the Zirconia ceramic material and the different veneering ceramics. The results are probably related to differences in the coefficient of thermal expansion between the veneering ceramics and the Zirconia core.

In a few samples the cone cracking caused partial delamination as a result of the extension of the cone crack up to and along the interface and radial cracks progressed along the veneering ceramic causing catastrophic failure through fracture of the Zirconia core material. This finding is consistent with claims made in a review of the mode of fracture of flat bi- and trilayered specimens tested with the veneering ceramic on top (Lawn *et al*., 2001). Formation of a cone crack developed as a result of the contact with a blunt indenter (corresponding to the loading piston of this study), followed by radial cracks that initiated at the inner tensile surface and extended radially outward within this layer. Such cracks are believed to be responsible for bulk fracture. That study also provided an analytic analysis that relates the radial cracks in bilayered specimens to the primary parameters and showed the linear dependence with strength and quadratic dependence with layer thickness.

The bilayer disc mechanical model used in the present study have been validated by finite element analysis and correlated with failure behavior

(Lang *et al*., 2001). Although finite element analysis can identify important trends and has relevance to more complex clinical situations, it does have some disadvantages (Kelly *et al.*, 1995; Kelly, 1999). It has much simpler geometry than a fixed partial denture. It lacks thinner stress-concentrating connectors. It does not have outer layers of ceramic on both the compressive and tensile surfaces, as fixed partial dentures often do. It is not supported by flexible dentine, a flexible periodontal ligament, or flexible bone. However, the same mechanical principles do apply to crowns and fixed partial dentures. The model is also relevant because both all-ceramic crowns and fixed partial dentures are thought to fail most often by crack initiation during tensile loading (Kelly *et al*., 1989; Thompson *et al*., 1994).

Predictive models, such as the finite element analysis bilayer disc models, are widely used to study well defined systems with known parameters. However, the current study suggests that factors such as less than optimal interfaces and residual stresses should be included in theoretical models, increasing their complexity and necessitating the initial exploration of such factors. Investigation of the effects of residual stresses, various interfacial bond strengths, and processing defects by relatively efficient theoretical methods could be most enlightening.

It is important to note that quasi-static mechanical strength tests, used in this study, are only a first step toward predicting clinical performance. Dental ceramics are susceptible to the effects of chemical fatigue, or stress-corrosion, as well as to the effects of cyclic mechanical fatigue (White, 1993; White *et al*., 1997). However, comparative quasi-static mechanical testing does provide a basis for initial comparison, and stronger dental ceramic systems are known to give superior clinical performance compared to weaker systems (Denry *et al*., 2008).

Numerous investigators have been interested in the maximal bite forces used during mastication (Anderson, 1956; Gibbs *et al*., 1981; Gibbs *et* *al*., 1986) in order to understand the resistance and mode of fracture of ceramic restorations. Apart from individual anatomic and physiologic characteristics, it has been shown that bite force varies with the region in the oral cavity. The greatest bite force was found in the first molar region, whereas at the incisors it decreased to only about one third to one fourth that in the molar region. In different studies, mean values for the maximal force level have varied from 216 to 847 N. (Helkimo *et al*., 1977; Howell *et al*., 1959; Linderholm *et al*., 1970; Ringqvist, 1973; Waltimo *et al*., 1993; Waltimo *et al*., 1993). For the incisal region, smaller values ranging from 108 to 299 N have been reported (Helkimo *et al*., 1977; Linderholm *et al*., 1970; Ringqvist, 1973; Waltimo *et al*., 1993). Men often achieve significantly greater bite forces than women (Waltimo *et al*., 1993; Waltimo *et al*., 1993). Additionally, cyclic fatigue loading and stress corrosion fatigue caused by the oral environment must be considered. According to the results of this study, such maximal loads would be sufficient to cause ceramic failure, but not core failure, on the tensile surfaces of fixed partial dentures and crowns of similar dimensions of the discs tested. For this reason it is again recommended that veneering ceramic not to be placed on tensile undersurfaces of connectors or other areas of high tensile stress that are not supported by the core material. Similarly, the results of this study suggest that Zirconia based restorations with a very thin core might not withstand maximal occlusal forces, even when the core is placed in tension and the veneering ceramic is protected in compression. For this reason, it is recommended that thin cores not to be used, even when the tensile undersurface is composed of core material.

Although much work has been focused on Zirconia ceramics during the last decade, most of this work has been performed in industrial, not academic settings. Consequently, abundant data are available in non-refereed commercial product technical data sheets, but much less has been reported in refereed scientific journals. Review of many commercial product data suggests that the data in this study are consistent with mid-range performance of similar partially yttria-stabilized Zirconia. This and some other strength values listed by the manufacturer fall within the upper ranges of those reported in referred and no refereed literature. It is possible that such a difference could be the result of differing specimen and test configuration, surface preparation, and experimental conditions. However, the experimental parameters used in this investigation fall within widely established guidelines for the flexural testing of brittle materials.

There is considerable controversy surrounding the strengths of various ceramic materials, and contradictory research will undoubtedly continue to appear in the literature. Reliable laboratory data concerning the strengths of brittle material are difficult to obtain and coefficients of variation are high. Values are affected by a variety of factors such as geometry, temperature, loading rates, technique variations, and fabrication and thermally-induced imperfections. When testing crowns, mounted on laboratory materials, inherent material strength may not be measured as much as the characteristics of the load – the base for loading, the load-bearing applicator, and the rate of load application. All these factors partially account for variations in reported flexure strengths. Flexure strength is used as a measure of crack propagation from surface microcrack, traditionally on the undersurface of a bar or a disk. While these and similar laboratory tests may be appropriate for comparison of inherent ultimate tensile strength of dental ceramics, results do not necessarily extrapolate to more complex test specimens or to the situation of the oral cavity. The laboratory cannot accommodate intraoral variables such as the periodontal ligament, the physical properties of the cement, the fit, and occlusion. When crowns are cemented intraorally, factors other than and likely more important than the inherent mechanical strength of the materials come into play. For example, a poorly fitting crown may be weaker in a practical sense than a tightly fitting crown, regardless of the materials used; a linear relationship exists between fit and breaking strength. Rank order strength may be affected as well as magnitude. In addition, the strength of the core materials, because they are used several times in thin cross-sections, may contribute only moderately to the strength of the final restoration, and then only when maximum tensile stresses occur at the internal surface of the restoration.

All these factors emphasize the need for a wide margin of safety when placing brittle materials in stressful environments. The strength of dental ceramics may be of less consequence than clinical factors such as case selection, tooth preparation, supporting structure, and the skill of the dentist and laboratory technician. Success remains dependent upon the skill of the dentist and his or her knowledge of the basic behavior and indications of restorative materials.

Furthermore, there is no single in vitro test variable that can predict clinical performance of ceramic prostheses. Based on reviews, there is an urgent need to develop a comprehensive classification system for identifying clinical prosthesis failures, technical complications and biologic complications. Guidelines on the retrieval of fractured prostheses and/or impressions that capture the fracture surface details should also be developed. The predictive power of in vitro data can be increased by finite element stress analysis and computer programs that estimate the time-dependent nature of ceramic structure survival.

The future of ceramics for dentistry is clearly open to new technologies. However, the greatest challenge in developing all-ceramic compositions or processing methods suitable for dental applications is satisfying strength as well as esthetics, while ceramic materials for industrial applications generally do not need to meet esthetic requirements. As pointed out earlier, research is now focusing on fractographic analysis of clinically failed restorations, measure of fatigue parameters, and lifetime prediction of ceramic restorations.

It is now established that at least a five-year evaluation period must be completed before a long-term prognosis can be proposed. The survival rates of Zirconia-based restorations are promising. However, important improvements of the veneering systems are required, and randomized, controlled clinical trials are necessary for restorations in function.

The metal-ceramic technique is still the most commonly used procedure in restorative dentistry, and the success of new all-ceramic systems will depend as much on developmental as on analytical research.

CHAPTER 6

CONCLUSIONS

From the three experimental studies performed in the present work, the following conclusions can be drawn:

1) The results require the rejection of the null hypothesis that there are no significant differences in the load fracture resistance, measured in terms of biaxial flexural strength, among different ceramic surface treatments in the feldspathic veneering ceramics.

