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Evaluation Method of Fictive Temperature of Synthetic Silica Glasses 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仮想温度評価法

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1. Introduction

Synthetic silica (SiO_2) glass is widely used as important material for lithography systems of mass-producting semiconductor devices and for optical fibers in telecommunications. Refractive-index homogeneity and high optical transmittance without defects are in essence required for optical-use SiO_2 glasses, and durability to ultraviolet (UV) lights is also needed for optical components in the production systems working at UV wavelength. Their characteristics are related not only to concentrations of dopants and/or impurities, such as OH and Cl, but also to fictive temperatures T_F. It is necessary to control T_F to fabricate SiO_2 glasses with desired optical properties. Conventionally, T_F is used to be obtained by Raman spectroscopy with a measurement resolution of ±60°C [1] or infrared (IR) spectroscopy with ±15°C [2]. Their measurement accuracies are insufficient for evaluation in order to obtain SiO_2 glasses with further improved optical characteristics.

In this paper, we develop an indirect, ultrasonic method of precisely measuring T_F for SiO_2 glasses by our ultrasonic microspectroscopy (UMS) technology, taking two kinds of specimens with different OH concentrations.

2. Specimens

Specimens were prepared from commercial SiO_2 glasses fabricated by the vapor phase axial deposition (VAD) method (ED-B, Tosoh Quartz Co.) and by the direct method (C-7980, Corning Inc.). OH concentrations, analyzed by IR spectroscopy [3], were 0 wtppm for ED-B and 1000 wtppm for C-7980. The characteristic temperatures of strain and annealing points (T_s and T_a) for both types of glasses were different, reflecting the difference in OH concentration. Dimensions of the specimens were 60 mm × 60 mm × 15 mm^t.

The specimens were heat-treated in air using an electric furnace. They were kept at the desired annealing temperatures for sufficiently long time, considering the structural relaxation times for each specimen [4], and cooled in the furnace by turning off the heater. They were processed by sandwiching them between two silica glass plates with dimensions of 70 mm × 70 mm × 10 mm^t, in order to reduce T_F distributions in the cooling processes. Annealing temperatures T_A were 1050°C, 1100°C, 1150°C, and 1200°C for ED-B, and 900°C, 1000°C, 1050°C, and 1100°C for C-7980. Both surfaces of the specimens were optically polished with a thickness of 10 mm after the heat-treatment.

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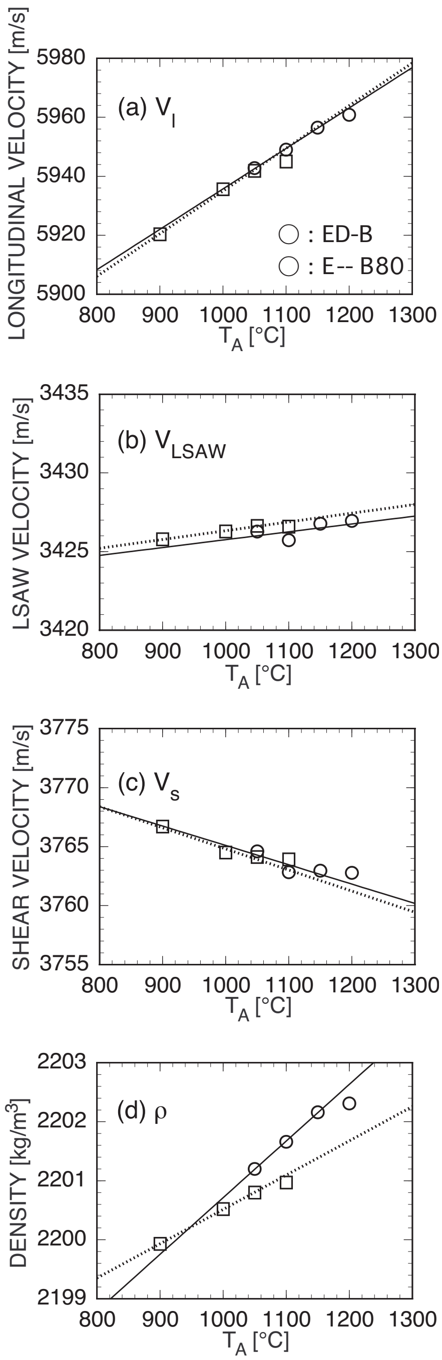

Fig. 1. Annealing temperature dependences of acoustic properties of SiO_2 glasses.

