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Measurement of Ultrasonic Velocity and Attenuation at Elevated Temperatures

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Abstract

Ultrasonic non-destructive testing is a sensitive tool not only for defect detection and evaluation in plant components and structures, but also to characterise microstructural features and for evaluation of mechanical properties. Even though, it provides high sensitivity and reliability for such measurements and very versatile to use at ambient temperatures, the technique fails when it is extended to high temperature studies beyond 600 K. This is due to the loss of piezoelectric properties of the transducer materials at higher temperatures. In order to overcome the above problem, an indigenous experimental set up has been designed and fabricated to carry out the ultrasonic velocities and attenuation measurements in solid materials over wide range of temperatures from room temperature to 500 K. In the present experimental set up, the through transmission technique has been employed for the ultrasonic measurements, using recrystallised alumina rods as waveguides. In the present experimental set up one can obtain the required temperature either in a dynamic mode or in a static mode as required, employing a microprocessor based temperature controller. The accuracy of the temperature in the sample region is ± 1 K. The experimental set up has been calibrated for the velocity measurements by measuring the precise transit time (t_1) using only the assembly of waveguides without any sample and the transit time (t_2) by inserting the sample between the waveguides, whose velocity is to be determined, corresponding to the same temperature condition. Thus, the transit time of the sample has been measured by taking the difference in the transit times ($\Delta t = t_2 - t_1$) at different temperatures. The present paper deals with the details of measurement procedure adopted for ultrasonic velocity and attenuation in solid samples as a function of temperature. The paper also gives the details of the calibration and validation of the experimental set up carried out through benchmark measurements made in materials with known phase transformations that occur at specific temperatures and are expected to influence ultrasonic parameters.

Keywords: High temperature experimental set up, Through transmission technique, Ultrasonic velocity, Ultrasonic attenuation.

1. Introduction

In materials characterisation, ultrasonic NDT technique is a versatile tool and plays a dominant role than the conventional techniques [1,2]. The ultrasonic velocity and attenuation based hence, the evaluation of mechanical properties will be more useful in exploring the informations such as physico-chemical properties, structural changes, phase changes, charge ordering, Curie temperature, metal – insulator transition, microstructural changes, grain size measurements etc. [3,4]. This is possibly due to the well developed basic understanding of ultrasonic wave propagation and scattering mechanisms in solids, and also free accessibility of the improved transducer technologies and instrumentation. The measured ultrasonic velocities directly linked to the elastic constants of the materials [5]. The ultrasonic NDT technique has gained momentum in view of the accuracy, reproduction etc., when compared with other techniques. It is mainly due to the availably of the wide frequency range and also the interaction of ultrasonic waves with macroscopic, microscopic and submicroscopic microstructural features under different scattering and absorption mechanisms.

Ultrasonic velocity and attenuation measurements in solids are generally made employing the pulse echo method [6]. In the pulse echo method, the ultrasonic transducers, which generate ultrasonic waves, are directly connected with the solid samples using a suitable couplant. Even though, it has many salient futures such as sensitivity, reliability and versatility to use at ambient temperatures, the technique fails when it is extended to high temperature studies. This is mainly due to the loss of piezoelectric properties of the transducer materials at higher temperatures.

In order to overcome the above problem, an indigenous experimental set up has been designed and fabricated to carry out ultrasonic velocities and attenuation measurements in solid materials over wide range of temperatures [7]. For the characterisation of materials at elevated temperatures, one cannot use either the pulse echo or through transmission technique without wave guides. This is mainly due to the raise in temperature in the sample region. In the case of high temperature measurements, the ultrasonic transducers will be affected by the thermal radiation. To avoid this damage, a special care was taken to rise the temperature from room temperature to high temperature without any damage to the transducers due to the temperature gradients from the furnace.

