

Refractive Index Evaluation of Synthetic Silica Glass by the Ultrasonic Microspectroscopy Technology

超音波マイクロスペクトロスコピー技術による合成石英
ガラスの屈折率評価

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1. Introduction

Synthetic silica (SiO₂) glass is widely used as optical components because of its high purity, high optical transmittance, and high homogeneity of refractive index. SiO₂ glass with the optical transmittance for wavelength at 193 nm higher than 99.8%, the distributions of refractive index normal and parallel to the optical axis less than 1×10^{-6} and 5×10^{-6} , respectively, and the birefringence lower than 2 nm/cm, has been developed for lens of the production systems of semiconductor devices (stepper)¹. It is necessary to improve the homogeneity of refractive index of SiO₂ glass for lens of stepper, in order to reduce the size of semiconductor integrated circuits. Refractive indices of SiO₂ glass depend not only to concentrations of impurities such as OH and chlorine, but also to fictive temperature (T_F)². We developed a method of evaluating T_F for SiO₂ glass by acoustic properties measurements using ultrasonic micro-spectroscopy (UMS) technology³. The resolution of T_F by longitudinal velocity measurements is ± 0.3 - ± 0.4 °C, and it is by one or two orders of magnitude greater than that measured by the conventional methods^{4,5}. In this paper, relationships among refractive index and acoustic properties for SiO₂ glass were obtained to establish an indirect ultrasonic method for evaluating refractive index distributions of SiO₂ glass ingots.

2. Specimens

Specimens were prepared from five synthetic silica glass ingots by the direct method (ES; Tosoh SGM Co.). OH concentrations of the ingots measured by infrared spectroscopy⁶ were 1040-1290 wtppm. In order to obtain T_F dependence of refractive index, the ingots were

heat-treated at the different annealing temperatures. Striae distributions were observed for the cubic blocks with sizes of 70 mm × 70 mm × 70 mm cut from the central parts of the ingots, and prisms for refractive index measurements were prepared from the homogeneous parts of each block. No striae were observed for three blocks and weak striae were observed for two blocks. Specimens for acoustic properties measurements were obtained from the neighborhood of the cubic blocks.

In order to obtain T_F dependence of acoustic properties, several specimens were also prepared from an ingot, and heat-treated at the different annealing temperature from 850°C to 1150°C.

3. Experiments and discussion

Refractive indices (n) at 632.82 nm were measured by the minimum deviation method using a spectrometer. Longitudinal velocities (V_1) were measured by the plane-wave ultrasonic material characterization system⁷. Densities (ρ) were measured by the Archimedes method⁸. Measurement accuracies for n , V_1 and ρ are ± 1 ppm ($\pm 1.46 \times 10^{-6}$ for SiO₂ glass), ± 0.05 m/s, and ± 0.05 kg/m³, respectively. Relationships among refractive indices, longitudinal velocities, and densities are presented in **Fig. 1**. The following relationships among V_1 [m/s], ρ [kg/m³], and n were obtained.

$$V_1 = -3.54 \times 10^5 \times n + 521608.9 \quad (1)$$

$$\rho = -7.72 \times 10^3 \times n + 13446.1 \quad (2)$$

V_1 and ρ become smaller, as n higher.

V_1 and ρ were measured for the specimens to obtain T_F dependences of acoustic properties. Annealing temperature (T_A) dependences of V_1 and ρ are shown in **Fig. 2**. The following relationships among V_1 , ρ , and T_F [°C] were obtained.

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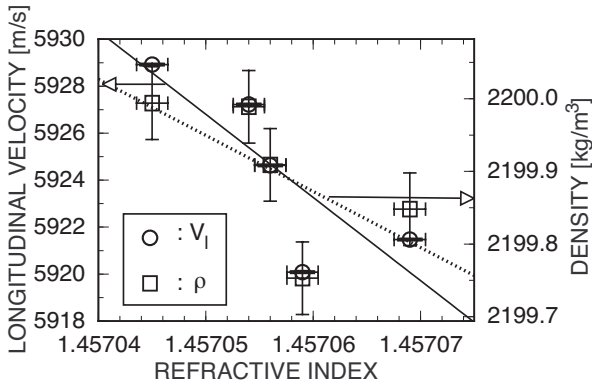


Fig. 1. Relationships among longitudinal velocities, densities, and refractive indices for SiO₂ glass specimens.

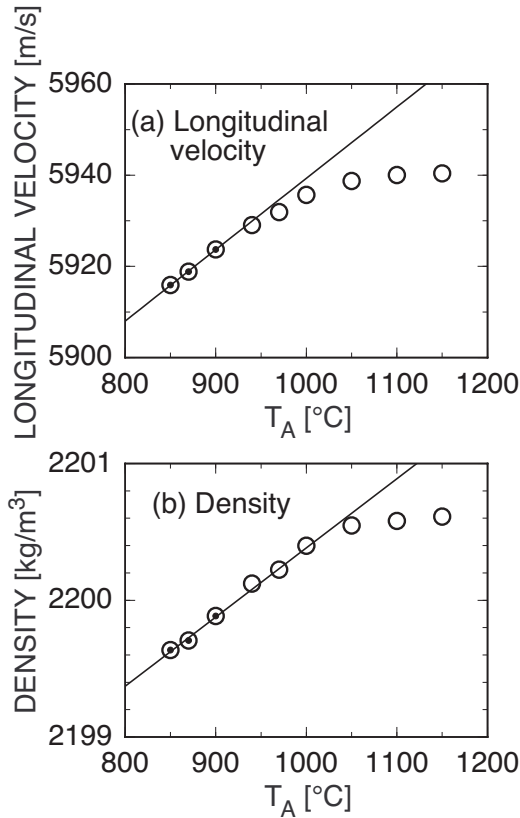


Fig. 2. Annealing temperature (T_A) dependences of longitudinal velocity and density of SiO₂ glass.

Table 1. Sensitivities and resolutions for refractive index of SiO₂ glass (ES) determined by acoustic properties measurements.

	Sensitivity	Resolution
V_1	$-2.8 \times 10^{-6}/(\text{m/s})$	1.4×10^{-7}
ρ	$-1.3 \times 10^{-4}/(\text{kg/m}^3)$	6.5×10^{-6}

$$V_1 = 0.1565 \times T_F + 5782.81 \quad (3)$$

$$\rho = 0.0051 \times T_F + 2195.33 \quad (4)$$

From eq. (1)-(4), we can understand n becomes smaller as T_F higher. From eq. (3), T_F of the SiO₂ ingots were estimated from 877°C to 934°C.

Table 1 presents sensitivities and resolutions of refractive indices determined by measuring acoustic properties. Resolution for refractive index of SiO₂ glass determined by longitudinal velocity is 1.4×10^{-7} , and it is by one order of magnitude higher than that measured by the minimum deviation method.

In Fig. 1, measured values ($n = 1.457059$) are deviated from the approximated lines. It might be caused by optical inhomogeneity of the prism.

4. Summary

In this paper, relationships among refractive indices and acoustic properties were discussed. UMS technology is very useful for evaluating refractive index distributions of SiO₂ glass ingot, and it will be extremely useful for improving the homogeneity of SiO₂ glass ingot. Hereafter, we will obtain more accurate calibration line by fabricating specimens with different T_F from a homogeneous SiO₂ glass ingot.

Acknowledgment

The authors are very grateful to Mr. H. Shishido for measurements for refractive index. This work was partially supported by a Research Grant in Aid from JST, a Grant in Aid for the Global COE program, and a Research Grant-in-Aid for Scientific Research from JSPS.

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