

Ultrasonic Microspectroscopy of Tempered Glasses

強化ガラスの超音波マイクロスペクトロスコピー

Jun-ichi Kushibiki^{1†}, Mototaka Arakawa¹, Yuji Ohashi¹, Kuniko Kawaguchi¹, and Nobuo Takeda¹ (¹Grad. School Eng., Tohoku Univ.)

櫛引 淳一[†], 荒川 元孝¹, 大橋 雄二¹, 川口 邦子¹, 竹田 宣生¹ (¹東北大院 工)

1. Introduction

Tempered glass^{1, 2)} is a kind of safety glass processed to increase resistance to break by introducing surface compressive stress. It has been used as window glass, dishes, and so on. Tempered glass is broadly divided into physically tempered glass and chemically tempered glass. The former is manufactured through rapid cooling by wind after heating to around softening temperature. The latter is manufactured by immersing in a bath containing a potassium salt, and this causes sodium ions in the glass surface to be replaced by potassium ions from the bath solution. Recently, thinner and more reliable tempered glasses are required for mobile display such as cell phones and Personal Digital Assistant (PDA) and windows for super tall buildings and high-speed rail trains. And, more reliable evaluation methods and their standardizations are also required.

We have studied the development of the ultrasonic microspectroscopy (UMS) technology and the application to material characterization. Line-focus-beam ultrasonic material characterization (LFB-UMC) system is able to measure propagation characteristics (velocity and attenuation) of leaky surface acoustic waves (LSAWs), excited and propagated on the water-loaded specimen surface.³⁾ It is important to evaluate compressive stress layer of substrate surface of tempered glasses. UMS technology will become a useful evaluation method for tempered glasses. In this paper, we discussed a method of evaluating tempered glasses using UMS technology.

2. Specimens

Five aluminosilicate glass {Gorilla glasses (Corning Inc.)} substrates were used as chemically tempered glass specimens. Four specimens were chemically strengthened by immersing in a bath containing KNO_3 at 425°C for 0.5 h, 1 h, 3 h, and 8 h. One specimen was not chemically processed,

kushi@ecei.tohoku.ac.jp

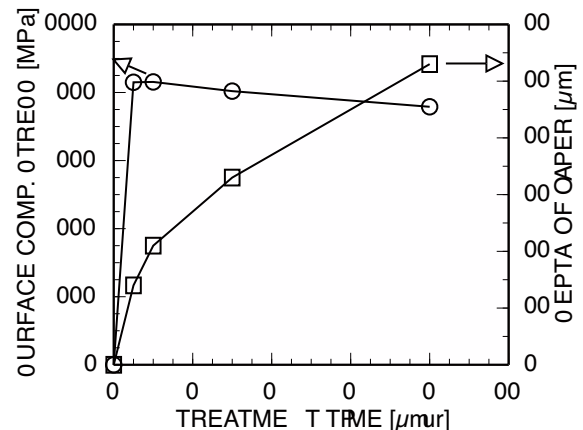


Fig. 1. Treatment time dependences of surface compressional stress and depth of layer for Gorilla glasses.

and used as reference. The surface compressive stress and the depth of compressive stress layer of the specimens were measured by a surface stress meter using an optical waveguide effect. The result is shown in **Fig. 1**. As the process time increases, the depth of compressive stress layer increases monotonically, although the surface compressive stress decreases slightly because of stress relaxation.

A commercial tempered substrate (Specimen H) and the non-processed substrate (Specimen N) were also prepared. The surface compressive stress and the depth of compressive stress layer measured by the optical method were 762 MPa and 49 μm , respectively.

3. Experiments and discussion

Leaky surface acoustic wave velocities were measured by the LFB-UMC system³⁾ at $f = 225$ MHz. Velocities of LSAW and leaky surface skimming compressional wave (LSSCW) were obtained according to the analytical procedure of $V(z)$ curve.⁴⁾ The wavelengths of LSAW and LSSCW for Gorilla glasses were 13-14 μm and 25-26 μm , respectively. Depth of compressive stress layer of the specimens are the order in the magnitude as the wavelength, therefore, the specimen is treated as a thin-film layer structure

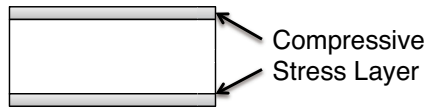


Fig. 2. Cross-section view of tempered glass.

shown in Fig. 2.

