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# Experimental Considerations on Water-Couplant Temperature for Accurate Velocity Measurements by the LFB Ultrasonic Material Characterization System

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**Abstract** - The line-focus-beam ultrasonic material characterization (LFB-UMC) system can measure the phase velocity of leaky surface acoustic waves (LSAWs) excited on the water-specimen boundary. The temperature distribution in the water-couplant, however, is the source of some measurement errors in the LSAW velocity. A method for obtaining the proper water-couplant temperature and longitudinal velocity has been developed using the measured temperature distribution and the small interference signals between the  $V(z)$  curve signals and the carrier leakage signals arising from the RF tone burst pulse generation circuit used in the system. A procedure to obtain the LSAW velocity accurately is established and the system achieves relative accuracy of LSAW velocity better than  $\pm 0.002\%$  at a single chosen point and better than  $\pm 0.003\%$  in two-dimensional measurements.

## I. INTRODUCTION

Material characterization with the line-focus-beam ultrasonic material characterization (LFB-UMC) system is made by measuring the phase velocity of leaky surface acoustic waves (LSAWs) excited on the water-specimen boundary [1]. In this technology, the longitudinal wave velocity in water, determined as a function of temperature, is used as the reference. Therefore, the measurement errors of the water temperature significantly affect the measurement accuracy of the LSAW velocity. The system has achieved the relative accuracy in LSAW velocity measurements to better than  $\pm 0.002\%$  at a single point of a specimen and  $\pm 0.005\%$  over a scanning area of  $75 \text{ mm} \times 75 \text{ mm}$  [2]. It is desired, however, that the accuracy of the system be improved further to enable highly accurate detection of slight changes of physical and chemical properties in and among material substrates.

In this paper, the temperature distribution in the water-couplant, which is the source of the measurement error in LSAW velocity, is discussed first. Next the measurement method developed for obtaining of the water-couplant temperature and the longitudinal wave velocity in water using the  $V(z)$  curve is presented. Then, according to these considerations, the measurement procedure, established

to obtain the LSAW velocity accurately at a single point and in two-dimensional measurements, is discussed.

## II. TEMPERATURE DISTRIBUTION IN WATER-COUPPLANT

The LFB-UMC system can measure the propagation characteristics of LSAWs by analyzing the  $V(z)$  curves, which are the transducer outputs recorded by changing the relative distance  $z$  between the LFB ultrasonic device and the specimen [1]. The LSAW velocity,  $V_{\text{LSAW}}$ , is obtained from the oscillation interval  $\Delta z$  of the  $V(z)$  curve using the relation

$$V_{\text{LSAW}} = \frac{V_w}{\sqrt{1 - \left(1 - \frac{V_w}{2f\Delta z}\right)^2}}, \quad (1)$$

where  $f$  is the ultrasonic frequency and  $V_w$  is the longitudinal wave velocity in water used as the couplant.  $V_w$  is the value at the temperature of measurement of the  $V(z)$  curve, and is obtained from the literature [3]. Therefore, the measurement errors of the water temperature directly affect the measurement accuracy of the LSAW velocity. The measurement accuracy of water temperature is, for example, required to be better than  $\pm 0.02^\circ\text{C}$  in order to achieve the measurement accuracy of  $\pm 0.002\%$  in the LSAW velocity from a numerical calculation using Eq. (1). A thermocouple is used to measure the temperature of the water couplant in the system. However, it is impossible, in principle, to insert the thermocouple directly into the region of propagation of the ultrasonic waves. In general, a temperature distribution exists in the couplant due to the heat of vaporization of water and due to the convective flow of air around the specimen. In order to measure the temperature distribution in the couplant, one thermocouple is positioned just under the LFB lens surface, denoted by the region A, where the ultrasonic waves propagate, and a second thermocouple is positioned near the lens surface, denoted by the region B, where the water temperature is usually measured during  $V(z)$  curve measurements, as shown in Fig. 1. The temperatures  $T_A$  at the region A and  $T_B$  at

the region B are measured simultaneously under the condition that the temperature distribution in the couplant is stable, and the temperature difference  $\Delta T (=T_A - T_B)$  between two regions is obtained.

