High temperature sound velocity measurement with piezoelectric transducers

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A technique for high temperature sound velocity measurement using piezoelectric transducers, that avoids the existence of the usual high temperature bond between the sample and the buffer, was developed. This bond is frequently responsible for a large attenuation and distortion of the sound field, with a consequent inaccuracy in the experimental results. This technique was successfully used by the authors to make good quality measurements of sound velocity in iron–silicon single crystals from room temperature to $T \approx 1000$ °C, unlike other authors who report strong attenuation in different regions of this temperature range for similar samples. The furnace, which is only 35 mm wide, allows work under magnetic field and a controlled atmosphere up to 1300 °C. © *1998 American Institute of Physics.* [S0034-6748(98)00401-8]

I. INTRODUCTION

The measurement of ultrasonic velocity at high temperatures using piezoelectric transducers is usually done by a pulse superposition technique with a single transducer that acts as emitter and receiver,[1–5] using a buffer between the sample and the transducer, so that the transducer may be kept at room temperature. This buffer is made of a material for which the thermal conductivity and the ultrasonic attenuation are as low as possible. Even when these two conditions are fulfilled, this approach relies on the possibility of making a good buffer to sample bond. This bond, which is usually made with a high temperature ceramic adhesive, must accommodate the different thermal expansion of the two surfaces to bond, which introduce important stresses. For this reason, these bonds are often very difficult to make and, even when they are possible, frequently introduce a large attenuation and distortion of the sound field. Since this attenuation or distortion depend on the bond temperature, it is impossible to correct the experimental data for such effects. Therefore, a technique that avoids this high temperature bond would be very convenient.

In this work we describe an experimental technique of this type: the pulse transit time is the time difference between two echoes, one of which is generated by a cut in the sample a distance from its free end.[6] This makes it unnecessary to use a buffer and, therefore, a high temperature bond, with a consequent improvement in the accuracy of the experimental results.

II. THE EXPERIMENTAL TECHNIQUE

The sample geometry used in our technique is sketched in Fig. 1. Part of the sample is used as a buffer between the cooled region (that is used for the transducer bond) and the hot region (defined by a cut made approximately 10 mm away from the free end of the sample). With this geometry, the velocity measurement is made using the reflections from faces 2 and 3. The cut depth was adjusted so that the two echoes had similar amplitude. This was easily achieved by making the cut with a silicon carbide disk saw, with *in situ* monitoring of the two echoes.

For an iron–silicon cylindrical (8 mm diameter) sample, with the typical dimensions given in the figure, the power that must be injected in the ''buffer region'' to maintain a 1000 °C constant temperature in the hot region was estimated to be about 500 W, if the cooled region is to remain at room temperature. A special furnace was designed, with two heating elements, one of which is directly bonded over the buffer region of the sample. This heating element and the whole sample holder (basically a water cooled cylinder to which the sample was soldered) are schematically drawn in Fig. 2. The surface of the buffer region of the sample was coated with a thin layer of a high temperature ceramic, over which the sample heating element was wound. This heating element was then covered with the same ceramic. Two thermocouples were implanted by arc discharge at the limits of the hot region. As is schematically represented in Fig. 3, the sample holder is inserted into the top of the furnace so that the free end of the sample lies above the hotter part of the second heating element (fixed in the body of the furnace) to ensure that it is always possible to obtain a zero temperature gradient in the hot region of the sample. The furnace uses a proportional controller attached to the sample heating element to control the temperature measured by the thermocouple near the cut. The other heating element is driven by a current proportional to the difference of the temperatures measured by the two thermocouples. With this experimental setup it was possible to obtain a stable temperature in the hot region of the sample, with a difference in the temperature readings of the two thermometers always below 0.2 °C even at the highest temperatures (~ 1000 °C) used. The upper limit of the temperature attainable with our furnace was 1300 °C. The overall lateral dimension of the furnace was only 35 mm, which allowed its insertion between the poles of an electromagnet, for measurements under high magnetic field.

This experimental setup was successfully used by the authors to make good quality sound velocity measurements in iron–silicon single crystals from room temperature to $T \approx 1000$ °C, unlike other authors[7,8] who, for similar samples, report that it was impossible to make measurements in different regions of this temperature range using the conven-

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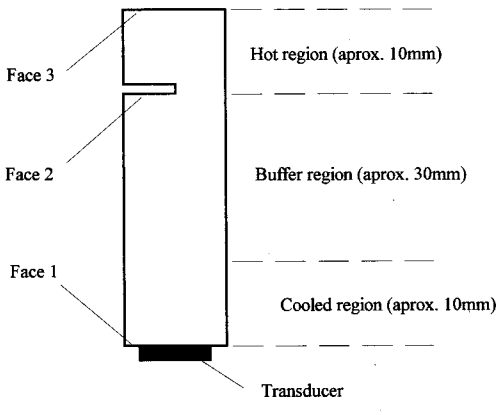

FIG. 1. Sample geometry used for ultrasonic velocity measurements at high temperatures. The velocity is measured using the echoes from faces 2 and 3.