2. Experimental Set up - Design and Fabrication

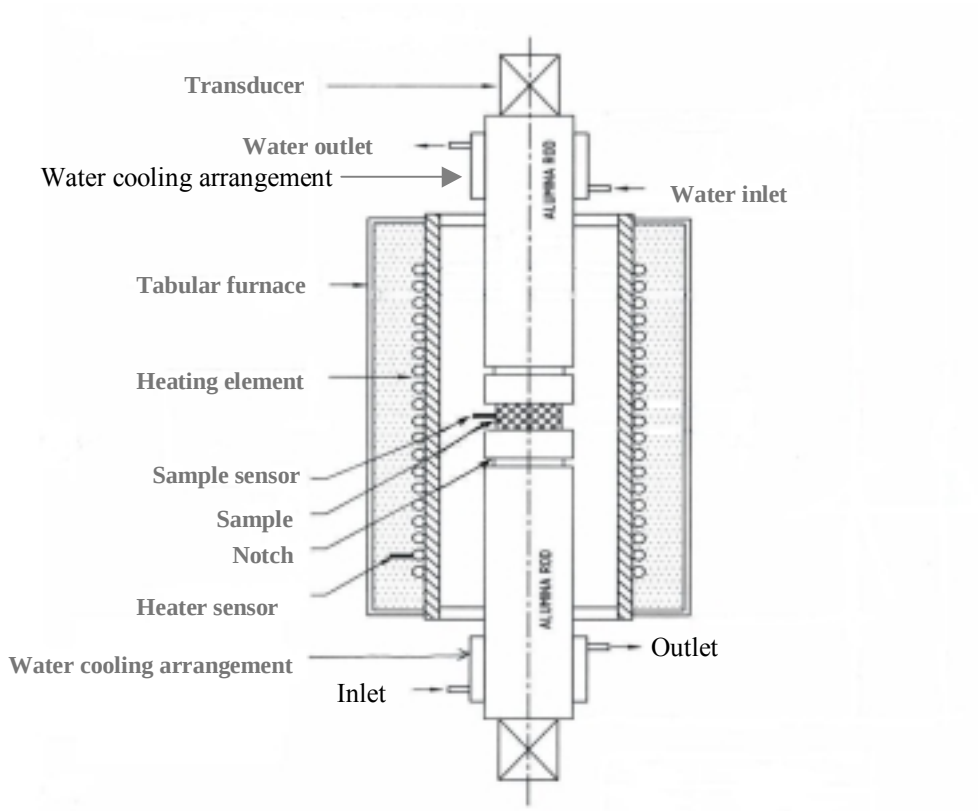


Fig.1. Cross sectional view and mechanical arrangement of the specimen, waveguide and transducer in the high temperature experimental set up using through transmission technique

The cross sectional view of the high temperature experimental set up with sample, waveguides and transducers arrangement for ultrasonic velocities and attenuation measurements employing through transmission technique is shown in Fig.1.

The furnace is cylindrical split type with outer shell made out of stainless steel insulated with superior grade Zirconia insulation fiber blankets to eliminate the thermal loss. The main chamber of the furnace was heated by a superior grade Kanthal APM heating element capable of handling operating temperature up to 1100 K on a continuous basis. The power to the heating element was fed through a thyristor power control device employing a programmable temperature controller (M/s. Eurotherm). A temperature sensor which is kept very close to the sample is used to monitor the temperature of the sample. Recrystalised alumina rod (AD 94 grade) with mirror finished ends was used as a waveguide, which has less temperature gradient. The circumferential notch near the sample region in both wave guides was provided to minimise the scattering effect of ultrasonic waves. The water cooling arrangements employed at outer ends of the wave guides, safe as shown in Fig. 1 guards the transducer from thermal radiations. The furnace has the facilities for holding ultrasonic transducers at both ends of waveguides. An adjustable spring loaded arrangement is used for applying uniform pressure at the top surface of the transducers (Fig.1).

The transducers are mounted on the probe holder and then, the parallelism is achieved. The probe holder can be moved up or down by adjusting the screw arrangements. This helps to bring the transducers, waveguides and sample all in a line and also provides a good contact and uniform pressure between different media such as waveguides, sample and transducers.

