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Study on on-site fabrication of $Bi_4Ti_3O_{12}/TiO_2+SrCO_3$ by a rubber heater

Mako Nakamura[1‡], Takeshi Hamada[1], Ryota Ono[1], Kei Nakatsuma[1], and Makiko Kobayashi[1*]
([1]Kumamoto Univ.)

1. Introduction

In recent years, ultrasonic waves have been widely used as Non-destructive testing (NDT) tools in industrial fields. In the conventional way of NDT, ultrasonic waves, backing material, and couplant material are used. The backing material absorbs vibrations in the back side of the piezoelectric film. The couplant material improves the acoustic junction between the measurement object and the transducer. However, they cannot withstand high environments, so ultrasonic NDT was difficult in high-temperature environments. Therefore, sol-gel composite ultrasonic transducers used in high-temperature environments were developed[1,2]. These films have many pores that substitute for backing materials and can be directly attached to measurement objects without couplant.

We are studying the on-site fabrication of sol-gel composite piezoelectric films for direct attachment to large and immovable pipes in power plants. PZT($Pb(Zr,Ti)O_3$)), which is a common piezoelectric material, has to be fired at 650°C. And it conventionally requires extensive equipment such as an electric furnace. PZT also contains toxic lead. Therefore, we proposed a firing method using a rubber heater in direct contact with the film[3,4]. In this process, $Bi_4Ti_3O_{12}/TiO_2+SrCO_3$ (BiT/TO+Sr), which can be fired at a low temperature (200°C) and is lead-free, was used[5,6]. In this study, the proposed firing method and conventional furnace firing method were compared.

2. Sample fabrication

As the sol-gel composite, BiT ceramic powder and TO+Sr sol-gel solution were mixed for 24 hours by a ball milling machine. The BiT/TO+Sr was sprayed onto 30mm × 30mm × 3mm titanium substrates which were prepared in six samples. After spraying, the piezoelectric films were dried at room temperature and 150°C for 5 minutes each. And then, they were fired at 200°C for 5 minutes. Three samples of the six were fired by an electric furnace as the conventional method (**Fig. 1(a)**). And the others were fired by the rubber heater. BiT/TO+Sr films were directly contacted with the rubber heater (**Fig. 1(b)**) and adhered to the rubber heater by weight. The temperature of the rubber heater was controlled at 200°C by a temperature controller. This process from the spraying to firing was repeated until the film thickness reached 50µm. After the fabrication of the films, the BiT/TO+Sr films were poled with positive corona discharge at less than 20% humidity. The distance from the tip of the electrode needle to the surface of the BiT/TO+Sr piezoelectric film was fixed at approximately 3cm, and the DC power supply voltage was approximately 40 kV. After poling, Ag paste was attached to the surfaces of the films. The optical image of one of the three completed samples is shown in **Fig. 2**.

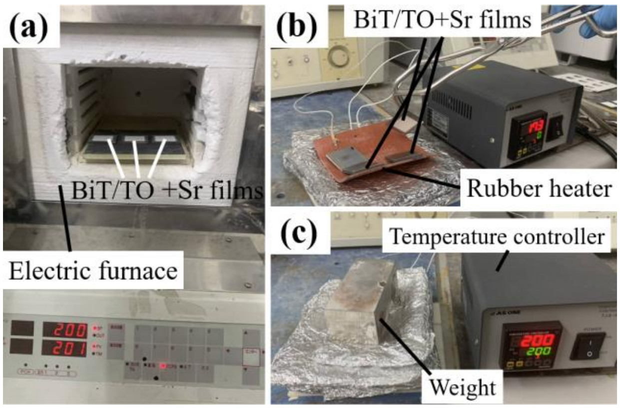

Fig. 1 Firing methods: (a) Firng by electric furnace, (b) Place films on a rubber heater, (c) Firing with a weight

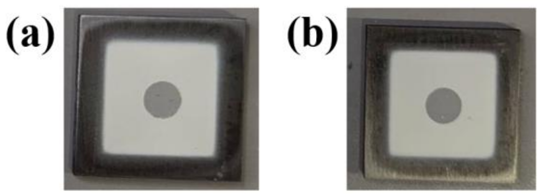

Fig. 2 One of three completed samples: (a) Fired by a rubber heater, (b) Fired by an electric furnace

3. Results and discussion

The performance evaluation of the films was investigated in terms of piezoelectric constant (d_{33}), ultrasonic reflected wave, and sensitivity.