2) The results require the rejection of the null hypothesis that there are no significant differences in the load fracture resistance, measured in terms of biaxial flexural strength, among different ceramic surface treatments in each feldspathic veneering ceramic.

3) The results require the rejection of the null hypothesis that there are no significant differences in the load fracture resistance, measured in terms of biaxial flexural strength, among the feldspathic veneering ceramics/Zirconia ceramic frameworks independently of the side tested under tensile stress.

4) The results require the partial rejection of the null hypothesis that there are no significant differences in the load fracture resistance, measured in terms of biaxial flexural strength, among each feldspathic veneering

ceramic/Zirconia ceramic frameworks independently of the side tested under tensile stress.

5) The results require the rejection of the null hypothesis that there are no significant differences in the mode of fracture among the feldspathic veneering ceramics/Zirconia ceramic frameworks independently of the side tested under tensile stress.

6) Strength values of veneering ceramics for Zirconia are in the same range to those of veneering ceramics for the metal-ceramic technique.

7) Grinding decreases the strength of veneering ceramics and in some cases significantly.

8) Polishing and glazing have improved the strength of all materials and in same cases significantly.

9) The results of this study suggest that surface roughness determines the strength of a ceramic material, except where the material has an inner structure which causes an even larger stress concentration than that caused by the combination of surface roughness and flaws.

10) The Weibull modulus results for the three ceramic types tested are in good agreement with values obtained in other studies found in the literature, and correspond to the Weibull modulus range reported for most ceramics.

11) The strength, reliability, and mode of fracture of bilayered ceramic specimens are mainly dictated by the material undergoing biaxial tensile stress.

12) In bilayered ceramic specimens with the core material in tension, the strength and reliability were improved by the core material that processed better mechanical properties

12) The contribution of stronger and tougher core materials to the performance of all-ceramic Zirconia-based restorations may be offset by the weaker vennering ceramic if the design of the restorations does not take into account the actual distribution of the tensile stresses.

13) In Zirconia-based restorations the veneering ceramic can be considered the weakest factor in terms of material resistance.

14) When the Zirconia core material was placed in tension there was a significant increase of the extension of the Hertzian cone crack that was accompanied by partial delamination of the veneering ceramic without fracture of the Zirconia core.

15) When the veneering ceramic was placed in tension, the fracture tended to originate from a major defect at the bottom surface of the ceramic and propagate toward the interface. The cracks were deflected laterally when the stronger Zirconia core was reached demonstrating a system that could have a tendency to produce porcelain “pop-off” rather than catastrophic failure.

SUMMARY

New processing techniques have facilitated the use of Zirconia core materials in all-ceramic dental prostheses. Zirconia has many potential advantages compared to existing core materials; however its performance when layered with veneering ceramics has not been clearly evaluated. Moreover the veneering ceramics used with Zirconia may be ground, polished or glazed during laboratory procedures and/or clinical adjustments. These treatments may affect their strength by introducing microscopic flaws and defects. The purposes of this study were to investigate the effects of surface treatments on the mean biaxial flexural strength of three feldspathic veneering ceramics used to layer Zirconia cores: NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Sweden), Lava™ Ceram veneer ceramic (3M™, ESPE™, Germany), and Vita® VM®9 veneer ceramic (Vita®, Zahnfabrick, Germany) and compare the mean biaxial flexural strength, its reliability, and mode of fracture of bilayered Zirconia discs veneered with the three feldspathic veneering ceramics.

For the first part of the study one hundred and eighty monolithic disc specimens (12.7 mm x 2.2mm), sixty for each feldspathic veneering ceramic were prepared according to the manufacturer's instruction and divided into eighteen groups, 6 groups for each feldspathic veneering ceramic with 10 specimens for each group. The six groups for each feldspathic veneering ceramic were untreated, grounded, grounded and polished, grounded and glazed, grounded polished and glazed and polished and glazed. Mean biaxial flexural strength and Weibull modulus were appraised. Statistical significance

among groups of population was analyzed using one-way and two-way ANOVA and Fisher's PLSD comparison tests. For the second part of the study sixty bilayered disc specimens (12.7 mm x 2.2 mm), twenty for each feldspathic veneering ceramic were prepared using sixty Zirconia core discs (12.7 mm x 1.1 mm) layered with the three feldspathic veneering ceramics according to the manufacturer's instruction and divided into 6 groups of 10 specimens for each material. Mean biaxial flexural strength and Weibull modulus were appraised, and a scanning electron microscope was used to describe surface features. Statistical significance among groups of population was analyzed using two-way ANOVA, Fisher's PLSD and Student's t-test comparison tests.

For the first part of the study and when the veneering ceramics were analyzed together the data provided strong evidence that there was a significant difference in biaxial flexural strength between the grinding groups and all other groups. When the feldspathic veneering ceramics were analyzed individually data revealed more heterogeneity between the mean biaxial strength of different groups. However, grinding decrease the strength of veneering ceramics and in some cases significantly. For this reason, grinding should always be avoided if any other procedure is to be done, as this will either create or change the developed crack dimensions or increase the volume loss. Conversely polishing and glazing improved the strength of all materials and in same cases significantly. These procedures are recommended to counteract the detrimental effects of grinding which was related to the ability of the procedures to improve the condition of the ceramic's surface and free it from various defects and flaws. The Weibull modulus values for the veneering ceramics tested varied with different treatments. They showed higher values for polished, glazed and untreated groups, and lower values for ground groups.

For the bilayered specimens when the veneering ceramics were

analyzed together, specimens with the core material on the bottom surface were statistically stronger and more reliable than those with the veneering ceramics on the bottom surface. When analyzed individually only in the NobelRondo™ Zirconia veneer ceramic there was no significant difference when the core material or the veneering ceramic was on the bottom surface. Two different modes of fracture were observed in the bilayered specimens according to which material was on the bottom surface.

The material that underwent tensile stress dictated the strength, reliability, and fracture mode of the specimens. The design of the restorations and the actual distribution of the tensile stresses must be taken into account, otherwise the significant contribution of stronger and tougher core materials to the performance of all-ceramic Zirconia-based restorations may be offset by the weaker veneering ceramics.

Keywords: Zirconia, veneering ceramics, surface treatments, biaxial flexural strength, mode of fracture.

RESUMO

Com o objectivo de ultrapassar as limitações dos materiais em cerâmica pura tradicionais, diversas companhias introduziram em medicina dentária reabilitadora um material de elevada resistência composto por cerâmica de Zircónia. A sua aplicação em prostodontia está a emergir devido fundamentalmente às suas excelentes propriedades mecânicas, biológicas e estéticas e ao desenvolvimento de novas tecnologias, como a tecnologia CAD/CAM, que permitem a confecção de coroas unitárias e próteses parciais fixas de uma forma standardizada e eficiente.

Apesar das enormes e aparentes vantagens da Zircónia, comparativamente aos materiais cerâmicos tradicionais utilizados como infra-estrutura de restaurações protéticas, o seu desempenho clínico quando estratificada com cerâmicas feldspáticas de revestimento não tem sido até agora avaliada com evidência. Do ponto de vista da selecção de material, a substituição de materiais cerâmicos tradicionais ou mesmo alumina por Zircónia com maior resistência deveria melhorar a performance clínica das coroas tendo como referência a origem da fractura.

No entanto, a resistência das cerâmicas feldspáticas, e consequentemente de uma restauração em cerâmica pura com núcleo de Zircónia, está dependente do grau de polimento final da restauração e dos diferentes procedimentos de fabricação no laboratório e ou ajustes clínicos que possibilitem uma correcta adaptação e ou oclusão. Os procedimentos de processamento e ou ajustes clínicos são passíveis de provocar pequenos defeitos microcópicos e ou fissuras sub-críticas, que poderão ser

acompanhados por uma alteração e consequente redução de resistência à fractura. A presença destas fissuras pode como consequência de carga clínica e ou presença de humidade crescer para uma situação crítica limite levando a falha catastrófica. O efeito dos procedimentos de processamento de materiais cerâmicos tem sido estudado por numerosos investigadores. No entanto, existe ainda controvérsia no que respeita ao melhor método para produzir a superfície mais polida e resistente.