An LFB ultrasonic device designed for 225-MHz operation, which is usually employed for material characterization by the LFB-UMC system, was used. The distance between the two thermocouples was approximately 3 mm and the distance between the lens surface and the specimen was the focal length of 1.15 mm, at the region A. The thermocouples were calibrated in absolute accuracy within  $\pm 0.01^\circ\text{C}$ , and in relative accuracy within  $\pm 0.005^\circ\text{C}$ . The  $xy$ -stage used in the system was set at the origin ( $x=y=0$ ) of the system coordinate. The entire mechanical system, including the specimen, is installed into a temperature-controlled chamber in which the relative humidity was within  $40 \pm 1\%$  during the measurements.

First, the relationship between the temperature distribution and the quantity of the couplant was studied using the GGG substrate as the specimen. The measured temperatures  $T_A$  and  $T_B$  are given in Fig. 2. Each of the plotted values is the averaged of several tens of measurements exhibiting a deviation of  $\pm 0.02^\circ\text{C}$ .  $T_B$  is less than  $T_A$ , due to the vaporization heat of water. As the quantity of the couplant increased, both temperatures  $T_A$  and  $T_B$  monotonically decreased. The obtained values of  $\Delta T$  are also given in Fig. 2, where it is seen that  $\Delta T$  became larger as the quantity of couplant decreased. When the quantity of the couplant was in the range 0.6 to 1.2 cc for the present system, the values of  $\Delta T$  were within  $0.057 \pm 0.003^\circ\text{C}$ , corresponding to the measurement error of  $-0.0057 \pm 0.0003\%$  in LSAW velocity from the previous numerical calculation using Eq. (1). Next, the relationship between the temperature difference and the thermal conductivity of materials was investigated using the couplant quantity of 0.7 cc. We examined many specimens such as Teflon, Pyrex glass, single crystal plates of  $\text{LiTaO}_3$ ,  $\text{LiNbO}_3$ ,  $\alpha$ -quartz, GGG, and Si, and metal plates of Al and Au, with the different thermal conductivities and the different thicknesses. The results are shown in Fig. 3, where the thermal conductivity  $\kappa/h$  is used by considering the thicknesses of the specimens. The temperature was greater for the specimen with larger thermal conductivity, as shown in Fig. 3(a).  $\Delta T$  was the greatest for the smallest thermal conductivity, as shown in Fig. 3(b). It is found that the values of  $\Delta T$  were within  $0.063 \pm 0.008^\circ\text{C}$ , corresponding to the measurement error of  $-0.0063 \pm 0.0008\%$  in LSAW velocity, for the specimens such as single crystals, glasses, and metallic materials, usually characterized by the LFB-UMC system, having the thermal conductivity more than  $2 \times 10^2 \text{ W}/(\text{m}^2 \cdot \text{K})$ .

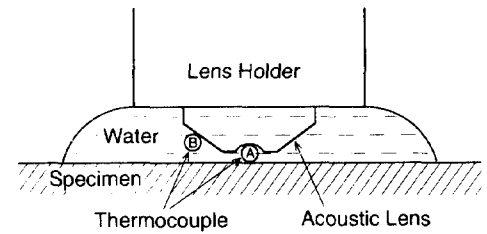


Fig. 1 Schematic view of measuring temperature distribution in water-couplant using two thermocouples.

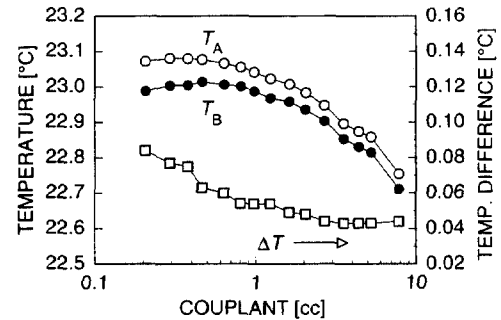


Fig. 2 Dependences of temperature  $T_A$  and  $T_B$  and temperature difference  $\Delta T$  in water-couplant on quantity of the couplant.

