Effect of Electrical Properties on Poling Temperature of Bi₄Ti₃O₁₂-based Sol-gel Composite

Naoki Kambayashi1[†], Naoki Zaito1, and Makiko Kobayashi1 (1Kumamoto Univ.)

1. Introduction

Non-destructive testing (NDT) have important role in industrial field. NDT can investigates the state of defects and deterioration without damage. In order to perform NDT in a high temperature environment, piezoelectric ceramics that can withstand the high temperature are required. The piezoelectric ceramics fabricated by the sol-gel composite method do not require a couplant or backing material, and the piezoelectric ceramics themselves have high temperature durability.¹⁻²⁾ In recent years, piezoelectric was required to use lead-free material. Bi₄Ti₃O₁₂(BiT) is lead-free piezoelectric material and research is being conducted alternative lead material. Previous BiT/BiT transducer was fabricated. studies, BiT/BiT ultrasonic transducer has shown high temperature durability up to 500°C.³⁻⁴⁾ However, since it must be poled at 400°C, there are difficult for mass production and practical use. Therefore, it was required to develop a lead-free and easy to polarize sol-gel composite material.

In recent studies, BiT/TiO₂(TO) can be poled at room temperature without lead material. In addition, BiT/TO was further improved to produce BiT/TO+Sr that can be fired at low-temperature. BiT/TO+Sr showed high temperature durability up to 660°C and stable ultrasonic respons.⁵⁻⁶⁾ Even when the TO+Sr sol-gel solution is used to another piezoelectric powder, there are cases where the poling temperature is significantly improved,⁷⁾ and it is showed that the TO+Sr solution is useful for improving the poling temperature in the sol-gel composite.

In this study, The electrical properties of BiT/TO+Sr sample were measured. By measuring resistance, capacitance, and comparing BiT/TO+Sr and BiT/BiT, investigated the cause of the decrease in poling temperature.

2. Fabrication of BiT Sol-gel Composite

BiT-based sol-gel samples are fabricated by sol-gel composite method automatic spray coating. First, the BiT piezoelectric powder and each sol-gel solution were mixed in a ball mill for about 1 day to obtain clay suitable for the spray coating process. Spraying coating was performed on titanium substrate having a thickness of 3mm. Titanium substrate has heat capacity and high durability.



Fig. 1 Image of BiT/TO+Sr with gold electrode on 3mm titanium substrate

After the spray coating process, the samples were dried at 150°C and fired for 5 minutes each. Spray coating, drying, and firing were repeated until the film thickness reached 50µm. Preparing the sample, poling process was performed at room temperature. BiT/BiT was first heated at 400°C in furnace, and after being sufficiently heated, electric field due to corona discharge was applied to perform poling. The next BiT/TO+Sr was poled by corona discharge at room temperature, it was after forming a piezoelectric film by spraying, and was not heated in a furnace. The output voltage of the power supply was about 40kV. The poling time was 5 minutes and the distance between the corona discharge needle and the sample was 30 mm. Gold was used for the upper electrode. Gold electrode made by spattering system, the vacuum was conducted down to 4.3×10^{-4} Pa and the sputtering pressure 1.3Pa. Fig. 1 shows image of BiT/TO+Sr sample with gold electrode on 3mm titanium substrate.

3. Experimental results

The measurement method was to set the BiT samples in an electric furnace and measure the characteristics of the sample by connecting the sample and the LCR meter through a platinum wire. Since the poling temperature of BiT/BiT is 400°C, the electrical properties were measured from room temperature up to 400°C in increments of 50°C.



Fig. 2 Resistance of BiT samples after poling fabricated on a titanium substrate up to 400°C



Fig. 3 Capacitance of BiT samples after poling fabricated on a titanium substrate up to 400°C

In addition, the temperature on the graph is a record of the temperature of the substrate with thermocouple, and does not indicate the setting temperature of the furnace. Fig. 2 and Fig. 3 shows the transition of resistance and capacitance of BiT-based samples. Fig. 2 shows BiT/BiT sample's resistance is highest value in this measurement. It found that the resistance of TO+Sr was higher than that of BiT, because this result showed that changing the BiT sol-gel solution to TO+Sr solution significantly decreased the resistance. Fig. 3 shows BiT/TO+Sr (400°C fired) have highest capacitance at room temperature in this result, and BiT/TO+Sr 200°C fired sample have also higher capacitance peak than BiT/BiT at room temperature.

4. Conclusion

In this study, BiT/TO+Sr was prepared by the sol-gel composite method, and the electrical properties were compared with the BiT/BiT sample prepared in the same sol-gel composite method. BiT/BiT was poled at 400°C and BiT/TO+Sr can be poled at room temperature by corona discharge. After forming the gold electrode on the sol-gel composite, the resistance and capacitance were measured from room temperature to 400°C.

In this study, the reason why BiT/TO+Sr could be poled at room temperature is considered to be related to the resistance. From the resistance measurement, it was found that the resistance of the BiT composite decreased by changing the sol-gel solution to TO+Sr, indicating that the resistance of TO + Sr is lower than that of BiT. It is thought that the decrease in the resistance of the sol-gel solution layer caused the electric field to concentrate on the BiT piezoelectric layer during poling, making it easier to poled. In addition, the use of TO+Sr also increased the capacitance, and it is considered that the poling became easier from the viewpoint of the dielectric constant.

Future research should investigate other electric properties, piezoelectric properties and changes in the crystal structure to elucidate the further mechanism which BiT sol-gel composite was able to reduce the poling temperature by using the TO+Sr sol-gel solution.

References

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