No sentido de avaliar todas estas suposições os objectivos deste estudo foram avaliar a resistência à fractura medida através da resistência à flexão biaxial de cerâmicas feldspáticas de revestimento de Zircónia quando submetidas a tratamentos de superfície nomeadamente, desgaste, polimento e tratamento térmico; e avaliar a resistência, a fiabilidade e o modo de fractura de restaurações em cerâmica pura com infra-estrutura de Zircónia estratificadas com diferentes cerâmicas de revestimento.

Para alcançar estes objectivos, foram efectuadas avaliações quantitativas da resistência à flexão biaxial, avaliações qualitativas da ultra-morfologia e modo de fractura da interface cerâmica de revestimento – infra-estrutura de Zircónia. A estratégia seguida levou à formulação das seguintes hipóteses experimentais:

H_1.0: Não existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, entre os diferentes tratamentos de superfície no conjunto das cerâmicas feldspáticas de revestimento.

H_1.1: Existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, entre os diferentes tratamentos de superfície no conjunto das cerâmicas feldspáticas de revestimento.

$H_2.0$: Não existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, entre os diferentes tratamentos de superfície em cada uma das cerâmicas feldspáticas de revestimento.

$H_2.1$: Existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, entre os diferentes tratamentos de superfície em cada uma das cerâmicas feldspáticas de revestimento.

$H_3.0$: Não existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, no conjunto das cerâmicas feldspáticas de revestimento/infra-estruturas de Zircónia independentemente do material submetido a stress tensional.

$H_3.1$: Existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, no conjunto das cerâmicas feldspáticas de revestimento/infra-estruturas de Zircónia independentemente do material submetido a stress tensional.

$H_4.0$: Não existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, em cada uma das cerâmicas feldspáticas de revestimento/infra-estruturas de Zircónia independentemente do material submetido a stress tensional.

$H_4.1$: Existem diferenças significativas na resistência à fractura, medida em termos de resistência à flexão biaxial, em cada uma das cerâmicas feldspáticas de revestimento/infra-estruturas de Zircónia independentemente do material submetido a stress tensional.

$H_5.0$: Não existem diferenças significativas no modo de fractura das cerâmicas feldspáticas de revestimento/infra-estruturas de Zircónia independentemente do material submetido a stress tensional.

H_5.1: Existem diferenças significativas no modo de fractura das cerâmicas feldspáticas de revestimento/infra-estruturas de Zircónia independentemente do material submetido a stress tensional.

Na primeira parte da investigação uma amostra de conveniência de cento e oitenta (180) espécimes em forma de disco (12.7 mm x 2.2 mm) foram preparados e usados neste estudo. Os discos foram fabricados com cerâmica feldspática utilizada para estratificar infra-estruturas de Zircónia 3Y-TZP de três (3) marcas comerciais: NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Suécia), Lava™ Ceram veneer ceramic (3M™, ESPE™, Alemanha), e Vita® VM®9 veneer ceramic (Vita®, Zahnfabrick, Alemanha).

Sessenta (60) espécimes monolíticos de cada cerâmica foram fabricadas de acordo com as instruções ISO/DIS 6872: 1995 (three-point and biaxial flexural strength) usando um molde de aço inoxidável (ISO/DIS 6872: 1995). O líquido de mistura e o pó cerâmico foram combinados nas proporções recomendadas pelo fabricante. A mistura resultante de cerâmica feldspática foi vibrada e compactada no molde e posteriormente sinterizada em forno específico para cerâmica (Programat P500, Ivoclar Vivadent AG, Liechtenstein). Após a primeira sinterização mais cerâmica foi adicionada por forma a compensar a contracção resultante da primeira sinterização.

Os discos de cerâmica produzidos foram examinados com um estereomicroscópio (Nikon SMZ-U, Tokyo, Japan) com uma ampliação X75 para avaliar a presença de pequenas fissuras ou poros. Espécimes que demonstrassem defeitos visíveis foram substituídos. A superficie de todas os espécimes foi posteriormente polida com discos de carbureto de silício (grão P220, P500, P1200 - Ultra-Prep, Buehler Ltd., Lake Bluff, IL, EUA) numa lixadora mecânica (Ecomet® 3, Buehler Ltd., Lake Buff, IL, EUA) de acordo com ISO 6344-1: 1998 (ISO/DIS 6344-1: 1998).

Este procedimento foi efectuado até serem obtidos espécimes com 2.2(± 0.1) mm por 12.7(± 0.1) mm de espessura e diâmetro respectivamente. Um transportador especial de aço inoxidável foi utilizado para assegurar a uniformização da espessura e paralelismo das superficies durante o corte e polimento. As dimensões dos espécimes foram avaliadas através da utilização de um medidor digital (Digimatic Caliper Series 500, Mitutoyo America Corporation, Aurora, IL, EUA) por forma a garantir espessura e diâmetros exactos. Finalmente, todas os espécimes foram limpas com água destilada num banho de ultra-sons (Eurosonic® 4D, Euronda, Vicenza, Italia) durante 15 minutos e posteriormente colocados no forno específico para cerâmica onde foram auto-glazeados. Após o glaze, a espessura final e o diâmetro foram novamente avaliados através da utilização do mesmo medidor digital até ao centésimo de milimetro.

Os cento e oitenta (180) espécimes foram aleatoriamente distribuidos por dezoito grupos, seis grupos para cada cerâmica, cada grupo composto por dez espécimes. Os seis grupos experimentais de cada cerâmica foram fabricados como anteriormente descrito e submetidos aos seguintes tratamentos de superfície: 1) preparação segundo as instruções do fabricante (grupo de controlo) (C), 2) com desgaste/corte da superfície com instrumento de diamante (G), 3) com desgaste/corte da superfície com instrumento de diamante seguido de glaze (GG), 4) com desgaste/corte da superfície com instrumento de diamante seguido de polimento (GP), 5) com desgaste/corte da superfície com instrumento de diamante seguido de polimento e glaze (GPG), 6) Com polimento da superfície seguido de glaze (PG).

A resistência máxima à fractura foi medida através do método “piston-on-three-ball” utilizando uma máquina de testes mecânicos universal Instron (Modelo TT-BM Instron Corp., Canton, MA), e de acordo com o standard ISO/DIS 6872 para cerâmicas dentárias (ISO/DIS 6872: 1995). A resistência máxima à fractura (N) foi registada e conjugada com a seguinte formula (ASTM F 394-78, 1996), por forma a obter e calcular a resistência à flexão biaxial para cada espécime: $S = - 0.2387\ P(X - Y)/d^2$ (ISO/DIS 6872: 1995). Conjuntamente foi calculado o Weibull modulus para a resistência à flexão biaxial obtida.

A análise estatística dos resultados foi efectuada através da utilização do método one-way and two-way ANOVA com ajustamento de Fisher's PLSD *post-hoc* para comparações múltiplas (p=0.05), para avaliar as diferenças de resistência à fractura entre grupos.

Na segunda parte da investigação uma amostra de coveniência de sessenta (60) espécimes em forma de disco (12.7 mm x 2.2 mm) foram preparados e usados neste estudo. Os discos foram fabricados utilizando sessenta discos de Zircónia 3Y-TZP produzidos por CAD/CAM (Nobel Biocare™ AB, Suécia) (12.7 mm x 1.1 mm) que foram revestidos com cerâmica feldspática utilizada para estratificar infra-estruturas de Zircónia 3Y-TZP de três (3) marcas comerciais: NobelRondo™ Zirconia veneer ceramic (Nobel Biocare™ AB, Suécia), Lava™ Ceram veneer ceramic (3M™, ESPE™, Alemanha), e Vita® VM®9 veneer ceramic (Vita®, Zahnfabrick, Alemanha).

A preparação dos sessenta (60) espécimes, vinte (20) de cada cerâmica foi efectuada de acordo com as instruções ISO/DIS 6872: 1995 (three-point and biaxial flexural strength) (ISO/DIS 6872: 1995) usando as recomendações específicas de cada fabricante e de acordo com o procedimento efectado para os espécimes monolíticos de cada cerâmica.

Os sessenta (60) espécimes foram aleatoriamente distribuidos por seis grupos, dois grupos para cada cerâmica, cada grupo composto por dez espécimes.