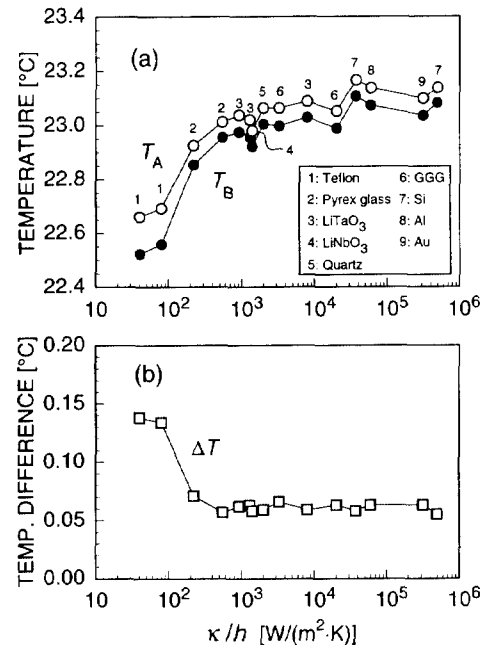


Fig. 3 Dependences of temperature  $T_A$  and  $T_B$  (a) and temperature difference  $\Delta T$  (b) in water-couplant on thermal conductivities  $\kappa$  of the specimens with couplant quantity of 0.7 cc. Thermal conductivity is divided by the specimen thickness  $h$ .

### III. MEASUREMENT PROCEDURE

#### A. Water-Couplant Temperature

1. **Single Point Measurement:** The temperature  $T_A$  in the region A, where the ultrasonic waves propagate, during the  $V(z)$  curve measurement is determined accurately using  $T_B$  and  $\Delta T$  by the relation

$$T_A = T_B + \Delta T, \quad (2)$$

where  $T_B$  is measured during the period of  $V(z)$  curve measurements using the thermocouple, and  $\Delta T$  is obtained in advance for each specimen under the same conditions as  $V(z)$  curve measurements at a single point on the specimen surface.

2. **Two-Dimensional Measurement:** For two-dimensional measurements, the values of  $\Delta T$  could, in general, be different at different positions on the specimen surface. Therefore, a method of determining the water temperature and the longitudinal wave velocity in water has been developed using  $V(z)$  curves to obtain the longitudinal wavenumber in water,  $k_w$ . A carrier leakage signal, denoted by  $V_c$ , arises from insufficient on/off ratio in the RF tone burst pulse generation circuit used in the system. The  $V_c$  signal does not depend upon the relative distance  $z$ , and is received and detected together with the  $V(z)$  curve signal. Figure 4(a) shows the typical  $V(z)$  curve measured for a (111) GGG substrate at 225 MHz with LSAW propagating along the  $[\bar{1}\bar{1}2]$  direction. The on/off ratio of the RF pulse generation circuit was 75 dB. The small interference signals between the  $V(z)$  curve signal and the  $V_c$  signal exist over the entire  $V(z)$  curve. The interference signals are derived from the  $V(z)$  curve and the spectral distribution of the interference signals was obtained by the FFT analysis as shown in Fig. 4(b). The two peaks of the spectrum corresponding to the wavenumbers  $k_0$  ( $=2k_w$ ) and  $k_1$  ( $=2k_w \cos \theta_{LSAW}$ ), associated with the phasors  $V_0$  and  $V_1$  determining the  $V(z)$  curves [4], were obtained, where  $\theta_{LSAW} = \sin^{-1}(k_{LSAW}/k_w)$ , and  $k_{LSAW}$  and  $k_w$  are the wavenumbers of the LSAWs and of the longitudinal waves in water, respectively. Therefore,  $k_w$  was successfully obtained by  $k_w = k_0/2$ . The system can also measure the complex  $V(z)$  curves from which  $k_w$  is obtained.

