Bi4Ti3O12/Al2O3 Sol-Gel Composite Ultrasonic Transducer

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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 in industrial applications, there is a demand for manufacturing on a curved surface.

In our previous work, we have developed $Bi_4Ti_3O_{12}(BiT)/Pb(Zr,Ti)O_3(PZT)$ sol-gel composite and it showed high temperature durability up to 600°C. However, there is a possibility of Pb evaporation in the PZT sol-gel phase and it would affect the environment, so development of a leadfree piezoelectric material has been desired. Thus, sol-gel composite ultrasonic transducers using leadfree sol-gel phase has been developed. The BiT/BiT sol-gel composite ultrasonic transducer showed high temperature durability up to $600^{\circ}C.^{7)}$ However, it required high temperature for poling process and it increased difficulty for practical usage.

In this research lead-free BiT/Al_2O_3 sol-gel composite material has been developed and high temperature durability of BiT/Al_2O_3 was investigated.

2. Fabrication process

The manufacturing process is similar to previous works.³⁻⁷⁾ After mixing the self-made Al₂O₃ sol-gel solution with the commercially available BiT powder, the mixture was ball-milled until a suitable viscosity for spray coating was achieved. Thereafter, the prepared mixture was coated on a 3mm thick titanium substrates by a manual spray coating method. After spray coating, drying and firing were carried out at 150 °C and 650 °C for 5 minutes, respectively. 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 50 μ m, 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 a distance of 30 mm in order to prevent dielectric breakdown of the film due to arc discharge. Optical image of BiT/Al₂O₃ film onto titanium substrate is shown in **Fig.1**.



Fig.1 Optical image of BiT/Al₂O₃ 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 no reflected echo was visible. The temperature was raised by 100 °C up to 600 °C and by 10 °C above 600°C. At each temperature, the temperature was maintained for 5 minutes. **Figs. 2,3** shows the ultrasonic response in time domain at room temperature and 720 °C, respectively. Reflected echo disappeared at 730 °C, but at 720 °C we were able to clear multiple echoes with relatively high signal-to-noise ratio (SNR).



Fig.2 The ultrasonic response of BiT/Al₂O₃ transducer on 3mm thick titanium substrate at room temperature.



Fig.3 The ultrasonic response of BiT/Al_2O_3 transducer on 3mm thick titanium substrate at 720 °C

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

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

where V_1 is the reference amplitude, which is 0.1 V_{p-p} in this experiment, V_2 is the V_{p-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 the sensitivity from room temperature to

720 °C. It demonstrated the possibility as a lead-free high-temperature ultrasonic transducer up to 500° C continuously.



Fig.4 The temperature dependency of BiT/Al₂O₃ transducer sensitivity.

4. Conclusions

 BiT/Al_2O_3 sol-gel composite film was fabricated on a 3-mm thick titanium substrate by a spray coating method. In order to clarify high temperature durability of BiT/Al_2O_3 ultrasonic transducer, ultrasonic measurement was carried out in an electric furnace until reflection echoes disappeared. As a result, the ultrasonic response could be confirmed up to 720°C even though poling was operated at room temperature. Thermal cycle test is necessary to determine long-term operation temperature.

References

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