A resistênicia máxima à fractura foi medida através do método piston-on-three-ball utilizando uma máquina de testes mecânicos universal Instron

(Modelo TT-BM Instron Corp., Canton, MA), e de acordo com o standard ISO/DIS 6872 para cerâmicas dentárias (ISO/DIS 6872: 1995). Em trinta (30) espécimes, dez por cerâmica, a força foi aplicada na cerâmica felsdpática de revestimento. Nos restante trinta (30), dez por cerâmica, a força foi aplicada na infra-estrutura de Zircónia. O objectivo de inverter a posição dos espécimes foi perceber a influência que a cerâmica de revestimento teria na origem interna ou externa da fractura do material. A máxima resistência à fractura (N) foi registada e conjugada com a seguinte formula (ASTM F 394-78, 1996), por forma a obter e calcular a resistência à flexão biaxial para cada especime: $S = - 0.2387 P(X – Y)/d^2$ (ISO/DIS 6872: 1995). Conjuntamente foi calculado o Weibull modulus para a resistência à flexão biaxial obtida.

A análise estatística dos resultados foi efectuada através da utilização do método two-way ANOVA com ajustamento de Fisher's PLSD *post-hoc* e Student’s t-test para comparações múltiplas (p=0.05), para avaliar as diferenças de resistência à fractura entre grupos.

Após os testes de fractura todos os espécimes foram analisados com um estereomicrocópio (Nikon SMZ-U, Tokyo, Japan) com uma ampliação de X75 no sentido de caracterizar a origem e modo de fractura. A caracterização morfológica dos diferentes tipos de fractura registados na interface Zircónia/cerâmica feldspática de revestimento foi efectuada através da utilização de microcopia electrónica de varrimento (SEM) (Amray 1820, Bedford, MA, USA). Seis espécimes representativos, dois de cada grupo, foram seleccionados e fotografias de diferentes amplitudes foram efectudas.

Os resultados para a primeira parte do estudo demonstraram que quando as cerâmicas feldspáticas de revestimento são analizadas em conjunto, os grupos de desgaste (G) apresentarm diferenças significativas em relação a todos os outros grupos. Foram encontradas também diferenças significativas entre os grupos de controlo (C) e os grupos de polimento/glaze (PG). Nenhuma outra diferença significativa de resistência à flexão biaxial, entre os restantes grupos de tratamento de superfície foi encontrada.

Quando as cerâmicas feldspáticas de revestimento foram analizadas individualmente, os resultados encontrados da resistência à flexão biaxial entre os diferentes grupos de tratamento de superfície, foram mais heterogéneos. No entanto, em todos as cerâmicas o tratamento de superfície de degaste/corte (G) provocou uma diminuição de resistência à flexão biaxial, e em alguns casos de forma estatisticamente significativa. Estes resultados sugerem que o desgaste da superfície destas cerâmicas deve ser sempre evitado se nenhum outro tratamento de superfície for efectuado posteriormente, uma vez que o desgaste ou corte com instrumento de diamante poderá criar ou alterar as dimensões de fissuras ou poros pré-existententes diminuindo a resistência do material. Pelo contrário, os resultados da resistência à flexão biaxial para os tratamentos de superficie de polimento e/ou glaze (GG, GP, GPG, PG) melhoraram a resistência à flexão biaxial dos materiais cerâmicos, e em alguns casos de forma estatisticamente significativa. Os resultados sugerem que estes tratamentos de superfície limitam os efeitos do desgaste/corte, devido à sua capacidade para melhorar as condições da superfície da cerâmica, através da eliminação ou diminuição de defeitos, fissura e/ou poros.

Os resultados sugerem que as diferenças de distribuição da resistência à flexão biaxial entre os diferentes grupos de tratamento de superfície estão mais dependentes da rugosidade de superfície de cada cerâmica e consequentemente do tratamento de superfície efectuado, do que com a sua estrutura; excepto quando a estrutura interna do material possa provocar uma concentração de stress superior aquela originada pela rugosidade de superfície e/ou a presença de poros ou fissuras.

Os resultados encontrados demonstraram também, que os valores obtidos para as cerâmicas feldspáticas de revestimento para Zircónia (grupo de controlo), se encontram dentro dos valores que as cerâmicas feldspáticas de revestimento nos sistemas metálicos apresentam.

Os resultados encontrados para o Weibull modulus das três cerâmicas testadas são semelhantes aos valores obtidos para outras cerâmicas feldspáticas de revestimento. Valores mais elevados foram encontrados para os grupos de polimento, glaze e de controlo, demonstrando maior homogeneidade de valores obtidos, do que para os grupos de desgaste.

Os resultados para a segunda parte do estudo demonstraram que, quando os valores da resistência à flexão biaxial das cerâmicas feldspáticas de revestimento/infra-estrutura Zircónia são analizadas em conjunto, os espécimes que foram testados com a infra-estrutura de Zircónia na superfície inferior, apresentaram valores estatisticamente superiores aqueles que apresentaram as cerâmicas feldspáticas de revestimento sob tensão. Quando analizadas individualmente apenas a NobelRondo^{TM} Zirconia veneer ceramic não demonstrou diferenças significativas independentemente do material que era colocado sob tensão. Estes resultados demonstram, que o contributo de infra-estruturas mais resistentes no desempenho clínico de restaurações em cerâmica pura, poderá não ser significativo se o desenho da restauração não tiver em atenção a distribuição do stress tensional sobre ela exercido.

Os resultados encontrados para o Weibull modulus das três cerâmicas testadas são semelhantes aos valores obtidos para outras cerâmicas feldspáticas de revestimento quando estratificadas sobre infra-estruturas diferentes da Zircónia. Valores mais elevados foram encontrados para os grupos que apresentavam a Zircónia sob tensão, evidenciando maior homogeneidade de valores obtidos.

Dois modos de fractura diferentes foram predominantemente encontrados nos espécimes, dependendo do material que era colocado em tensão. Quando a Zircónia era colocada em tensão, um cone Hertziano estava presente na superfície da cerâmica feldspática em practicamente todos os espécimes. A presença deste cone, era acompanhado por traços de

fractura que se propagavam lateralmente causando eventual delaminação parcial da cerâmica feldspática de revestimento sem fractura da infra-estrutura de Zircónia. Pelo contrário, quando as cerâmicas de revestimento eram colocadas sob tensão, a fractura tinha origem tendencialmente num defeito da superfície da cerâmica que se propagava até à interface cerâmica de revestimento/ infra-estutura de Zircónia. Estes traços de fractura eram defletidos lateralmente quando a infra-estrutura de Zircónia era atingida provocando delaminação da cerâmica feldspática de revestimento, demonstrando um sistema que poderá ter tendência para delaminação parcial ou completa em vez de falha catastrófica. A continuição da aplicação da força provocou em practicamente todos os espécimes fractura da infra-estrutura de Zircónia.

Palavras Chave: Zircónia, cerâmicas feldspáticas de revestimento, tratamentos de superfície, resistência à flexão biaxial, modo de fractura.