3. Instrumentation

A high power ultrasonic process control system (Model: FUI1050 of M/s. Fallon Ultrasonics Inc. Ltd., Canada), a digital storage oscilloscope (DSO) (Model: 54600B of M/s. Hewlett Packard, USA) and a Pentium-III computer were employed for recording ultrasonic (rf) signals. The transducers (M/s. Panametrics, USA) operating at a fundamental frequency of 5 MHz were used for the generation and reception of ultrasonic waves (both longitudinal and shear waves).

Fig.2. Experimental arrangement of the high temperature furnace for the ultrasonic velocities and attenuation measurements

The photograph of the high temperature experimental set up is shown in Fig. 2.
The block diagram of the experimental set up used for ultrasonic measurements is shown in Fig.3. The transducers were connected to the ultrasonic pulser receiver. In the present experimental set up, one can obtain the required temperature either by dynamic mode or static mode depending on the requirements, employing Eurotherm temperature controller.

The accuracy of the temperature in the sample region is ± 1 K. All the systems are connected through the IEEE interface cable via the computer. The ultrasonic velocities and attenuation measurements have been made from the room temperature (300 K) to 500 K with a slow rate of heating (1 $Kmin^{-1}$). The error in the measurement of temperature is ± 0.1 K.

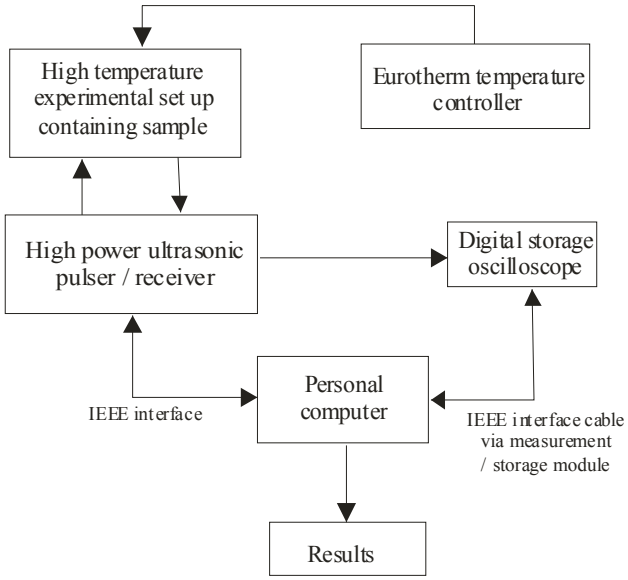


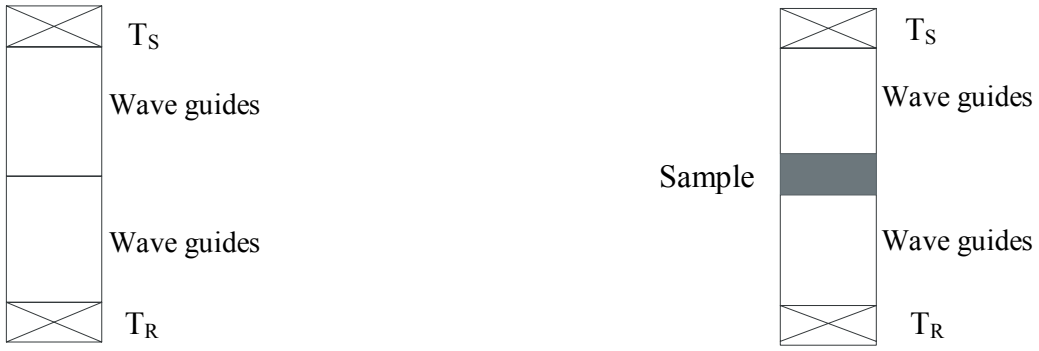
Fig. 3 Block diagram of the experimental set up for the ultrasonic velocities and attenuation measurements.

4. Ultrasonic velocity and attenuation measurements

In order to achieve proper impedance matching and a good contact between the sample, waveguides and transducers for the propagation of ultrasonic waves into the sample, the opposite surfaces of both the sample and the waveguides were highly polished. The plane parallelism between the two opposite faces of the sample was checked using a surface plate and a dial gauge.

