

## Polarization and High Temperature Characteristics of $\text{Bi}_4\text{Ti}_3\text{O}_{12}/\text{Al}_2\text{O}_3$ Sol-Gel Composite Ultrasonic Transducer

$\text{Bi}_4\text{Ti}_3\text{O}_{12}/\text{Al}_2\text{O}_3$  超音波トランスデューサーの分極と高温特性に関する研究

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### 1. Introduction

In recent years, non-destructive inspection (NDT) is required in the industrial field for reasons of safety and economy.<sup>1),2)</sup> The sol-gel composite ultrasonic transducer was developed for industrial NDT applications and does not require a less heat resistant couplant/backing material compared to conventional ultrasonic sensors, allowing it to be used at higher temperatures.<sup>3-5)</sup> It is also possible to measure on curved surfaces, and curved surfaces are required for various industrial applications.  $\text{Bi}_4\text{Ti}_3\text{O}_{12}(\text{BiT})/\text{Pb}(\text{Zr},\text{Ti})\text{O}_3(\text{PZT})$  sol-gel composite has shown a stable ultrasonic response up to 500°C, but contains lead in the PZT sol-gel phase.<sup>3)</sup> Lead can adversely affect the environment and the human body. Therefore, the development of a lead-free sol-gel composite material has been desired. Therefore, a sol-gel composite ultrasonic transducer using a lead-free sol-gel phase has been developed. The BiT/BiT sol-gel composite ultrasonic transducer showed high temperature durability up to 600°C, but it is difficult to put into practical use because it requires high temperature for polarization treatment.<sup>5)</sup> In the previous research, BiT/ $\text{Al}_2\text{O}_3$ , which was lead-free and polarizable at room temperature, was prepared using a hand spray method and showed high temperature durability up to 600°C.  $\text{Al}_2\text{O}_3$  is excellent in that it is lead-free, inexpensive, and easy to manufacture.

In this study, BiT/ $\text{Al}_2\text{O}_3$  was prepared by an automatic spray method, and the samples were poled by positive and negative polarization, respectively, and the high temperature characteristics were investigated.

### 2. Sample Fabrication

BiT/ $\text{Al}_2\text{O}_3$  was prepared by sol-gel spray technology. After mixing home-made  $\text{Al}_2\text{O}_3$  sol-gel solution with commercial BiT powder, the mixture was ball-milled until the viscosity was suitable for

spray coating. Next, the adjusted mixture was coated on a titanium substrate with a thickness of 3mm by an automatic spray coating method. After spray coating, it was dried at 150° C. for 5 minutes and baked at 650° C. for 5 minutes. These steps, spray coating, drying and baking were repeated until the film thickness reached the target thickness. The target film thickness for this study was 50  $\mu\text{m}$  and these processes were repeated 6 times. Platinum was applied as the top electrode of the prepared film. The platinum paste was cured by heat treatment at 150° C. and 700° C. for 2 hours each. After producing the upper electrode, polarization treatment was performed at room temperature. The output voltage of the power supply was about 30kV. In this study, the distance between the tip of the needle and the film was adjusted to 20 mm to prevent the film from dielectric breakdown due to arcing. Two samples were prepared, and two types of polarization treatment were performed: positive polarization and negative polarization. An optical image of the BiT/ $\text{Al}_2\text{O}_3$  sample is shown in Fig.1.

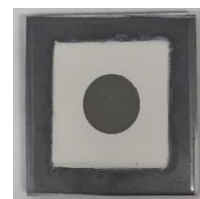


Fig.1 50 $\mu\text{m}$  thick BiT/ $\text{Al}_2\text{O}_3$  film with platinum top electrode on 3mm thick titanium substrate.