APPENDIX 1- MATERIALS, MANUFACTURERS, COMPONENTS AND BATCH NUMBERS

Material	Manufacturer	Components	Batch Number
NobelRondo Base Liner	Nobel Biocare AB, Gothenburg, Sweden	Dental Ceramic NobelRondo Zirconia Base Liner	Ref 32453 Lot 0508
NobelRondo Dentine	Nobel Biocare AB, Gothenburg, Sweden	Dental Ceramic NobelRondo Zirconia Dentin A2	Ref 32439 Lot 0709
Lava Ceram Framework Modifier	3M Espe AG, Seefeld, Germany	Zirconia Overlay Porcelain for Lava Frame MO A2	Ref 68577 Lot 167786
Lava Ceram Dentin	3M Espe AG, Seefeld, Germany	Zirconia Overlay Porcelain for Lava Frame D A2	Ref 68501 Lot 189943
Vita VM9 Base Dentine	Vita, Zahnfabrick, Germany	Dental Ceramic Vita VM9 2M2 Base Dentin	Ref 069 Lot 7933
Vita VM9 Dentine	Vita, Zahnfabrick, Germany	Dental Ceramic Vita VM9 2M2 Dentin	Ref 039 Lot 7565

APPENDIX 2- FIRING SCHEDULES OF THE FELDSPHATIC VENEERING CERAMICS

Veneering Ceramic	Pre Drying Temperature (°C)	Pre Drying Time (min)	Heating Rate (°C/min)	Firing Temperature (°C)	Holding Time (min)
NobelRondo Base Liner	575	8	45	930	1
NobelRondo Dentine	575	9	45	910	1
Lava Ceram Framework Modifier	450	6	45	810	1
Lava Ceram Dentin	450	6	45	800	1
Vita VM9 Base Dentine	500	8	55	950	1
Vita VM9 Dentine	500	7	55	910	1

APPENDIX 3- GLOSSARY OF CHEMICAL COMPOUNDS

Name	Chemical formula
Alumina	Al_2O_3
Calcia	CaO
Ceria	CeO_2
Feldspar	$K_2O\ Al_2O_3\ 6SiO_2$
Kaolin	$Al_2O_3\ 2SiO_2\ 2H_20$
Leucite	$K\ Al\ Si_2O_6$
Lithium Disilicate	$Li_2O\ 2SiO_2$
Lithium Orthophosphate	Li_3PO_4
Magnesia	MgO
Silica	SiO_2
Silicate	SiO_3
Titanium Oxide	TiO_2
Yttria	Y_2O_3
Zircon	$Zr\ SiO_4$
Zirconia	ZrO_2
Zirconia Calcia	$ZrO_2\ CaO$
Zirconia Ceria	$ZrO_2\ CeO_2$
Zirconia Magnesia	$ZrO_2\ MgO$
Zirconia Yttria	$ZrO_2\ Y_2O_3$

APPENDIX 4 - GLOSSARY FOR GENERAL ACRONYMS

Abbreviates	Definition
CAD/CAM	Computer Added Design/Computer Added Machining
Ca-TZP	Calcia Tetragonal Zirconia Polycrystals
Ce-TZP	Cerium Tetragonal Zirconia Polycrystals
FPD	Fixed Partial Denture
GPa	GigaPascal
HIPped	Hot Isostatically Pressed
Mg-PSZ	Magnesium Partially Stabilized Zirconia
MPa	MegaPascal
OM	Optical Microscopy
PSZ	Partially Stabilized Zirconia
RBFPD	Resin Bonded Fixed Partial Denture
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
TZP	Tetragonal Zirconia Polycrystals
Y-TZP	Ytrrium Tetragonal Zirconia Polycrystals
ZTA	Zirconia Toughened Alumina

APPENDIX 5 – RAW DATA

Feldspathic veneering ceramic NobelRondo control group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo	CN	1	12,73	2,20	461,08	136,61
NobelRondo	CN	2	12,77	2,29	262,81	75,21
NobelRondo	CN	3	12,72	2,24	278,39	87,73
NobelRondo	CN	4	12,75	2,28	362,51	106,46
NobelRondo	CN	5	12,75	2,27	334,39	90,26
NobelRondo	CN	6	12,79	2,30	303,77	93,24
NobelRondo	CN	7	12,71	2,4	388,25	106,59
NobelRondo	CN	8	12,68	2,28	504,43	148,25
NobelRondo	CN	9	12,74	2,28	381,92	110,26
NobelRondo	CN	10	12,72	2,30	341,98	101,34

Feldspathic veneering ceramic NobelRondo grinding group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo	G	1	12,75	2,25	204,56	63,87
NobelRondo	G	2	12,74	2,21	189,67	61,39
NobelRondo	G	3	12,76	2,24	327,74	103,24
NobelRondo	G	4	12,71	2,19	237,79	78,41
NobelRondo	G	5	12,76	2,19	180,43	59,46
NobelRondo	G	6	12,75	2,21	290,39	93,98
NobelRondo	G	7	12,71	2,21	185,58	60,09
NobelRondo	G	8	12,75	2,2	206,93	67,58
NobelRondo	G	9	12,73	2,21	247.97	79,54
NobelRondo	G	10	12,74	2,22	207,79	66,65

Feldspathic veneering ceramic NobelRondo grinding and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo	GG	1	12,72	2,3	281,43	84,12
NobelRondo	GG	2	12,78	2,3	214,44	64,06
NobelRondo	GG	3	12,72	2,29	211,03	60,42
NobelRondo	GG	4	12,74	2,30	231,36	67,95
NobelRondo	GG	5	12,75	2,23	239,87	76,25
NobelRondo	GG	6	12,78	2,27	318,45	97,66
NobelRondo	GG	7	12,71	2,27	361,82	104,50
NobelRondo	GG	8	12,72	2,28	299,11	90,98
NobelRondo	GG	9	12,73	2,25	304,66	87,22
NobelRondo	GG	10	12,74	2,28	242,43	71,20

Feldspathic veneering ceramic NobelRondo grinding and polishing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo	GP	1	12,72	2,25	280,30	87,55
NobelRondo	GP	2	12,72	2,23	267,18	84,95
NobelRondo	GP	3	12,74	2,28	418.00	132,90
NobelRondo	GP	4	12,78	2,22	277,26	88,90
NobelRondo	GP	5	12,72	2,27	263,17	80,76
NobelRondo	GP	6	12,72	2,20	345,48	114,95
NobelRondo	GP	7	12,75	2,23	325,19	103,37
NobelRondo	GP	8	12,75	2,26	330.86	104,90
NobelRondo	GP	9	12,74	2,26	348,63	107,91
NobelRondo	GP	10	12,74	2,22	292,94	93,97

Feldspathic veneering ceramic NobelRondo grinding polishing and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo	GPG	1	12,7	2,17	188.88	63,43
NobelRondo	GPG	2	12,71	2,17	246,76	82,87
NobelRondo	GPG	3	12,68	2,2	307,50	100,50
NobelRondo	GPG	4	12,75	2,22	223,87	71,80
NobelRondo	GPG	5	12,67	2,11	309,29	109,91
NobelRondo	GPG	6	12,72	2,21	224,39	72,65
NobelRondo	GPG	7	12,68	2,22	296,67	95,22
NobelRondo	GPG	8	12,71	2,22	341,30	109,51
NobelRondo	GPG	9	12,7	2,2	277,62	90,72
NobelRondo	GPG	10	12,71	2,19	258,49	85,23

Feldspathic veneering ceramic NobelRondo polishing and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo	PG	1	12,62	2,28	206,04	62,74
NobelRondo	PG	2	12,75	2,23	275,84	87,68
NobelRondo	PG	3	12,73	2,30	269,08	77,04
NobelRondo	PG	4	12,72	2,30	269,46	77,15
NobelRondo	PG	5	12,78	2,28	334,93	101,81
NobelRondo	PG	6	12,78	2,30	363,60	108,61
NobelRondo	PG	7	12,74	2,20	246,31	72,34
NobelRondo	PG	8	12,72	2,30	269,13	80,44
NobelRondo	PG	9	12,74	2,28	245,55	68,53
NobelRondo	PG	10	12,71	2,29	312,20	90,17

Feldspathic veneering ceramic Lava Ceram control group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram	CN	1	12,71	2,22	260,19	83,49
Lava Ceram	CN	2	12,75	2,22	399,33	128,08
Lava Ceram	CN	3	12,79	2,23	316,24	100,37
Lava Ceram	CN	4	12,72	2,16	222,77	75,50
Lava Ceram	CN	5	12,79	2,27	341,85	104,75
Lava Ceram	CN	6	12,65	2,21	323,91	104,95
Lava Ceram	CN	7	12,72	2,22	288,69	92,62
Lava Ceram	CN	8	12,76	2,28	329,15	100,08
Lava Ceram	CN	9	12,79	2,22	327,19	104,90
Lava Ceram	CN	10	12,77	2,23	293,15	93,16