Through transmission technique has been employed for the ultrasonic velocities and attenuation measurements. In the absence of the sample, ultrasonic waves transmitted by the transducer T_S travel along the buffer rods and the waves were received by the receiving transducer T_R as shown in Fig.4a, even though some of them get reflected. Thus, the transit time (t_1) through the buffer rods has been determined. Then, the sample whose velocity is to be determined is sandwiched between the two buffer rods without disturbing the buffer rods and transducers (Fig.4b). The transducer is coupled with the sample using a suitable couplant in order to get a steady back wall echo train which provides good impedance matching between transducer, sample and buffer rods. The transmitted pulses get shifted by a small distance. During the above process, some of the waves gets reflected back into the first buffer rod and a part is transmitted into the sample. Further, reflection takes place at this stage and additional attenuation of the signal that is sent into the second buffer rod. The ultrasonic waves transmitted into the second buffer rod are received by the transducer (T_R), where the received signal is stored through benchmark measurements using Bench Link Scope ultrasonic testing software available in the computer. Therefore, the corresponding transit

time (t_2) has been determined. The difference between the transit times t_1 and t_2 gives the transit time $\Delta t = (t_2-t_1)$ of the ultrasonic waves in the sample.



(a) Buffer rods and transducers (b) Sample, buffer rods and transducers

Fig. 4 Through transmission technique - ultrasonic velocity & attenuation measurements

In the whole process, the scattering of ultrasonic wave is a main factor to be considered. However, we are taking only the relative measurements and hence, the scattering was not considered as a major factor. The ultrasonic velocity was measured using the following relation:

$$U = \frac{d}{\Delta t} \tag{1}$$

Knowing the sample thickness (d) in micron resolution and transit time (Δt) in nanosecond resolution, the overall accuracy obtained in the measurement of velocity is ± 5 ms^{-1}. The attenuation of the ultrasonic waves in the sample was measured using the relation [7,8]:

$$\alpha(f) = \frac{1}{d}\left(\ln T + \ln\left(\frac{A_w(f)}{A_s(f)} \right) \right) \tag{2}$$

where $A_w(f)$ refers to the amplitude of the received signal with the waveguides only and $A_s(f)$ refers to the amplitude of the received signal when the sample is inserted between the wave guides. In the present study, the first and second back wall echoes were used.

$$T = \frac{4Z_w Z_s}{(Z_w + Z_s)^2} \tag{3}$$

where T is the combined transmission coefficient at the sample and waveguide interface, Z_w and Z_s are the acoustic impedance of the waveguide and the sample respectively.

In order to validate the present set up, pulse echo overlap / cross correlation technique has also been used for the ultrasonic velocity and attenuation measurements. The precise transit time (t) was obtained from the time domain trace of the back wall echo train obtained in the screen and hence, velocity of ultrasonic waves in the sample can be measured by using pulse echo overlap and cross correlation (CC) technique. The ultrasonic velocity in sample is determined using the relation,

$$U = \frac{2d}{t} \tag{4}$$

where t is the time taken for the to and fro distance traveled by the ultrasonic waves the sample and d the thickness of the sample.

The attenuation coefficient can be determined by measuring the amplitudes of the echoes from the time domain trace using the following equation,

$$\alpha = \left(\frac{-20}{2(m-n)d} \right) \log \left(\frac{I_m}{I_n} \right) \quad (5)$$

where I_m and I_n are, respectively, the maximum amplitude (voltage) of the *m*th and *n*th pulse echoes. In the present study, the first and second back wall echoes were used. The percentage error in the attenuation measurement is ± 2 %. The couplant correction for velocity and attenuation was carried out by the standard procedure.