### 3. Experimental results

A maximum temperature test was conducted to confirm the high temperature durability of the BiT/ $\text{Al}_2\text{O}_3$  sample. The sample was put in a furnace, a platinum wire was connected to the upper electrode and the titanium substrate to make an electrical connection, and a ceramic weight was

placed thereon. Since it uses a ceramic weight, it has high temperature durability, and there was no peeling due to thermal expansion deviation due to the adhesive material. After that, ultrasonic measurement was performed in the pulse echo mode, the temperature of the furnace was raised by 100° C., and after being held for 5 minutes, the ultrasonic response at each temperature was recorded by a digital oscilloscope. Recording was performed in 20°C increments after 600°C, and in 10°C increments after 700°C. The test was terminated when there was no ultrasonic response. The maximum temperature test was performed on the positively poled sample and the negatively poled sample, respectively. Fig.2 and Fig.3 show the ultrasonic response of BiT/Al<sub>2</sub>O<sub>3</sub> samples with positive and negative polarization at 760°C.

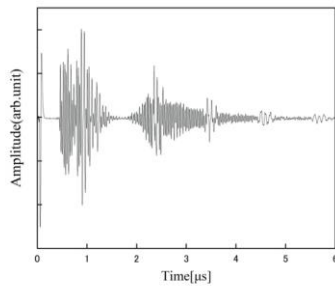


Fig.2 Ultrasonic response of positively poled BiT/Al<sub>2</sub>O<sub>3</sub> sample at 760°C

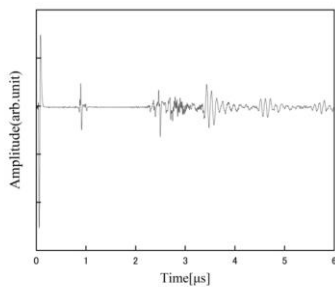


Fig.3 Ultrasonic response of negatively poled BiT/Al<sub>2</sub>O<sub>3</sub> sample at 760°C

To evaluate the test results quantitatively, the sensitivity was calculated by the formula:

$$\text{Sensitivity} = -20\log_{10}(V_1/V_2 + \text{P/R Gain}) \text{ [dB]}$$

Where V<sub>1</sub> is the reference amplitude, which is 0.1 V<sub>pp</sub> in this experiment. V<sub>2</sub> is the V<sub>pp</sub> of the third reflected echo from the bottom surface of the titanium substrate. Since P/R stands for pulser/receiver, this equation calculates the true required gain of the pulser receiver to achieve 0.1V.

-1 is multiplied to help the essential understanding. Fig.4 shows the sensitivity transition of BiT/Al<sub>2</sub>O<sub>3</sub> with positive and negative polarization. As the temperature increased for both positive and negative poled samples, the sensitivity decreased and there was no ultrasonic response after 770°C. Therefore, both the positively poled sample and the negatively poled sample showed high temperature durability up to 760°C. As for the sensitivity, after 700 °C, the same sensitivity transition was observed for positive and negative polarization.

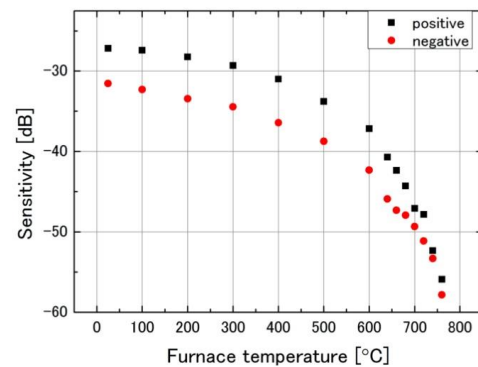


Fig.4 Sensitivity of BiT/Al<sub>2</sub>O<sub>3</sub> samples in maximum temperature test

#### 4. Conclusions

In this study, BiT/Al<sub>2</sub>O<sub>3</sub> sol-gel composites were prepared by an automated spray method. The sample was poled at room temperature and subjected to the maximum temperature test with positive and negative polarization, and both showed high temperature durability up to 760°C. Sensitivity was also the same for positive polarization and negative polarization. From the above results, no significant difference was found between BiT/Al<sub>2</sub>O<sub>3</sub> in positive and negative polarization. The reason is that it has a very low dielectric constant and high resistivity, so it is not easily affected by the pulser receiver.

#### References

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