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Evaluating the Homogeneity of a Synthetic Silica Glass Ingot Using 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インゴットの均質性評価

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1. Introduction

Synthetic silica (SiO_2) glass is widely used for optical components because of its high purity and high optical transmission. As semiconductor large-scale integrated circuits using such glass become miniaturized, the homogeneity of the refractive index must be further improved for lenses in optical lithography systems. The refractive index of SiO_2 glass depends both on concentrations of dopants and impurities such as hydroxyl (OH) and chlorine and on the fictive temperature (T_F), a parameter related to the thermal history[1]. We proposed a method of evaluating T_F of SiO_2 glass using ultrasonic microspectrosocpy (UMS) technology by measuring longitudinal-wave velocity[2] and demonstrated that the resolution of T_F is one to two orders of magnitude greater than that measured by conventional methods[3,4]. We applied our method to evaluating the homogeneity of a SiO_2 glass ingot.

2. Specimens

Specimens were prepared from a 600 mm (ϕ) × 600 mm (l) commercial SiO_2 glass ingot (ES; Tosoh SGM) produced by the direct method. Fabricated SiO_2 glass ingots are usually annealed at high temperatures in order to reduce residual stress. To compare acoustic properties before and after annealing, a specimen was first prepared in the radial direction at about 200 mm from the top of the ingot without annealing. The rest of the ingot was annealed. An annealed specimen with the same height as the ingot was also prepared. Several specimens were also prepared from the central parts for heat-treatment at desired annealing temperatures from 850°C to 1150°C to obtain calibration lines between acoustic properties and T_F.

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3. Experiments and discussion

Leaky-surface-acoustic-wave velocities (V_{LSAW}) were measured by a line-focus-beam ultrasonic material characterization (LFB-UMC) system at 225 MHz[5]. Longitudinal velocities (V_l) were measured around 200 MHz by replacing the LFB ultrasonic device with a plane-wave ultrasonic device. Densities (ρ) were measured based on the Archimedes principle. OH concentrations {C(OH)} were measured by infrared spectroscopy[6]. Optical retardation was measured by optical heterodyne interferometry.

Acoustic properties were measured for specimens heat-treated at different annealing temperatures. We obtained linear relationships between acoustic properties and annealing temperatures for specimens annealed at temperatures below 900°C and could thus obtain the following relationships among V_l [m/s], V_{LSAW} [m/s], ρ [kg/m^3], and T_F [°C].

$$V_l = 156.5 \times 10^{-3} \times T_F + 5782.8 \quad (1)$$

$$V_{LSAW} = 0.9 \times 10^{-3} \times T_F + 3419.7 \quad (2)$$

$$\rho = 6.0 \times 10^{-3} \times T_F + 2194.5 \quad (3)$$

Table 1 presents sensitivities and resolutions of fictive temperatures determined by measuring acoustic properties. Measured V_{LSAW} and V_l distributions are presented in **Figs. 1**(A) and (B) as a function of distance from the center. Error bars in Fig. 1(B) represent maximum errors caused by thickness distributions of specimens except for the center positions. Maximum differences of V_{LSAW}

Table 1. Sensitivities and resolutions for fictive temperature of SiO_2 glass (ES) determined by acoustic property measurements.

	Sensitivity	Resolution
Longitudinal velocity	6.4°C/(m/s)	0.3°C
LSAW velocity	1075°C/(m/s)	183°C
Density	168°C/(kg/m^3)	8.4°C

(V_l) is 2.31 m/s (3.10 m/s). We observed that V_{LSAW} increased from the center to the edge, took a local maximum at 218 mm, and decreased around the ingot edge. V_{LSAW} distributions before and after annealing exhibited similar tendencies, although V_{LSAW} slightly increased after annealing. We observed that V_l took a local minimum at 190 mm and increased around the ingot edge. V_l decreased by 8 to 9 m/s after annealing. Figure 1(C) presents the results of OH concentration distribution for the annealed specimens. OH concentrations were constant for specimens taken from around the center of the ingot and decreased by 140 wtppm around the ingot edge. Acoustic properties also depend on C(OH). The maximum difference of T_F for the annealed specimen was estimated from V_l variation using eq. (1) to be 19°C. Here, velocity changes caused by C(OH) distributions were corrected using the results of ref. 2. T_F around the central axis was estimated to be 982°C before annealing and 933°C after annealing. Annealing thus deceased T_F about 50°C. We also measured T_F distributions for the annealed specimen by IR spectroscopy[3], and the results indicated a tendency similar to those estimated from V_l. Optical retardation for the annealed specimen indicated residual stresses around the ingot edge {Fig. 1(D)}. V_{LSAW} decreased in the same region, so V_{LSAW} could be influenced by the residual stresses. These effects could not be reduced by annealing, considering the result of V_{LSAW} distributions.

4. Summary

We demonstrated that T_F distributions of SiO_2 glass ingots can be obtained by V_l measurements and that variations caused by the residual stresses could be evaluated by V_{LSAW} measurements. This ultrasonic method is extremely useful for improving the homogeneity of SiO_2 glass ingots.

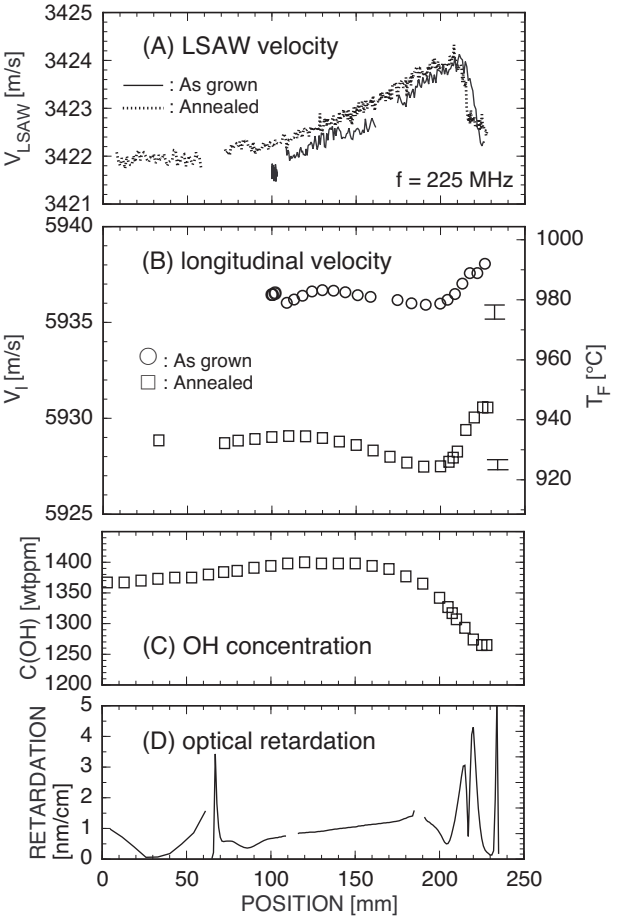

Fig. 1. Distributions of LSAW velocities (A), longitudinal velocities (B), OH concentrations (C), and optical retardation (D) measured for synthetic silica glass specimens.

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