The wavenumbers, however, obtained from the  $V(z)$  curves were slightly different from the true value of  $k_w$  because of the characteristics of the ultrasonic device employed. Therefore, a procedure to measure the water temperature,  $T_w(x, y)$ , and the longitudinal wave velocity in water,  $V_w(x, y)$ , at the position  $(x, y)$  on the specimen

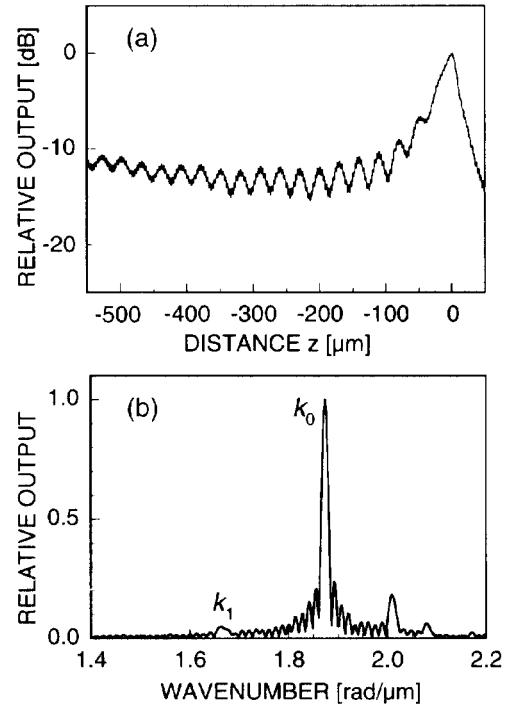


Fig. 4  $V(z)$  curve measured for (111)- $[\bar{1}\bar{1}2]$  GGG specimen at 225 MHz and spectral distribution of the interference signals on the  $V(z)$  curve.

surface in two-dimensional measurements has been developed using the wavenumber, expressed as  $k_w'$ , obtained from the  $V(z)$  curve.  $T_w(x, y)$  is given using  $T_{A0}$  as a reference value obtained at a single point measurement by

$$T_w(x, y) = T_{A0} + \Delta T_w(x, y). \quad (3)$$

Since  $V_w'$  is equal to  $2\pi f/k_w'$ ,  $\Delta T_w(x, y)$  is given by

$$\Delta T(x, y) = \frac{V_w'(x, y) - V_{w0}'}{\frac{dV_w'}{dT}} = \frac{2\pi f \{ k_{w0}' - k_w'(x, y) \}}{k_{w0}' \cdot k_w'(x, y) \cdot \frac{dV_w'}{dT}}, \quad (4)$$

where  $k_{w0}'$  at  $T_{A0}$  is obtained from the  $V(z)$  curves measured at a single point on the specimen surface under stable temperature conditions, and  $k_w'(x, y)$  is obtained from the  $V(z)$  curve measured at the position  $(x, y)$ . The changing ratio of  $V_w'$  for temperature is obtained from the literature [3], as it is nearly equal to that of  $V_w$ . Then,  $T_w(x, y)$  is obtained by substituting (4) for (3). Similarly,  $V_w(x, y)$  is obtained using  $V_{w0}$  at  $T_{A0}$  as a reference value by

$$V_w(x, y) = V_{w0} + \Delta V_w(x, y), \quad (5)$$

where  $\Delta V_w(x, y)$  is represented by

$$\Delta V_w(x, y) = \frac{dV_w}{dT} \cdot \Delta T_w(x, y) \quad (6)$$

Therefore, if  $\Delta T_w(x, y)$  is determined,  $V_w(x, y)$  is obtained by substituting (6) for (5).  $\Delta V_w(x, y)$  is also given by substituting (4) for (6) as

$$\Delta V_w(x, y) = \frac{2\pi f \{k'_{w0} - k'_w(x, y)\}}{k'_{w0} k'_w(x, y)} \quad (7)$$

## B. LSAW Velocity

**1. Single Point Measurement:** A flowchart of the measurement procedure for obtaining the LSAW velocity at a single point is shown in Fig. 5. First, the  $V(z)$  curves are obtained for a standard specimen used in system calibration [2] and for the specimen examined (step S1).  $T_B$  should be corrected using the value of  $\Delta T$  measured in advance (step S4) if the values of  $\Delta T$  are different between them (step S2). However, it need not be corrected (step S3) if the values of  $\Delta T$  are the same. Next, the  $V(z)$  curve analysis and a calibration method are applied, and  $V_{LSAW}$  is obtained for the specimen examined (steps S5, S6). However,  $V_{LSAW}$  is obtained within the estimated error using  $T_B$ , if the values of  $\Delta T$  for the examined and standard specimen are slightly different.