Feldspathic veneering ceramic Lava Ceram control group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram	CN	1	12,71	2,22	260,19	83,49
Lava Ceram	CN	2	12,75	2,22	399,33	128,08
Lava Ceram	CN	3	12,79	2,23	316,24	100,37
Lava Ceram	CN	4	12,72	2,16	222,77	75,50
Lava Ceram	CN	5	12,79	2,27	341,85	104,75
Lava Ceram	CN	6	12,65	2,21	323,91	104,95
Lava Ceram	CN	7	12,72	2,22	288,69	92,62
Lava Ceram	CN	8	12,76	2,28	329,15	100,08
Lava Ceram	CN	9	12,79	2,22	327,19	104,90
Lava Ceram	CN	10	12,77	2,23	293,15	93,16

Feldspathic veneering ceramic Lava Ceram grinding group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram	G	1	12,67	2,17	312,80	105,09
Lava Ceram	G	2	12,74	2,17	248,46	83,41
Lava Ceram	G	3	12,78	2,16	302,78	102,55
Lava Ceram	G	4	12,74	2,19	189,97	62,69
Lava Ceram	G	5	12,77	2,17	275,51	92,46
Lava Ceram	G	6	12,71	2,20	317,83	103,85
Lava Ceram	G	7	12,72	2,22	257,69	82,68
Lava Ceram	G	8	12,71	2,20	268,68	88,11
Lava Ceram	G	9	12,72	2,18	275,36	91,62
Lava Ceram	G	10	12,71	2,19	233,65	77,04

Feldspathic veneering ceramic Lava Ceram grinding and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram	GG	1	12,65	2,17	245,65	82,55
Lava Ceram	GG	2	12,79	2,16	315,61	106,81
Lava Ceram	GG	3	12,72	2,18	221,91	73,83
Lava Ceram	GG	4	12,70	2,19	382,71	126,63
Lava Ceram	GG	5	12,80	2,20	326,25	106,49
Lava Ceram	GG	6	12,74	2,19	307,96	101,51
Lava Ceram	GG	7	12,80	2,17	419,54	140,76
Lava Ceram	GG	8	12,74	2,20	330,55	107,97
Lava Ceram	GG	9	12,73	2,17	340,81	114,43
Lava Ceram	GG	10	12,74	2,18	320,63	106,66

Feldspathic veneering ceramic Lava Ceram grinding and polishing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram	GP	1	12,68	2,18	296,28	98,62
Lava Ceram	GP	2	12,74	2,19	375,07	123,63
Lava Ceram	GP	3	12,77	2,21	357,32	115,62
Lava Ceram	GP	4	12,67	2,19	329,47	108,68
Lava Ceram	GP	5	12,74	2,20	276,69	90,37
Lava Ceram	GP	6	12,66	2,21	245,14	79,42
Lava Ceram	GP	7	12,67	2,20	266,66	87,16
Lava Ceram	GP	8	12,80	2,22	238,23	76,32
Lava Ceram	GP	9	12,74	2,20	356,24	116,36
Lava Ceram	GP	10	12,71	2,20	323,77	105,79

Feldspathic veneering ceramic Lava Ceram grinding polishing and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram	GPG	1	12,80	2,17	292,66	98,17
Lava Ceram	GPG	2	12,70	2,18	272,36	90,70
Lava Ceram	GPG	3	12,80	2,21	324,83	105,07
Lava Ceram	GPG	4	12,72	2,18	262,66	87,39
Lava Ceram	GPG	5	12,80	2,21	333,98	108,03
Lava Ceram	GPG	6	12,78	2,20	330,09	107,77
Lava Ceram	GPG	7	12,78	2,12	298,02	104,78
Lava Ceram	GPG	8	12,82	2,19	358,96	118,22
Lava Ceram	GPG	9	12,79	2,17	340,53	114,26
Lava Ceram	GPG	10	12,77	2,18	320,01	106,42

Feldspathic veneering ceramic Lava Ceram polishing and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram	PG	1	12,74	2,26	332,39	102,88
Lava Ceram	PG	2	12,80	2,29	280,76	84,58
Lava Ceram	PG	3	12,79	2,21	247,51	79,98
Lava Ceram	PG	4	12,74	2,26	357,10	110,53
Lava Ceram	PG	5	12,71	2,26	254,36	78,76
Lava Ceram	PG	6	12,68	2,29	308,71	93,12
Lava Ceram	PG	7	12,65	2,21	312,53	101,26
Lava Ceram	PG	8	12,74	2,17	297,11	99,75
Lava Ceram	PG	9	12,80	2,19	340,64	112,20
Lava Ceram	PG	10	12,75	2,24	377,54	111,94

Feldspathic veneering ceramic Vita VM9 control group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9	CN	1	12,66	2,23	267,20	85,02
Vita VM9	CN	2	12,67	2,22	252,63	81,10
Vita VM9	CN	3	12,71	2,21	272,14	88,11
Vita VM9	CN	4	12,79	2,18	282,75	94,01
Vita VM9	CN	5	12,67	2,24	301,15	94,95
Vita VM9	CN	6	12,74	2,27	282,14	86,56
Vita VM9	CN	7	12,74	2,26	301,96	93,46
Vita VM9	CN	8	12,62	2,27	312,73	96,09
Vita VM9	CN	9	12,68	2,24	304,38	95,96
Vita VM9	CN	10	12,72	2,23	301,84	95,97

Feldspathic veneering ceramic Vita VM9 grinding group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9	G	1	12,64	2,21	262,76	85,14
Vita VM9	G	2	12,61	2,15	204,53	70,11
Vita VM9	G	3	12,63	2,11	264,43	94,01
Vita VM9	G	4	12,68	2,11	199,68	71,63
Vita VM9	G	5	12,63	2,18	227,37	75,73
Vita VM9	G	6	12,63	2,15	225,59	77,24
Vita VM9	G	7	12,67	2,16	200,83	68,10
Vita VM9	G	8	12,68	2,12	238,66	84,00
Vita VM9	G	9	12,67	2,15	228,56	78,23
Vita VM9	G	10	12,61	2,14	259,40	84,79

Feldspathic veneering ceramic Vita VM9 grinding and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9	GG	1	12,72	2,17	269,04	90,34
Vita VM9	GG	2	12,64	2,12	259,40	91,34
Vita VM9	GG	3	12,66	2,16	275,41	93,72
Vita VM9	GG	4	12,68	2,20	264,88	86,57
Vita VM9	GG	5	12,62	2,17	233,99	78,66
Vita VM9	GG	6	12,64	2,18	275,29	91,67
Vita VM9	GG	7	12,69	2,20	266,43	87,07
Vita VM9	GG	8	12,65	2,18	243,59	81,11
Vita VM9	GG	9	12,67	2,17	291,77	98,03
Vita VM9	GG	10	12,66	2,18	296,43	98,69

Feldspathic veneering ceramic Vita VM9 grinding and polishing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9	GP	1	12,67	2,19	263,80	87,02
Vita VM9	GP	2	12,71	2,20	265,30	86,68
Vita VM9	GP	3	12,77	2,12	240,37	84,52
Vita VM9	GP	4	12,62	2,15	242,13	82,92
Vita VM9	GP	5	12,77	2,16	239,97	81,20
Vita VM9	GP	6	12,80	2,18	259,55	86,27
Vita VM9	GP	7	12,68	2,19	269,54	89,00
Vita VM9	GP	8	12,64	2,24	248,56	78,40
Vita VM9	GP	9	12,72	2,18	289,55	96,34
Vita VM9	GP	10	12,70	2,17	286,33	96,17

Feldspathic veneering ceramic Vita VM9 grinding polishing and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9	GPG	1	12,73	2,19	256,73	84,63
Vita VM9	GPG	2	12,63	2,19	239,98	79,20
Vita VM9	GPG	3	12,72	2,19	283,51	93,47
Vita VM9	GPG	4	12,71	2,18	257,57	85,71
Vita VM9	GPG	5	12,64	2,17	233,43	78,45
Vita VM9	GPG	6	12,80	2,21	280,49	90,71
Vita VM9	GPG	7	12,80	2,20	235,11	76,73
Vita VM9	GPG	8	12,70	2,18	240,61	80,07
Vita VM9	GPG	9	12,72	2,19	268,88	88,65
Vita VM9	GPG	10	12,71	2,19	267,66	88,25

