Comparison of High Temperature Characteristics of $Bi_4Ti_3O_{12}$-based, Lead-Free Ultrasonic Transducers

非鉛 $Bi_4Ti_3O_{12}$ ベース超音波トランスデューサの高温特性の比較

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1. Introduction

In recent years, online non-destructive inspection has been required in the industrial field. One of the main non-destructive inspection methods is non-destructive inspection with an ultrasonic transducer. The sol-gel composite ultrasonic transducer does not require a couplant and backing material and can be used at high temperatures.[1)] $Bi_4Ti_3O_{12}$(BiT)/Pb(Zr,Ti)O_3(PZT) sol-gel complex ultrasonic transducer produced in the previous study showed operational stability up to 500℃, but there was a problem that the material used contained lead.[2)] Lead is harmful to the human body and the environment. The lead-free BiT/BiT developed there showed operational stability up to 600 ℃, but it requires polarization at high temperatures. An ultrasonic transducer that is lead-free and can be poled at room temperature is suitable for practical use.[3)] BiT/TiO_2+Sr developed there showed high temperature durability equivalent to that of BiT/PZT and BiT/BiT, and showed a high sensitivity transition. In addition, the sample fired at 400 ℃ showed better performance than the conventional 650℃ firing. It is considered that the performance was improved because BiT/TiO_2+Sr became a single phase by firing at 400°C.[4-5)]

In this study, various samples such as low-temperature firing were prepared for the purpose of comparing the high-temperature characteristics of lead-free BiT-based ultrasonic transducers, and high-temperature tests were conducted to investigate their performance.

2. Sample Fabrication

BiT/TiO_2+Sr was fabricated by automatic sol-gel spray technology. BiT/TiO_2+Sr was prepared by mixing BiT piezoelectric powder and TiO_2+Sr sol-gel solution and crushing them with a ball mill machine until the clay was suitable for spray coating. After crushing with a ball mill machine, a sol-gel composite was spray-coated on a titanium substrate having a thickness of 3 mm. After spray coating, natural drying, 150℃ drying, and 400℃ firing were performed for 5 minutes each. This time, samples with firing temperatures of 400°C and 200°C were prepared. Spraying, drying and firing were repeated 6 times until the target film thickness was 50μm. The prepared sample was subjected to polarization treatment at room temperature in order to have piezoelectricity. The polarization was performed for 5 minutes by adjusting the distance between the tip of the needle and the film to 3cm. At that time, the output voltage was about 40kV. After that, the top electrode was fabricated by sputtering. Copper and titanium were used as the materials for the top electrodes. The completed optical image of BiT/TiO_2+Sr is shown in **Fig.1**.

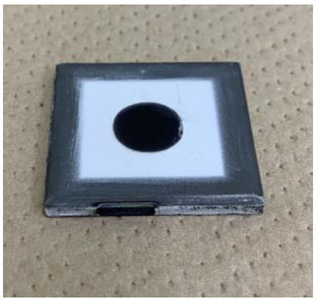

Fig.1 50μm thick BiT/TiO_2+Sr (400°C firing) has copper and titanium top electrodes on a 5 mm thick titanium substrate

3. Experimental Results

A maximum temperature test was performed to confirm the high temperature durability of BiT/TiO_2+Sr(200°C firing and 400°C firing).
A platinum wire was connected to the top electrode of the sample placed in the furnace and the titanium substrate, and ultrasonic measurement was performed in pulse echo mode. The temperature of the furnace was raised from room temperature, and the waveform at each temperature was recorded with a digital oscilloscope. Recording was performed by raising the temperature until the waveform disappeared. At the time of measurement, the substrate temperature was measured using a thermocouple. **Fig.2** and **Fig.3** show the ultrasonic response at room temperature(RT) and 660°C at BiT/TiO_2+Sr (400°C firing).

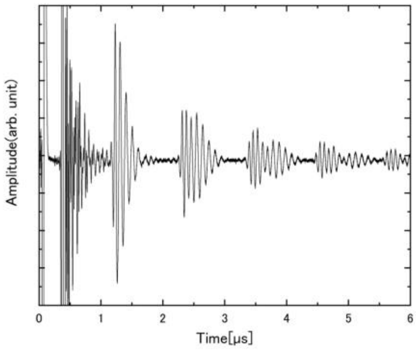

Fig.2 Ultrasonic response of BiT/TiO_2+Sr(400℃ firing) on 3mm thick titanium substrate at 660℃ in maximum temperature test

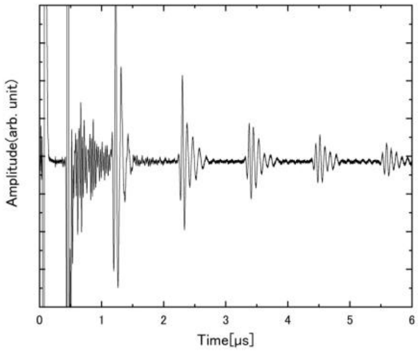

Fig.3 Ultrasonic response of BiT/TiO_2+Sr(200℃ firing) on 3mm thick titanium substrate at 660℃ in maximum temperature test

To quantitatively evaluate the results of the maximum temperature test, the sensitivity was calculated by the following formula.

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

where V_1 is the reference amplitude, which is 0.1 V_{pp} in this experiment. V_2 is the V_{pp} of the third reflected echo from the bottom of the titanium substrate. Since P/R represents the pulsar / receiver, this formula calculates the true gain required for the pulsar receiver to achieve 0.1V. -1 is multiplied to aid in essential understanding.

Fig.4 shows the sensitivity transition of BiT/TiO_2+Sr(200°C firing and 400°C firing) in the maximum temperature test. Both showed high temperature durability up to 660°C and high sensitivity in the high temperature range of 600°C or higher.

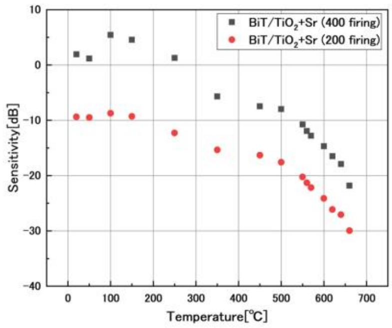

Fig.4 Temperature dependence of sensitivity of BiT/TiO_2+Sr(400℃ firing) and BiT/TiO_2+Sr(200 firing) on 3mm thick titanium substrate in maximum temperature test

4. Conclusions

In this study, BiT/TiO_2+Sr was prepared and the comparison of performance by low-temperature firing was investigated. The sample was spray-coated on a titanium substrate having a thickness of 3mm by a sol-gel spray method. A 50μm thick film was poled to give it piezoelectricity. Copper and titanium were used for the top electrode by sputtering. This time, two samples were prepared, one was fired at 400°C and the other was fired at 200°C. A maximum temperature test was performed using two samples to examine high temperature durability. Both showed high temperature durability up to 660°C. In addition, it showed a high sensitivity transition even at high temperatures.

In the future, it will be necessary to prepare a BiT-based sol-gel complex and compare its performance under conditions such as low-temperature firing.

References

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