Low Temperature Fabrication of $Bi_4Ti_3O_{12}/Al_2O_3$ Sol-Gel Composite Ultrasonic Transducer

$Bi_4Ti_3O_{12}/Al_2O_3$ 超音波トランスデューサの低温作製に関する研究

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1. Introduction

In recent years, on-line ultrasonic nondestructive testing (NDT) in the industrial fields has been demanded[1),2)] and sol-gel composite ultrasonic transducers have been developed for industrial NDT applications.[3-7)] Compared with conventional ultrasonic sensors, no coupling agent nor backing material is required, so ultrasonic NDT can be performed at high temperature during operation. It also makes it possible to measure on curved surfaces. Considering the ultrasonic NDT in various situations of industrial applications, there is a demand for manufacturing on a curved surface as well.

$Bi_4Ti_3O_{12}$(BiT)/Pb(Zr,Ti)O_3(PZT) sol-gel composite showed high temperature durability up to 500°C.[3)] However, there is a possibility of Pb evaporation in the PZT sol-gel phase and it would contaminate the environment. Therefore, development of lead-free sol-gel composites has been desired. Thus, sol-gel composite ultrasonic transducers using lead-free sol-gel phase have been developed. BiT/BiT sol-gel composite ultrasonic transducer showed high temperature durability up to 600°C.[6)] However, the poling processes require high temperatures, making it difficult to put into practical use.

In our previous study, BiT/Al_2O_3 sol-gel composite ultrasonic transducer poled at room temperature showed high temperature durability up to 600°C. Thereafter, low temperature firing using TiO_2 sol-gel solution showed higher characteristics than the high temperature firing. [7)] In this study, ultrasonic performance and high-temperature durability of BiT/Al_2O_3 with different sintering temperatures were investigated.

2. Fabrication process

BiT/Al_2O_3 sol-gel composite film was fabricated by sol-gel spray technique and the manufacturing process is similar to previous works.[3-7)] After mixing the self-made Al_2O_3 sol-gel solution with the commercially available BiT powder, the mixture was ball-milled until a suitable viscosity for spray coating was achieved. Then, the prepared mixture was coated on a 3mm thick titanium substrates by a manual spray coating method. After spray coating, drying was carried out at 150°C for 5 minutes, and firing was carried out at 450°C or 650 for 5 minutes. These steps, spray coating, drying and firing were repeated until the film thickness reached the target thickness. The target film thickness for this study was 50μm, and these processes were repeated four times. Platinum paste was applied as a top electrode on the prepared film. For the platinum paste curing, heat treatment at 150 °C and 700 °C was carried out for 2 hours each. After preparation of the top electrode, poling treatment was carried out at room temperature. The output voltage of the power supply was about 27 kV. In this study, the distance between the needle tip and the film was adjusted to 25 mm in order to prevent dielectric breakdown of the film due to arc discharge. Optical image of BiT/Al_2O_3 sample fired at 450°C is shown in **Fig.1**.

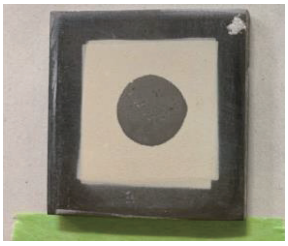

Fig.1 Optical image of BiT/Al_2O_3 sample fired at 450 °C film on 3mm thick titanium substrate.

3. Experimental results

High temperature durability of BiT/Al_2O_3 was investigated. A platinum wire was connected to the upper electrode and the titanium substrate to establish electrical connection, a ceramic weight was placed on it, and platinum wire was used for

high temperature durability. Ceramic weight was used because it has high temperature durability and there was no peeling due to thermal expansion mismatch due to adhesive material. The entire sample was placed in a furnace ultrasonic measurements were performed in pulse echo mode and recorded with a digital oscilloscope at various temperatures. In the pulse echo mode, the reflected echoes from the bottom of a 3 mm thick titanium substrate were measured from room temperature until to 600 ° C in increments of 100 ° C. This thermal cycle test was conducted for 3 cycles. **Figs.2,3** shows the ultrasonic response in time domain at room temperature and 600°C of the BiT/Al_2O_3 sample fired at 450°C.

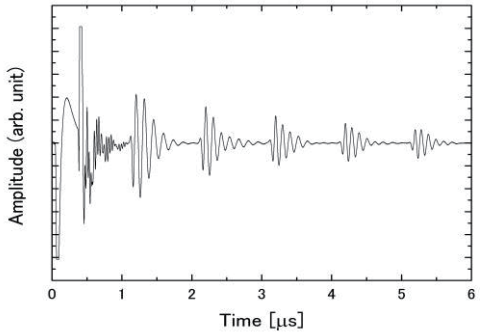

Fig.2 Ultrasonic response at room temperature of the BiT/ Al_2O_3 sample fired at 450°C

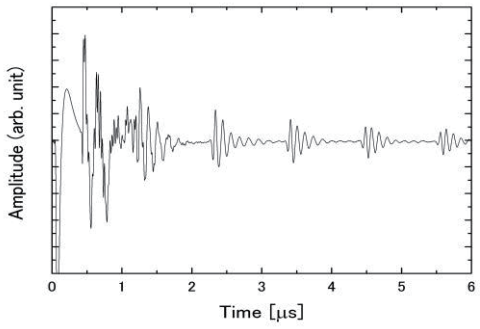

Fig.3 Ultrasonic response at 600°C of the BiT/ Al_2O_3 sample fired at 450°C

In order to determine the temperature effect quantitatively, sensitivity was calculated as following equation;

$$Sensitivity = -(20\log\frac{V_1}{V_2} + Gain\ of\ \mathrm{P/R}) \quad (1)$$

where V_1 is the reference amplitude, which is 0.1 $V_{p\text{-}p}$ in this experiment, V_2 is the $V_{p\text{-}p}$ of the third reflected echo from the bottom surface of the titanium substrate. P/R means pulser/receiver so that this equation calculate true required gain of pulser/receiver in order to achieve 0.1V. -1 is multiplied to assist intrinsic understanding. **Fig.4** shows a comparison between 450°C firing and 650°C firing. Performance of BiT/Al_2O_3 fired at 450°C was comparable to that of BiT/Al_2O_3 fired at 650°C.

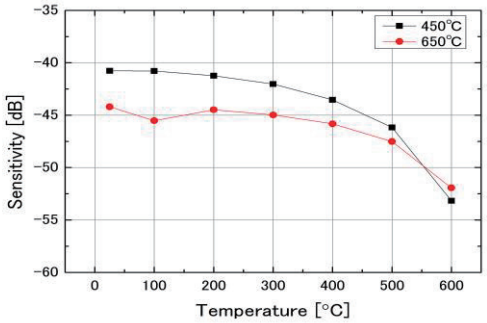

Fig.4 The sensitivity comparison in the 3rd cycle of the thermal cycle test

4. Conclusions

BiT/Al_2O_3 sol-gel composite film was fabricated by room temperature poling and low temperature firring for practical use. As a result, the ultrasonic response could be confirmed up to 600°C even though poling was operated at room temperature. Thermal cycle test is necessary to determine long-term operation temperature. As a result of conducting a three-cycle test from room temperature to 600°C, it was possible to confirm the ultrasonic response also in the third cycle. Also, performance of BiT/Al_2O_3 fired at 450°C was comparable to that of BiT/Al_2O_3 fired at 650°C. However, platinum curing temperature was high. Top electrode will be replaced with low curing temperature paste for final conclusion.

References

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