Treatment time dependences of leaky surface acoustic wave velocities are shown in Fig. 3. Velocities of tempered glass specimens are larger than that of the unprocessed specimen. Both the LSAW and LSSCW velocities increased until 3 h and saturated. This is caused by that the depth of compressive stress layer becomes larger than the wavelengths of LSAW and LSSCW and that the surface compressional stress becomes smaller as treatment time increases. Figure 4 shows the relationships among LSAW velocities, LSSCW velocities, and the depth of compressive stress layer. Linear relationships were observed for depth of layer and velocities of LSAW and LSSCW within one wavelength. Depth of compressive stress layer could be estimated from leaky acoustic wave velocities by using the results.

Next, longitudinal velocities along the specimen thickness direction were measured for specimens H and N by changing LFB ultrasonic device to plane-wave ultrasonic device.⁵⁾ Densities were measured by the Archimedes method. Measurement results are shown in Table 1. Longitudinal velocity V_1 and density ρ of the specimen H are larger than those of the specimen N. Longitudinal velocity, density, and elastic constant c_{11} ($c_{11} = \rho V_1^2$) of the compressive stress layer were estimated based on the measurement model in Fig. 2 by assuming that the velocity and density changes of the tempered glass are caused by the compressive stress. Changes of c_{11} , ρ , and V_1 were estimated to be +9.1%, +1.2%, and +3.9%, respectively. Therefore, velocity changes of the compressive stress layer were mainly caused by the change of elastic constant.

4. Summary

In this paper, an evaluation method of tempered glasses using UMS technology is discussed. We demonstrated that the depth of compressive stress layer of tempered glass could be evaluated by the leaky surface acoustic wave velocities measurements. This method could be also applied for physically tempered glasses.

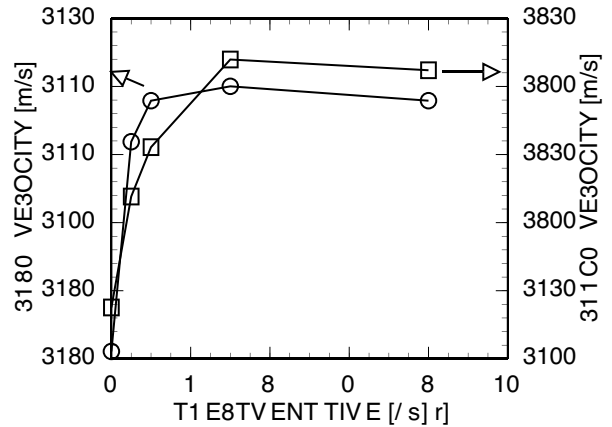


Fig. 3. Treatment time dependences of LSAW and LSSCW velocities for Gorilla glasses.

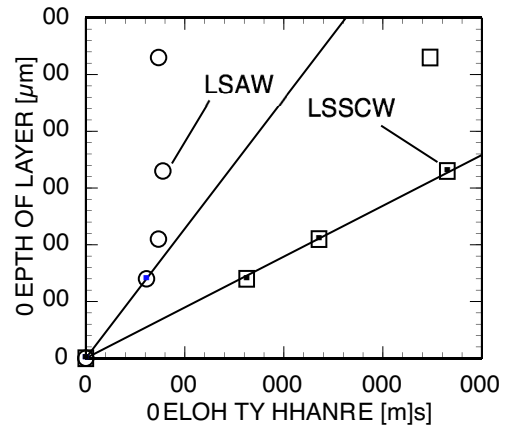


Fig. 4. Relationships between velocity changes of LSAW and LSSCW and depth of layer for Gorilla glasses.

Table 1 Density, Longitudinal velocity, and c_{11} for Gorilla glass.

	ρ [kg/m ³]	V_1 [m/s]	c_{11} [$\times 10^{10}$ N/m ²]
Specimen N	2429.1	5734.2	7.9870
Specimen H	2433.0	5765.8	8.0885
Compressive stress layer	2457.3	5960.1	8.7119

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