**2. Two-Dimensional Measurement:** A flowchart of the procedure for two-dimensional measurements is shown in Fig. 6.  $T_B$  measured for a standard specimen should be corrected using  $\Delta T$  (steps S1-S3).  $T_w(x, y)$  and  $V_w(x, y)$  in two-dimensional measurements for the specimen examined are determined by the proposed method using the  $V(z)$  curves (steps S4-S7). Then,  $V_{LSAW}$  is obtained by analyzing  $V(z)$  curves and applying a calibration method (steps S8-S10). If  $\Delta T_w(x, y)$  in Eq. (3) is small,  $V_{LSAW}$  is obtained within the estimated error using  $T_{A0}$  as  $T_w(x, y)$ .

## IV. MEASUREMENTS AND RESULTS

The measurement accuracy of LSAW velocity in the two-dimensional measurements was investigated with the measurement procedure shown in Fig. 6, using a specimen of 36° rotated Y-cut X-propagating LiTaO<sub>3</sub> substrate for surface acoustic wave (SAW) devices. An ultrasonic frequency of 225 MHz was employed and a (111) GGG standard specimen was used for the system calibration [3].

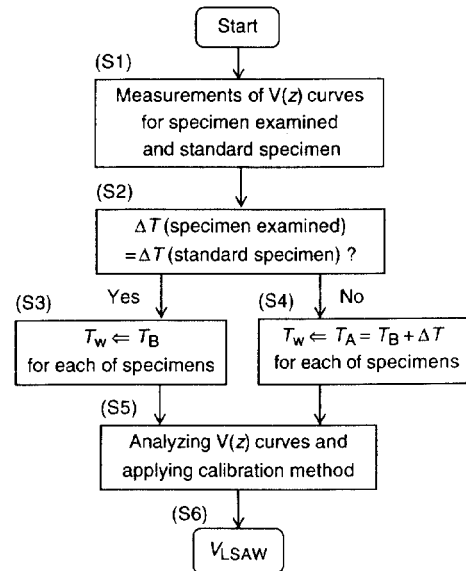


Fig. 5 Measurement procedure of LSAW velocities at a single point.

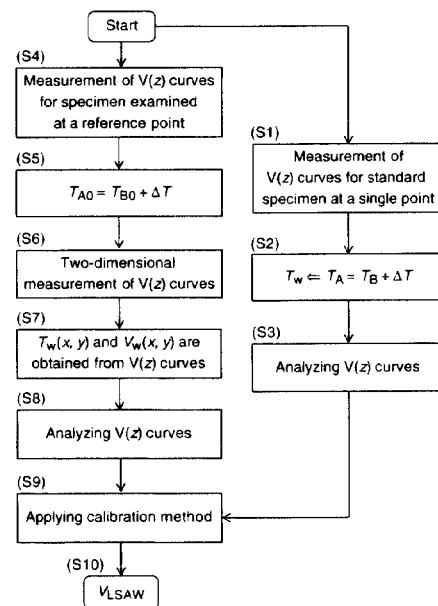


Fig. 6 Measurement procedure of LSAW velocities in two-dimensional measurements.

Both specimens have sufficient thickness to avoid the influence of the waves, reflected from the back surface of the specimen, on the LSAW velocity measurements [5].

