Measurement of Elastic Constants of Ceramics by a Scanning Laser Acoustic Microscope*

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A scanning laser acoustic microscope, SLAM, is applied to measure velocities of sound and to evaluate elastic constants of ceramics. The method of measuring sound velocity is based on spectral interferometry utilizing the interference fringes for a sample and for a reference material. In this study, a suitable sample holder is made to excite the ultrasonic waves to the desired mode, i.e., the longitudinal or the transverse mode, within the sample. Young's modulus, shear modulus, and Poisson's ratio of partially stabilized zirconia ceramics are evaluated from the measured velocities of sound. Then tensile tests are performed on the same sample and the elastic constants are measured. The elastic constants evaluated by SLAM are in agreement with those obtained by the tensile tests within a deviation of about 10 %. The method described here is useful for small specimens of the ceramic materials.

Key Words: Elasticity, Scanning Laser Acoustic Microscope, Velocity of Sound, Elastic Constant, Partially Stabilized Zirconia Ceramics

1. Introduction

Young's modulus, the shear modulus, and Poisson's ratio of a material are fundamental and important parameters showing elastic behavior of the material. Mechanical tests and dynamic tests are generally performed to obtain those elastic constants. For structural Ceramics, the bending test and dynamic techniques such as a resonance method and an ultrasonic pulse echo method are applied to measure the elastic modulus⁽¹⁾. Because of the principle of the measurement and the nature of ceramics, however, a relatively long specimen, e. g., 40 mm in length, or a relatively thick specimen, e.g., 10 mm in thickness, is required to measure Young's modulus by the bending test or the ultrasonic pulse echo method, respectively. A small and thin plate specimen cannot be measured by these methods.

A scanning laser acoustic microscope⁽²⁾, SLAM, and a mechanically scanned acoustic microscope⁽³⁾, SAM,

are drawing much attention not only as a nondestructive inspection apparatus but also as an effective apparatus for the analysis of the elastic property of materials^{(4)~(10)}. In this study, SLAM is applied to evaluate the elastic constants of zirconia ceramics by measuring velocities of sound.

2. Principle of SLAM

Figure 1 and Fig. 2 show a schematic diagram of SLAM used in this study and an illustration of the

Fig. 1 Schematic diagram of SLAM

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laser detection of the acoustic energy to produce the acoustic micrographs, respectively. The details of SLAM are described in ref. 2. A sample is placed on a stage, where it is insonified with continuous plane ultrasonic waves of frequency of about 100 MHz through a liquid coupler of small incident angle. The refracted waves transmit through the sample and finally produce microdistortions called dynamic ripple on the upper surface opposite the incident sound source. The distortion pattern is transmitted via the coupler to the reflective coating of a plastic coverslip placed on the sample. This distortion pattern is detected by a scanning focused laser beam which illuminates the coverslip. The laser beam is reflected to a photodetector with frequency modulation by Doppler shift, and is converted to an electrical signal. By heterodyning the received electrical signal with the original unshifted signal, an acoustic interference image is obtained on CRT monitor⁽¹¹⁾.

3. Method of Elastic Constant Measurement

The front view of Fig. 3 shows directions of wave penetration and phase fronts in a sample of thickness T and in an equivalent thickness of a reference medium when a sample adjoins a reference medium. The reference medium is often liquid and also serves as a coupler. The ultrasonic waves are refracted at the sample-coupler interface because of the difference in sound velocities between the sample and the reference medium. The refracted waves are represented by solid lines while the waves passing through the reference medium are not refracted, as

Fig. 2 Illustration of laser detection of the acoustic energy

shown by chain lines. Acoustic field is made visible at the surfaces of the sample and the reference medium. The acoustic interference images with fringes are obtained on the monitor. These images show lateral shifts of the fringes at the boundary of the sample and the reference medium as shown in the top view of Fig. 3. The ratio of the lateral shift to the interval of the fringes, $\Delta L/L$, is defined as the fringe shift, N , and is presented by Eq. (1).

$$N = \frac{\Delta L}{L} = \frac{fT \sin \theta_1}{c_1} \left(\frac{1}{\tan \theta_1} - \frac{1}{\tan \theta_2} \right) \quad (1)$$

where c_1 is sound velocity in the reference medium, θ_1 is the refraction angle of ultrasonic waves in the reference medium which corresponds to the incident angle, θ_2 is the refraction angle in the sample, T is the thickness of the sample and the reference medium, and f is the ultrasonic wave frequency. From Eq. (1) and Snell's law represented by Eq. (2), the sound velocity in the sample, c_2 , is derived as shown by Eq. (3)⁽⁴⁾.

$$\frac{c_1}{\sin \theta_1} = \frac{c_2}{\sin \theta_2} \quad (2)$$

$$c_2 = \frac{c_1}{\sin \theta_1} \sin \left[\tan^{-1} \left(\frac{1}{\frac{1}{\tan \theta_1} - \frac{N c_1}{f T \sin \theta_1}} \right) \right] \quad (3)$$