Feldspathic veneering ceramic Vita VM9 polishing and glazing group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9	PG	1	12,68	2,19	254,87	84,06
Vita VM9	PG	2	12,71	2,21	295,21	95.58
Vita VM9	PG	3	12,64	2,20	286,77	93,77
Vita VM9	PG	4	12,69	2,24	302,62	95,40
Vita VM9	PG	5	12,71	2,23	317,89	101,09
Vita VM9	PG	6	12,61	2,28	275,47	83,89
Vita VM9	PG	7	12,67	2,27	300,82	94,87
Vita VM9	PG	8	12,65	2,24	267,48	84,36
Vita VM9	PG	9	12,67	2,23	304,27	96,80
Vita VM9	PG	10	12,68	2,23	281,49	89,54

Feldspathic veneering ceramic NobelRondo and Zirconia group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo Zirconia	ZR Bottom	1	12,68	2,44	1392,79	378,55
NobelRondo Zirconia	ZR Bottom	2	12,67	2,4	2126,96	597,80
NobelRondo Zirconia	ZR Bottom	3	12,69	2,43	1816,37	497,74
NobelRondo Zirconia	ZR Bottom	4	12,68	2,33	2113,96	689,31
NobelRondo Zirconia	ZR Bottom	5	12,66	2,41	1952,62	544,26
NobelRondo Zirconia	ZR Bottom	6	12,67	2,50	1456,48	376,92
NobelRondo Zirconia	ZR Bottom	7	12,66	2,42	1813,04	501,14
NobelRondo Zirconia	ZR Bottom	8	12,67	2,47	2115,30	560,94
NobelRondo Zirconia	ZR Bottom	9	12,68	2,43	1821,56	499,21
NobelRondo Zirconia	ZR Bottom	10	12,66	2,42	1956,11	540,69

Feldspathic veneering ceramic NobelRondo and Zirconia group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
NobelRondo Zirconia	ZR Top	1	12,66	2,41	1012,09	282,10
NobelRondo Zirconia	ZR Top	2	12,67	2,43	808,59	221,62
NobelRondo Zirconia	ZR Top	3	12,68	2,43	1792,30	491,19
NobelRondo Zirconia	ZR Top	4	12,69	2,41	1845,14	514,15
NobelRondo Zirconia	ZR Top	5	12,68	2,42	1944,10	537,26
NobelRondo Zirconia	ZR Top	6	12,66	2,39	1992,24	564,75
NobelRondo Zirconia	ZR Top	7	12,69	2,42	2088,42	577,08
NobelRondo Zirconia	ZR Top	8	12,69	2,40	2066,80	580,78
NobelRondo Zirconia	ZR Top	9	12,69	2,43	1880,89	515,42
NobelRondo Zirconia	ZR Top	10	12,67	2,44	1784,11	484,96

Feldspathic veneering ceramic Lava Ceram and Zirconia group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram Zirconia	ZR Bottom	1	12,66	2,32	1832.33	524,00
Lava Ceram Zirconia	ZR Bottom	2	12,61	2,40	1488.04	425,54
Lava Ceram Zirconia	ZR Bottom	3	12,63	2,38	1456,98	416,66
Lava Ceram Zirconia	ZR Bottom	4	12,64	2,40	1446,06	406,56
Lava Ceram Zirconia	ZR Bottom	5	12,65	2,41	1715,87	478,32
Lava Ceram Zirconia	ZR Bottom	6	12,66	2,38	1680,34	480,39
Lava Ceram Zirconia	ZR Bottom	7	12,67	2,41	2064,37	575,35
Lava Ceram Zirconia	ZR Bottom	8	12,69	2,37	1985,15	572,21
Lava Ceram Zirconia	ZR Bottom	9	12,68	2,38	1933,59	552,68
Lava Ceram Zirconia	ZR Bottom	10	12,64	2,41	1781,37	496,63

Feldspathic veneering ceramic Lava Ceram and Zirconia group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Lava Ceram Zirconia	ZR Top	1	12,67	2,37	802,15	231,26
Lava Ceram Zirconia	ZR Top	2	12,63	2,38	951,39	272,07
Lava Ceram Zirconia	ZR Top	3	12,64	2,42	800,47	221,30
Lava Ceram Zirconia	ZR Top	4	12,62	2,37	849,56	245,06
Lava Ceram Zirconia	ZR Top	5	12,66	2,41	872,84	243,29
Lava Ceram Zirconia	ZR Top	6	12,66	2,38	900,54	257,45
Lava Ceram Zirconia	ZR Top	7	12,66	2,41	902,37	251,52
Lava Ceram Zirconia	ZR Top	8	12,66	2,40	889,58	250,05
Lava Ceram Zirconia	ZR Top	9	12,68	2,34	889,34	263,07
Lava Ceram Zirconia	ZR Top	10	12,64	2,36	774,20	225,19

Feldspathic veneering ceramic Vita VM9 and Zirconia group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9 Zirconia	ZR Bottom	1	12,67	2,42	1864,33	515,27
Vita VM9 Zirconia	ZR Bottom	2	12,65	2,38	754,98	215,86
Vita VM9 Zirconia	ZR Bottom	3	12,65	2,42	1644,93	454,72
Vita VM9 Zirconia	ZR Bottom	4	12,64	2,41	1259,42	351,12
Vita VM9 Zirconia	ZR Bottom	5	12,68	2,37	1828,07	526,99
Vita VM9 Zirconia	ZR Bottom	6	12,64	2,41	1622,07	452,22
Vita VM9 Zirconia	ZR Bottom	7	12,65	2,42	1531,86	423,47
Vita VM9 Zirconia	ZR Bottom	8	12,63	2,40	1620,93	455,77
Vita VM9 Zirconia	ZR Bottom	9	12,68	2,39	1915,32	542,83
Vita VM9 Zirconia	ZR Bottom	10	12,67	2,42	1939,90	536,15

Feldspathic veneering ceramic Vita VM9 and Zirconia group

Ceramic Material	Group	Sample	Diameter	Thickness	Load Fracture (N)	Load Fracture (MPa)
Vita VM9 Zirconia	ZR Top	1	12,68	2,38	782,03	223,53
Vita VM9 Zirconia	ZR Top	2	12,68	2,42	950,40	262,65
Vita VM9 Zirconia	ZR Top	3	12,63	2,43	917,24	251,51
Vita VM9 Zirconia	ZR Top	4	12,64	2,43	869,32	238,34
Vita VM9 Zirconia	ZR Top	5	12,68	2,39	890,73	252,45
Vita VM9 Zirconia	ZR Top	6	12,64	2,40	795,50	223,65
Vita VM9 Zirconia	ZR Top	7	12,64	2,41	948,21	264,35
Vita VM9 Zirconia	ZR Top	8	12,65	2,40	830,32	233,42
Vita VM9 Zirconia	ZR Top	9	12,65	2,42	812,13	224,50
Vita VM9 Zirconia	ZR Top	10	12,65	2,41	866,21	241,47

Two-way ANOVA table for the biaxial flexural strength (load fracture) of the ceramic and surface finish/heat treatment of test specimens.

Two-Way ANOVA

	DF	Sum of Squares	Mean Square	F-Value	P-Value	Lambda	Power
Ceramic	2	5632,538	2816,269	16,135	<.0001	32,27	1
Surface	5	5610,372	1122,074	6,429	<.0001	32,143	0,998
Ceramic Surface	10	4656,725	465,673	2,668	0,0048	26,679	0,962
Residual	162	28276,09	174,544				

Fisher's PLSD table for the biaxial flexural strength (load fracture) of the ceramic and surface finish/heat treatments of test specimens

Fisher's PLSD for Load Fracture MPa

Effect: Ceramic

Significance Level: 5 %

	Mean Diff.	Crit. Diff	P-Value	
NobelRondo, Lava Ceram	-11,31	4,763	<.0001	S
NobelRondo, Vita VM9	1,041	4,763	0,6665	
Lava Ceram, Vita VM9	12,353	4,763	<.0001	S