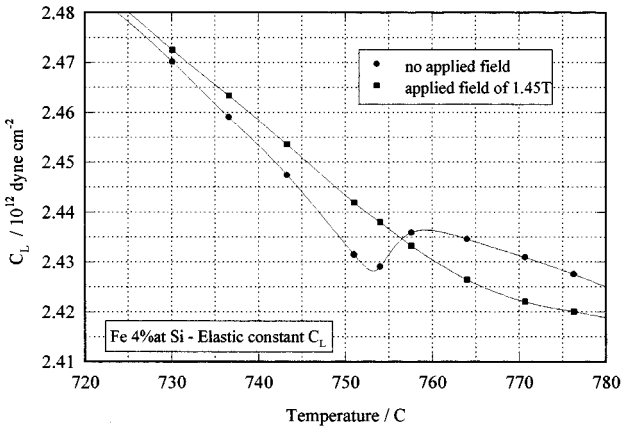

FIG. 4. Elastic constant C_L (propagation along [110] with longitudinal polarization) as measured in a Fe 4 at. % Si sample near the Curie temperature.

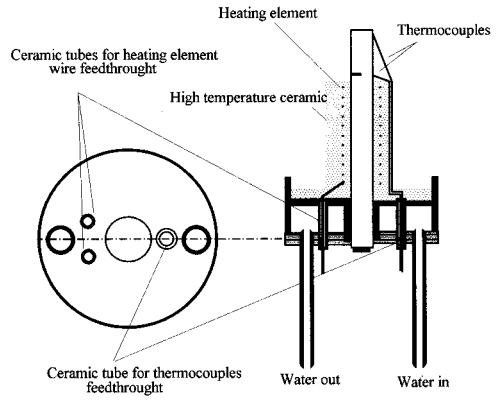

FIG. 2. Schematic view of the water cooled sample holder.

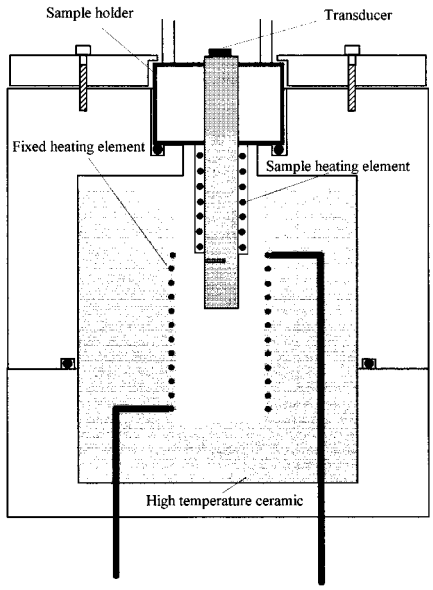

FIG. 3. Schematic representation of furnace, showing the fixed heating element and the inserted sample.

tional buffer technique. Five elastic constants were measured in three different samples of which only three are independent. The results agree within a relative error of 0.3% in the entire temperature range. This value is to be compared with a relative error of 2% obtained by previous authors using the conventional buffer technique.[7,8]

As an example of the quality of the results attainable with this method, we show in Fig. 4 the elastic constant C_L (wave vector along [110], longitudinal polarization) as measured near the Curie temperature of one of the samples (Fe 4 at. % Si) with and without an applied magnetic field. The expected critical behavior of this elastic constant[9–11] and its inhibition by a magnetic field is clearly seen.

We believe that, with this experimental technique, an increase of almost an order of magnitude in accuracy can be obtained, relative to the conventional buffer technique, for high temperature sound velocity measurements. Another important feature of our method is the reliability of the experimental setup, in opposition to the frequent breaking of the high temperature sample to buffer bond. Also, and although the sample geometry was not optimized for attenuation measurements (differences in the diffraction conditions can affect the wave front distribution between the two reflecting surfaces) with our method it was easy to conclude that previous authors' reports of strong sound attenuation at high temperatures in iron–silicon samples[7,8] is not real, but only a consequence of the measurement technique used by those authors.

The main disadvantage of the described technique is the need for relatively large samples. How large they must be depends on their heat conductivity and is easily estimated. The demonstrated possibility of using this method even in the case of a material such as iron is an indication that it can be applied to most materials.

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