For materials with large sound velocity, such as ceramics, and/or large thickness, the correspondence of the fringes produced by the same phase of the ultrasonic waves transmitted through the sample and the reference medium is unclear since the lateral shift of the fringes is much larger and $N > 1$. Therefore fringe shift cannot be accurately measured. In this study, spectral interferometry proposed by Oravec and Lees⁽⁴⁾ is used to avoid this problem. They noticed that the fringe shift is proportional to the frequency under the assumption that sound velocity in the sam-

Fig. 3 Direction of wave penetration and phase fronts in a sample and in a reference medium. Solid lines and chain lines in the front view of the figure show the phase fronts in a sample and in a reference medium, respectively

ple is independent of the frequency in the measurement range. In spectral interferometry, the rate of the change of fringe shift to the change of frequency, dN/df , is measured and it is not necessary to evaluate the fringe shift absolutely. Namely, the value of the relative change of the fringe shift is measured on the monitor as the frequency changes. Using dN/df , the sound velocity of the sample is obtained from Eq. (4).

$$c_2 = \frac{c_1}{\sin \theta_1} \sin \left[\tan^{-1} \left[\frac{1}{\frac{1}{\tan \theta_1} - \left(\frac{dN}{df} \right) \frac{c_1}{T \sin \theta_1}} \right] \right] \quad (4)$$

Velocities of a longitudinal wave and a transverse wave in an isotropic material are represented by Eqs. (5) and (6) ⁽¹²⁾.

$$c_l = \sqrt{\frac{E(1-\nu)}{\rho(1+\nu)(1-2\nu)}} \quad (5)$$

$$c_t = \sqrt{\frac{E}{2\rho(1+\nu)}} = \sqrt{\frac{G}{\rho}} \quad (6)$$

where c_l is the longitudinal wave velocity, c_t is the transverse wave velocity, and ρ , E , G , ν , are the density, Young's modulus, shear modulus, and Poisson's ratio of the material, respectively. Therefore E , G , and ν are given by Eqs. (7) to (9).

$$E = \frac{\rho c_l^2 (3c_l^2 - 4c_t^2)}{c_l^2 - c_t^2} \quad (7)$$

$$G = \rho c_t^2 \quad (8)$$

$$\nu = \frac{3c_l^2 - 4c_t^2}{2(c_l^2 - c_t^2)} - 1 \quad (9)$$

From Eqs. (7) to (9) the elastic constants can be evaluated using the values of the sound velocities and the density.

4. Experimental

In this study, 2.5 mol% Y_2O_3 partially stabilized zirconia ceramics are examined. The material was presintered at 1673 K for 2 h and then hot-isostatically sintered at 1673 K for 1.5 h. Using a diamond saw, the sintered material was cut into four rectangular specimens with dimensions of 100 mm by 10 mm by thickness, T , and was with a density of 6050 kg/m³. The specimens were ground with a diamond wheel. The thicknesses of the specimens measured by a micrometer were 1.87, 1.36, 1.08, and 0.83 mm. The velocities of ultrasonic waves for both modes, viz. the longitudinal wave and the transverse wave, were measured in two regions of about 1 mm². The measuring number was three per region, and the total number was six per specimen. The reference medium was distilled water which served as the coupler.

In order to measure the ultrasonic wave velocities, the wave mode of the interference image appearing on the monitor must be known first. Therefore the incident angle must be determined precisely by considera-

tion of the critical angle of the waves of both modes. From pre-experiments, it has become clear that the critical angle of the specimen prepared for the longitudinal wave is about 12 degrees. The acrylic plastic sample holder to adjust the incident angle was made to excite the ultrasonic waves to the desired mode within the specimen. The incident angle was determined by measuring the fringe interval in the reference medium since the incident angle relates to the fringe interval, frequency of the ultrasonic wave, and the sound velocity in the reference medium, as indicated by Eq. (10).

$$L = \frac{\lambda_1}{\sin \theta_1} = \frac{c_1}{f \sin \theta_1} \quad (10)$$

where θ_1 is the incident angle, L is the fringe interval, λ_1 is the ultrasonic wavelength in the reference medium, f is the frequency of the ultrasonic wave, and c_1 is the sound velocity in the reference medium. In this study, distilled water was used as the reference medium and the incident angle was 9.71 degrees or 13.7 degrees for the longitudinal mode or the transverse mode, respectively.