Fisher's PLSD for Load Fracture MPa

Effect: Surface

Significance Level: 5 %

	Mean Diff.	Crit. Diff	P-Value	
CN, G	18,06	6,74	<.0001	S
CN, GG	6,2	6,74	0,0712	
CN, GP	3,24	6,74	0,3443	
CN, GPG	6,22	6,74	0,0702	
CN, PG	7,66	6,74	0,0261	S
G, GG	-11,86	6,74	0,0007	S
G, GP	-14,82	6,74	<.0001	S
G, GPG	-11,84	6,74	0,0007	S
G, PG	-10,4	6,74	0,0027	S
GG, GP	-2,96	6,74	0,3868	
GG, GPG	0,02	6,74	0,9948	
GG, PG	1,46	6,74	0,6689	
GP, GPG	2,98	6,74	0,3832	
GP, PG	4,42	6,74	0,1967	
GPG, PG	1,44	6,74	0,6737	

One-Way ANOVA table for the NobelRondo feldspathic veneering ceramic and surface finish/heat treatment of test specimens

NobelRondo							
One-Way ANOVA on Surface Treatment							
	DF	Sum of Squares	Mean Square	F-Value	P-Value	Lambda	Power
Surface Treatment	5	7212,672	1442,534	5,409	0,0004	27,044	0,987
Residual	54	14401,661	266,697				

Fisher’s PLSD table for the NobelRondo feldspathic veneering ceramic and surface finish/heat treatments of test specimens

Fisher's PLSD for Load to Fracture in MPa

Effect: Surface Treatment

Significance Level: 5 %

	Mean Diff.	Crit. Diff	P-Value	
CN, G	32,101	14,642	<.0001	S
CN, GG	25,159	14,642	0,0011	S
CN, GP	6,843	14,642	0,3529	
CN, GPG	17,410	14,642	0,0207	S
CN, PG	22,944	14,642	0,0027	S
G, GG	-6,942	14,642	0,3461	
G, GP	-25,258	14,642	0,0011	S
G, GPG	-14,691	14,642	0,0493	S
G, PG	-9,157	14,642	0,2153	
GG, GP	-18,316	14,642	0,0152	S
GG, GPG	-7,749	14,642	0,2934	
GG, PG	-2,215	14,642	0,7628	
GP, GPG	10,567	14,642	0,1537	
GP, PG	16,101	14,642	0,0318	S
GPG, PG	5,534	14,642	0,4519	

One-Way ANOVA table for the Lava Ceram feldspathic veneering ceramic and surface finish/heat treatment of test specimens

Lava Ceram							
One-Way ANOVA on Surface Treatment							
	DF	Sum of Squares	Mean Square	F-Value	P-Value	Lambda	Power
Surface Treatment	5	1877,336	375,467	1,732	0,1429	8,661	0,548
Residual	54	11705,396	216,760				

Fisher’s PLSD table for the Lava Ceram feldspathic veneering ceramic and surface finish/heat treatments of test specimens

Fisher's PLSD for Load to Fracture in MPa

Effect: Surface Treatment

Significance Level: 5 %

	Mean Diff.	Crit. Diff	P-Value	
CN, G	9,840	13,201	0,1409	
CN, GG	-7,974	13,201	0,2311	
CN, GP	-1,407	13,201	0,8316	
CN, GPG	-5,291	13,201	0,4252	
CN, PG	0,590	13,201	0,9289	
G, GG	-17,814	13,201	0,0091	S
G, GP	-11,247	13,201	0,0933	
G, GPG	-15,131	13,201	0,0255	S
G, PG	-9,250	13,201	0,1658	
GG, GP	6,567	13,201	0,3230	
GG, GPG	2,683	13,201	0,6853	
GG, PG	8,564	13,201	0,1989	
GP, GPG	-3,884	13,201	0,5577	
GP, PG	1,997	13,201	0,7628	
GPG, PG	5,881	13,201	0,3757	

One-Way ANOVA table for the Vita VM9 feldspathic veneering ceramic and surface finish/heat treatment of test specimens

Vita VM9							
One Way ANOVA on Surface Treatment							
	DF	Sum of Squares	Mean Square	F-Value	P-Value	Lambda	Power
Surface Treatment	5	1177,089	235,418	5,86	0,0002	29,3	0,993
Residual	54	2169,396	40,174				

Fisher's PLSD table for the Vita VM9 feldspathic veneering ceramic and surface finish/heat treatments of test specimens

Fisher's PLSD for Load to Fracture in MPa

Effect: Surface Treatment

Significance Level: 5 %

	Mean Diff.	Crit. Diff	P-Value	
CN, G	12,225	5,683	<.0001	S
CN, GG	1,403	5,683	0,6226	
CN, GP	4,271	5,683	0,1377	
CN, GPG	6,536	5,683	0,0250	S
CN, PG	-0,562	5,683	0,8436	
G, GG	-10,822	5,683	0,0003	S
G, GP	-7,954	5,683	0,0070	S
G, GPG	-5,689	5,683	0,0498	S
G, PG	-12,787	5,683	<.0001	S
GG, GP	2,868	5,683	0,3161	
GG, GPG	5,133	5,683	0,0757	
GG, PG	-1,965	5,683	0,4911	
GP, GPG	2,265	5,683	0,4278	
GP, PG	-4,833	5,683	0,0939	
GPG, PG	-7,098	5,683	0,0153	S

Two-way ANOVA table for the biaxial flexural strength (load fracture) of the feldspathic veneering ceramic and core Zirconia location of test specimens

Two-Way ANOVA

	DF	Sum of Squares	Mean Square	F-Value	P-Value	Lambda	Power
Ceramic	2	269214,7	134607,35	20,592	<.0001	41,184	1
Zirconia Location	1	410757,766	410757,766	62,837	<.0001	62,837	1
Ceramic Zirconia Location	2	119490,853	59745,427	9,14	0,0004	18,279	0,978
Residual	54	352993,6	6536,919				

Fisher's PLSD table for the biaxial flexural strength (load fracture) of the feldspathic veneering ceramics and core Zirconia location of test specimens

Fisher's PLSD for Load Fracture MPa nAdj

Effect: Ceramic

Significance Level: 5 %

	Mean Diff.	Crit. Diff	P-Value	
Nobel Rondo, Lava Ceram	127,3	51,26	<.0001	S
Nobel Rondo, Vita VM9	153,3	51,26	<.0001	S
Lava Ceram, Vita VM9	25,94	51,26	0,3148	

Fisher's PLSD for Load Fracture MPa nAdj

Effect: Zirconia located

Significance Level: 5 %

	Mean Diff.	Crit. Diff	P-Value	
Top, Bottom	-165	41,85	<.0001	S

Unpaired t-test table for the NobelRondo feldspathic veneering ceramic and core Zirconia location of test specimens

Ceramic: NobelRondo

Unpaired t-test for Load Fracture MPa nAdj

Grouping Variable: Zirconia location

Hypothesized Difference = 0

	Mean Diff.	DF	t-Value	P-Value
Top, Bottom	-41,725	18	-0,849	0,4073

Group Info for Load Fracture MPa nAdj

Grouping Variable: Zirconia location

	Count	Mean	Variance	Std. Dev.	Std. Err
Top	10	476,931	15382,608	124,027	39,221
Bottom	10	518,656	8795,198	93,783	29,657

Unpaired t-test table for the Lava Ceram feldspathic veneering ceramic and core Zirconia location of test specimens

Ceramic: Lava Ceram

Unpaired t-test for Load Fracture MPa nAdj

Grouping Variable: Zirconia location

Hypothesized Difference = 0

	Mean Diff.	DF	t-Value	P-Value
Top, Bottom	-248,864	18	-11,565	<.0001

Group Info for Load Fracture MPa nAdj

Grouping Variable: Zirconia located

	Count	Mean	Variance	Std. Dev.	Std. Err
Top	10	246,026	268,627	16,39	5,183
Bottom	10	494,89	4361,773	66,044	20,885

Unpaired t-test table for the Vita VM9 feldspathic veneering ceramic and core Zirconia location of test specimens

Ceramic: Vita VM9

Unpaired t-test for Load Fracture MPa nAdj

Grouping Variable: Zirconia located

Hypothesized Difference = 0

	Mean Diff.	DF	t-Value	P-Value
Top, Bottom	-205,853	18	-6,379	<.0001

Group Info for Load Fracture MPa nAdj

Grouping Variable: Zirconia located

	Count	Mean	Variance	Std. Dev.	Std. Err
Top	10	241,587	243,921	15,618	4,939
Bottom	10	447,44	10169,385	100,843	31,889

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