1P2-23

Proceedings of Symposium on Ultrasonic Electronics, Vol. 34 (2013) pp. 81-82
20-22 November, 2013

Homogeneity Evaluation of Ultra-Low Expansion Ceramics 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超低膨張セラミックスの均質性評価

Yuji Ohashi[1†], Jun-ichi Kushibiki[1], Mototaka Arakawa[1], Shun-ichi Sasaki[2], and Mabito Iguchi[2] ([1]Tohoku Univ., [2]Nihon Ceratec)
大橋雄二 [1†] 櫛引淳一 [1] 荒川元孝 [1] 佐々木俊一 [2] 井口真仁 [2]([1] 東北大院工, [2] 日本セラテック)

1. Introduction

Ceramics compounded with a proper ratio of lithium aluminum silicate (LAS) and silicon carbide (SiC), which have negative and positive thermal expansion coefficients respectively, are attractive materials having zero expansion characteristics at room temperature, high stiffness, and good formability [1]. Therefore, those are widely applied to mirrors for laser interference, parts of precision stage for electron beam exposure system, and so on. Recently, those also are promising materials as an optical cavity for the next generation optical frequency standard. It is important to be homogeneous as well as to exhibit zero coefficient of thermal expansion (CTE) for use as a spacer of the optical cavity. We have developed a method for evaluating CTE of TiO_2-SiO_2 ultra-low expansion glass by using the ultrasonic microspectroscopy (UMS) technology [2, 3]. In this paper, we will expand our evaluation method to the ultra-low expansion ceramics.

2. Specimens

We prepared two specimens of zero expansion stiffness ceramics (ZPF) fabricated by Nihon Ceratec Co., Ltd. Dimensions of the specimens were 100 mm$^{\Phi}$ × 16.8 mm^{t} for ZPF-1, 50 mm × 50 mm × 8.0 mm^{t} for ZPF-2. Both specimens were optically polished for one surface.

3. Experiments

For the specimens shown above, two-dimensional velocity distributions of leaky surface acoustic wave (LSAW) propagating on a water loaded specimen surface were measured at ultrasonic frequency of 225 MHz using the line-focus-beam ultrasonic material characterization (LFB-UMC) system, one of the

E-mail: ohashi@ecei.tohoku.ac.jp

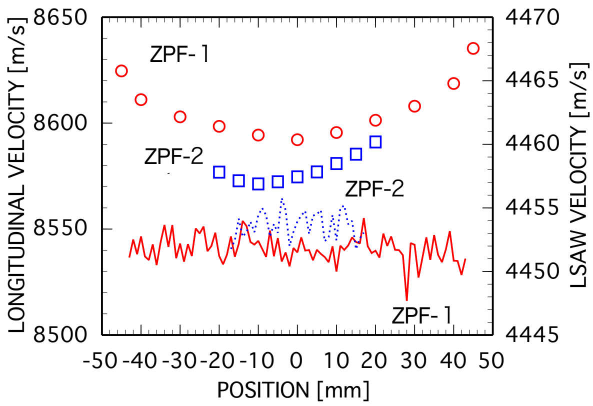

Fig. 1 Longitudinal and LSAW velocities distributions for ultra-low expansion ceramics.

UMS technology. Measured results of the average and variation of LSAW velocities were 4453.8 m/s and 9.2 m/s for ZPF-1, 4452.0 m/s and 7.7 m/s for ZPF-2. Fig. 1 shows the velocity distributions along diameter direction extracted from the results of two-dimensional data, exhibiting relatively flat velocity profile though the velocity variations were large.

Using the plane-wave ultrasonic material characterization (PW-UMC) system, we measured velocity distributions of longitudinal waves propagating along thickness direction of each specimen on the same scanning line of the LSAW velocity measurements shown in Fig. 1 at frequency range of 50 to 160 MHz. The results were also shown in Fig. 1. The result for ZPF-1 exhibited centrosymmetric velocity profile with 43.1 m/s increase at maximum at peripheral parts from the value of 8592.2 m/s at a central position. Assuming the position -10 mm for ZPF-2 specimen as a center position of the base material before forming ZPF-2 specimen, the velocity increases by 19.8 m/s at +20 mm as compared to 8571.3 m/s at central position. Although similar velocity distributions were observed for ZPF-1 and ZPF-2 exhibiting velocity increase of 15.8

m/s for ZPF-1 in the same scanning range of ZPF-2, the difference of 20.9 m/s was observed between the minimum velocities of two specimens.