5. Results

Figure 4 shows the typical interferogram obtained in this study. By the least squares method, the rate of fringe shift change was given as the slope of the apparent fringe shift-frequency line, as shown in Fig. 5. The velocity of sound in the material was derived from Eq. (4) by substituting dN/df . Results are summarized in Table 1. The values in Table 1 are mean values. Young's modulus, shear modulus, and Poisson's ratio which were calculated from Eqs. (7)

Fig. 4 Typical acoustic image in interference mode of partially stabilized zirconia ceramics and distilled water. Incident angle: 9.71 degree. Frequency: 100.0 MHz. The upper part of the image shows distilled water only and the lower part shows zirconia ceramics with distilled water

to (9) by using the mean values of sound velocities are shown in Table 2. The elastic constants measured by tensile tests described in the discussions are also represented in Table 2. The sound velocity in the distilled water serving as the reference medium was $1491.5 \text{ m/s}^{(13)}$ in consideration of the temperature condition of 296 K. The maximum standard deviation of measured sound velocity was 250 m/s and 140 m/s in the longitudinal wave and the transverse wave, respectively.

6. Discussions

Errors involved in the measured values of sound velocities arise from the uncertainty in the sound velocity in the distilled water serving as the reference

Table 1 Velocity of sound measured by SLAM

Sample	Thickness mm	Velocity of Sound	
		longitudinal m/s	transverse m/s
No.1	1.87	7540	3640
No.2	1.36	7570	3690
No.3	1.08	7890	3740
No.4	0.83	7890	3690

Table 2 Elastic constants evaluated by SLAM and tensile tests. The values of shear modulus in tensile tests were calculated from the measured Young's modulus, Poisson's ratio, and density

Sample	Method	Elastic Constants		
		Young's modulus GPa	Shear modulus GPa	Poisson's ratio
No.1	SLAM	216	80.1	0.348
	Tensile	212	80.4	0.317
No.2	SLAM	221	82.4	0.344
	Tensile	206	78.3	0.315
No.3	SLAM	229	84.6	0.355
	Tensile	209	79.9	0.306
No.4	SLAM	226	83.3	0.358
	Tensile	205	77.2	0.328

Fig. 5 Typical relation between apparent fringe shift and frequency. Sample: No. 1. Thickness: 1.87 mm.
○: longitudinal wave. ●: transverse wave

medium, the specimen thickness, the ultrasonic incident angle, and the fringe shift rate. The errors caused by the uncertainty in the sound velocity in the distilled water and the incident angle are not large. The value of sound velocity in distilled water is 1491.5 m/s determined in consideration of the temperature condition described above. The dependency of the sound velocity in distilled water on temperature is not significant. The sound velocity in distilled water may be lower than $1497 \text{ m/s}^{(13)}$ since the increase in temperature during the measurement was smaller than 2°C . The uncertainty in determination of the incident angle may be ± 0.1 degree since the error involved in the ultrasonic frequency may be 0.2 %. Therefore, the total error in the measured sound velocity in the specimen caused by these factors may be smaller than 2 %. On the other hand, the error caused by uncertainty in determination of the rate of fringe shift change is relatively large. The dependency of the measured value of sound velocity in the specimen on the rate of fringe shift change is more significant than that of the factors described above. In this study, the rate of fringe shift change obtained by the least squares method is reliable since the correlation coefficients between the change of fringe shift and the change of frequency are larger than 0.998. The maximum error in the measured sound velocity caused by uncertainty in determination of the rate of fringe shift change, however, reaches 3 %. As a result, the total error involved in the measured sound velocity of the specimen may be about 5 %.

To ensure the validity of the method by use of SLAM proposed in this study, the elastic constants of the same specimens were measured by tensile tests using Instron Material Test Machine 1185 under the condition of 0.5 mm/min crosshead speed. Two strain gauges to measure longitudinal strain were attached to the center area of the specimen surface and gauges to measure longitudinal strain and lateral strain were attached to the central area of the opposite surface of the specimen. The results are summarized in Table 2. The shear modulus was calculated from the measured Young's modulus, Poisson's ratio, and density.

The values of Young's modulus measured by using SLAM are about 10 % larger than those measured by the tensile test. The reasons for these differences are as follows:

- (1) The strain rate in the SLAM measurement, which is a dynamic method, is considerably larger than that in the tensile test, which is a static method.
- (2) Adiabatic Young's modulus is obtained in the SLAM measurement, while isothermal Young's modulus is obtained in tensile test, which is smaller than adiabatic Young's modulus.

The difference caused by the latter can be compensated for if the specific heat capacity and the thermal expansion coefficient are known. That difference would be less than 1% for the zirconia ceramics measured in this study. Therefore the primary reason for the differences in the Young's modulus as measured by SLAM and the tensile test would be the former.

The method described here can be applied to a small specimen when it is difficult to apply mechanical tests or the ultrasonic pulse echo method.

7. Conclusions

A method of measuring the velocity of sound and the elastic constants of ceramics by using SLAM was studied. Young's modulus, shear modulus, and Poisson's ratio of 2.5 mol% Y_2O_3 partially stabilized zirconia were evaluated by measuring the longitudinal ultrasonic velocity and the transverse ultrasonic velocity. The error involved in the measured sound velocity was about 5%. The elastic constants evaluated by SLAM were about 10% larger than those measured by the tensile test. This discrepancy is mainly due to the difference in the strain rate.

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