3. Experiments and discussion

Leaky-surface-acoustic-wave velocities (V_{LSAW}) were measured by the line-focus-beam ultrasonic material characterization (LFB-UMC) system at 225 MHz [5]. Longitudinal and shear wave velocities (V_l, V_s) were measured by the plane-wave UMC system in a frequency range of 50-250 MHz [6]. Densities (ρ) were measured by the Archimedes method.

T_A dependences of acoustic properties are shown in Fig. 1. We observed linear relationships between acoustic properties and T_A in temperature ranges up to about 50°C lower than the temperatures of T_a for each specimen, deviating from a straight line at higher temperatures of T_A. This was caused by a fact that structural relaxation times became smaller as T_A of the specimens became larger [4] and their T_F became smaller than T_A in their cooling processes. V_l varied most significantly among the acoustic properties.

It was reported that ρ is in a linear relationship with T_F for SiO_2 glasses [7, 8]. We plotted the data of V_l and ρ, as shown in Fig. 2, and found linear relationships for both types of specimens, and confirmed that V_l changes follow T_F changes properly. We estimated T_F through V_l. We observed linear relationships between acoustic properties and T_F.

V_l varied most sensitively with T_F, from the results of resolutions of T_F by the acoustic properties measurements. The resolutions were 0.3-0.4°C, and they were 40-150 times higher than the conventional methods [1, 2]. So, V_l measurements are extremely useful for evaluation of T_F.

Figure 3 shows the results of two-dimensional V_{LSAW} distributions for a C-7980 specimen annealed at 1000°C. Maximum velocity differences were 1.49 m/s and 1.26 m/s for top and bottom surfaces, respectively. These correspond to T_F distributions of 278°C and 236°C, respectively.

4. Summary

We developed an evaluation method of T_F for SiO_2 glasses. We clarified that V_l measurements are extremely useful with a resolution of T_F 0.3-0.4°C, and V_{LSAW} measurements can provide two-dimensional T_F distributions on the specimen surfaces.

Glass manufacturers can evaluate T_F of fabricated glass ingots, T_F distributions, and their fabrication processes by this ultrasonic method.

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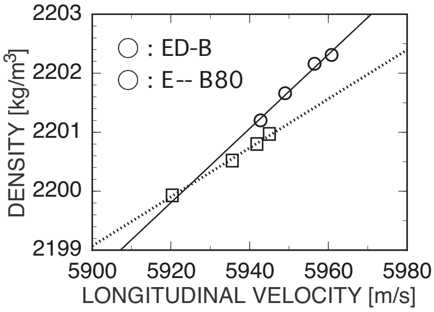

Fig. 2. Relationships between longitudinal velocities and densities for SiO_2 glass specimens.

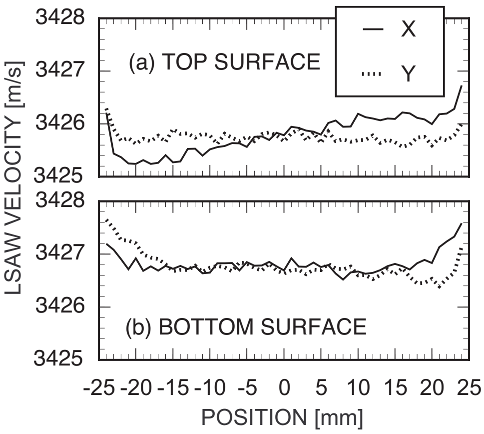

Fig. 3. LSAW velocity distributions for a C-7980 specimen annealed at 1000°C (f = 225 MHz).

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