Measurement result of density for ZPF-2 was 2497.1 kg/m^3.

Taking into account parabolic velocity profiles shown in Fig. 1, we prepared three specimens with dimensions of 5.5 mm$^{\Phi}$×13 mm^{l} for measuring CTE from three positions with different longitudinal velocities along diameter direction of each specimen. A dilatometer using double-path Michelson laser interference (LIX-1, ULVAC RIKO) was used for measurements of CTE. The measurements were conducted three times in a temperature range from -50 to 100°C, then we obtained an average CTE at 23°C. Fig. 2 shows relationships between the CTE results and longitudinal velocities measured at corresponding positions for each specimen. Error bars in Fig. 2 indicate the deviations for three times measurements of CTE. Although measurement accuracy of CTE was not sufficient in Fig. 2, we can observe a tendency that the CTE decreases with the increase of longitudinal velocity. We can also observe significant difference in CTE values between ZPF-1 and ZPF-2.

4. Discussion

The ZPF is fabricated through the following processes: crushing and mixing materials, granulation, compression, shaping, baking treatment, and final polishing. Therefore the distribution in chemical composition ratio is homogeneously fixed until the compression process. However, it is considered that the acoustic properties and CTE vary depending on variations in process conditions such as temperature distributions at the baking treatment. In Fig. 1, we observed the longitudinal velocities increased at the peripheral part of the specimens. It is thought to be due to high treatment temperature at the peripheral part of the specimen resulting in acceleration of crystallization. So the growth of the LAS microcrystals having negative thermal expansion was promoted and the CTE at the peripheral part of the specimens decreased.

On the other hand, the velocity profiles of LSAW along diameter direction were almost flat. Significant velocity differences between the two

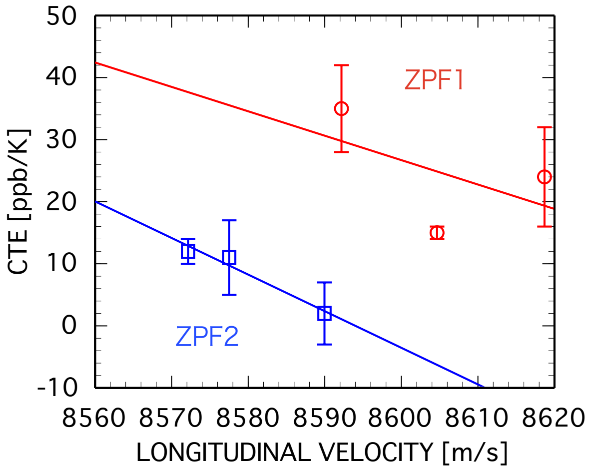

Fig. 2 Relationships between longitudinal wave velocities and CTE for ultra-low expansion ceramics.

specimens were detected by 1.9 m/s for LSAW velocity and 20.9 m/s for longitudinal velocity. This result reflects difference in chemical composition ratio of source materials.

5. Conclusion

We tried to evaluate the ultra-low expansion ceramics by using the UMS technology. Velocity distributions of LSAW and longitudinal wave were measured along diameter directions for two ultra-low expansion ceramics specimens. Large velocity changes were detected particularly in longitudinal wave exhibiting centrosymmetric changes with the maximum variation of 43 m/s. It is considered to be due to distribution of treatment temperature at the baking process. We can also detected the velocity differences in both of LSAW and longitudinal wave between two specimens due to difference in chemical composition ratio in source materials. Furthermore, we found that the CTE of the ultra-low expansion ceramics correlate linearly with longitudinal velocity. We successfully demonstrated that the UMS technology had capability of accurate CTE evaluation for ultra-low expansion ceramics.

Reference

[1] M. Iguchi, M. Ishii, and M. Umezu, J. Soc. Inorganic Mat. Jpn., **19** (2012) 110.

[2] J. Kushibiki et al., Jpn. J. Appl. Phys., **44** (2005) 4374.

[3] J. Kushibiki et al., Proc. SPIE **7636** (2010) 76362M.