First,  $V(z)$  curves were measured repeatedly fifty times at a reference single point on the specimen surface under stable temperature conditions, in order to obtain a reference temperature  $T_{A0}$ , where the temperature stability was confirmed using  $k_w'$  obtained from the  $V(z)$  curves.  $T_{A0}$  was obtained to be 22.957°C and the averaged value of  $V_{LSAW}$  was 3125.15 m/s with  $\pm 2\sigma$  of  $\pm 0.062$  m/s ( $\pm 0.002\%$ ) by the proposed procedure shown in Fig. 5 using each of  $\Delta T$ , where  $\sigma$  is the standard deviation. Next, the  $V(z)$  curves were measured in 2-mm steps over a distance of 60 mm on the specimen surface along the  $x$  axis of the system, where the  $y$ -stage was set at the origin ( $y=0$ ). The measured temperatures  $T_B(x)$  at the region B in Fig. 1, using a thermocouple during  $V(z)$  curve measurements, are shown in Fig. 7(b).  $T_B(x)$  rapidly decreased at the position of  $x < -26$  mm, near the edge of the specimen, since the thermocouple was set at the left side of the lens surface in water, and the maximum deviation was 0.063°C. The obtained  $V_{LSAW}$  by the conventional procedure using  $V_w$  obtained from the literature [3] using the values of  $T_B(x)$  are also shown in Fig. 7(a).  $V_{LSAW}$  decreased at the position of  $x < -26$  mm, as did  $T_B(x)$ , and the maximum deviation was 0.26 m/s.  $k_w'(x)$  and  $T_w(x)$  obtained from the  $V(z)$  curves are shown in Fig. 7(b). The average value of 22.947°C and the maximum deviation of 0.044°C were obtained for  $T_w(x)$ . It is found that the deviation of  $T_w(x)$  was less than that of  $T_B(x)$ , which shows that the temperature at the region just under the lens surface, where the ultrasonic waves propagate, was more stable than at the region where the temperature was measured by the thermocouple. The calibrated LSAW velocities obtained using  $k_w'(x)$  are shown in Fig. 7(a). The true values of LSAW velocity of the specimen were successfully determined with the maximum deviation of 0.18m/s and with  $\pm 2\sigma$  of  $\pm 0.0029\%$ , in which the slight variation of the chemical composition ratio in the specimen is included [6].

## V. SUMMARY

In this paper, the temperature distribution in the water-couplant was investigated for the present LFB-UMC system, and it was found that the temperature distribution depended upon the quantity of couplant and upon the thermal conductivities of the specimens. A measurement method of water temperature and longitudinal wave velocity in water was developed, and a measurement procedure has been established to obtain the accurate LSAW velocity at a single point and in two-dimensional measurements. The system achieved the accuracy of LSAW velocity better than  $\pm 0.002\%$  at a single chosen point and better than  $\pm 0.003\%$  in two-dimensional measurements.

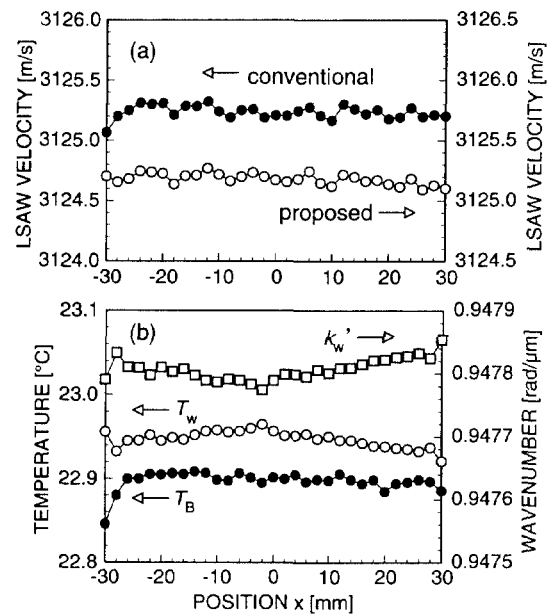


Fig. 7 Correction of LSAW velocity variations measured for 36°YX-LiTaO<sub>3</sub> substrate. (a): LSAW velocities; (b): water-couplant temperatures  $T_w$  and  $T_B$  and longitudinal wavenumber  $k_w'$ .

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