5. Calibration of the experimental set up

The present experimental set up has been calibrated without the sample between the buffer rods, from room temperature (300 K) to 500 K. The transit time t_1 has noted for both increasing and decreasing order of temperature for each 10 K temperature difference. In the above process recrystalised alumina rod was chosen as a waveguide after careful study on different materials. The Eurotherm temperature controller was employed to control the temperature to the accuracy of ± 1 K. A linear increase in the transit time (t_1) was observed with increase in the temperature as shown in Fig. 5. After inserting the sample between the buffer rods, the transit time t_2 would be measured corresponding to the same temperature conditions. The difference in the transit times (t_2-t_1) at a particular temperature is known as the transit time of the sample at the corresponding temperature.

Fig. 5 Calibration curve – Transit time measurement

The above experimental set up has been calibrated by measuring ultrasonic velocities on standard solid samples. The measured velocities in different solid samples at room temperature are compared with conventional techniques. The observed results show that the measured velocity employing through transmission technique for standard samples agrees well with the values obtained from other techniques. The obtained results employing the through transmission (TT) and PEO techniques are given in Table 1.

Table 1. Ultrasonic velocity on the standard samples at 5 MHz at room temperature

Sample	Thickness $x10^{-3}$ m	Ultrasonic velocity (ms^{-1})	
		TT	PEO
Brass	10.582	4409	4389
Quartz	10.019	5733	5721

PEO- Pulse echo overlap technique; TT- Through transmission technique

The temperature dependence of longitudinal wave velocity (U_L) of Al-Li (8090) alloy with temper T8771 is represented graphically in Fig. 6. The velocity measurements reveal the formation, growth and dissolution of δ' (Al_3Li) precipitates in the temperature range 450 to 480 K [10]. The observed deviation of increase in velocity from 470 K from the general decreasing trend with raising temperature is attributed to the formation of δ'. As the temperature increases beyond 470 K, the δ' precipitates start dissolving leading to the large reduction in the velocity from the peak value (6580 m/sec). Beyond 480 K, the general trend of decrease in velocity with increase in the temperature is restored.

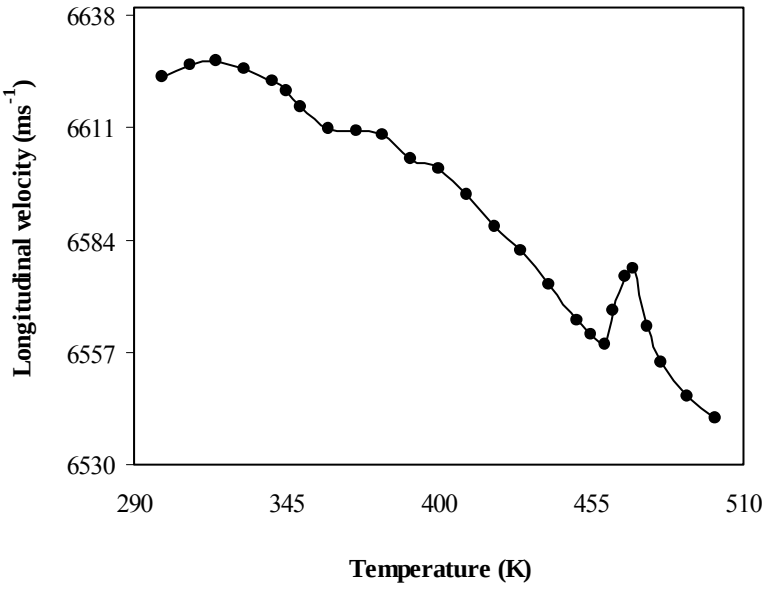


Fig. 6 Longitudinal velocity as a function of temperature in Al-Li alloy

6. Conclusions

The followings are the conclusions drawn from the present investigation:

1. An indigenous experimental set up has been deigned and fabricated to carry out the ultrasonic velocities and attenuation at higher temperatures.
2. The calibration studies carried out in 8090 Al alloy reveal the nucleation, growth and dissolution of δ' in the temperature range 450 – 480K.

3. The observed results reveal high accuracy and reproduction for the measurements of velocity and attenuation in the solid samples as a function of temperature.
4. The present experimental set up provides a solution for the characterisation of the materials as a function of temperature, which fulfills the demands for the materials characterisation at elevated temperatures.

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