E-mail: ‡242d9364@st.kumamoto-u.ac.jp,
*kobayashi@cs.kumamoto-u.ac.jp

Fig. 3 shows the results of d_{33} and the error bars indicate the variations. The values of d_{33} were obtained by taking the average of three for each. The sensors of the rubber heater had slightly lower values. However, the variance was smaller and the performance was more stable.

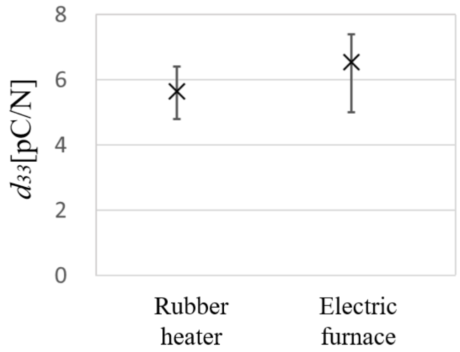

Fig. 3 The averages of d_{33} and the variations for the rubber heater's sensors and electric furnace's sensors

As for the ultrasonic reflected waves, those were obtained to connect the pulser/receiver, oscilloscope, and fabricated samples. From those waves, the sensitivities were calculated using the following equation.

$$Sensitivity = -(20\log(V_1/V_2) + Gain) \qquad (1)$$

The reference amplitude V_1 was set to 0.1V, and V_2 was set to $V_{P\text{-}P}$ of the first reflected wave from the bottom of the substrate. **Fig. 4** shows the results of the reflected waves. It was found that reflected waves were clear at both heater firing and firing. The results of the sensitivities are shown in **Fig. 5**. The sensitivities show the averages of each three and the error bars indicate the variations. The sensitivity of the rubber heater was about 5dB lower than that of the electric furnace. As the theory suggests, this magnitude relation of sensitivity was equal to that of d_{33}. There was a sample with extremely low sensitivity and d_{33}, resulting in low average values for heater firing. This can be attributed to the temperature bias on the surface of the rubber heater.

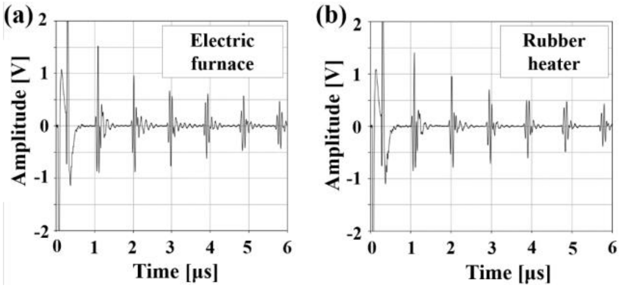

Fig. 4 The reflected waves that were the most sensitive of the three, respectively

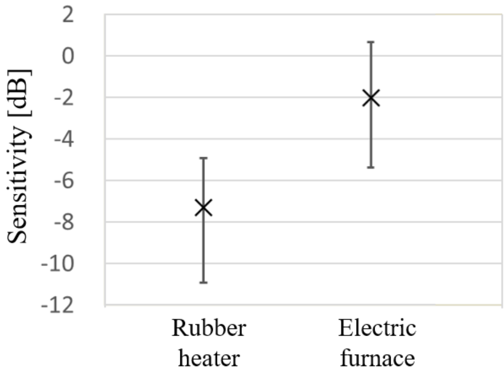

Fig. 5 The average of sensitivities and the variations for the rubber heater's sensors and the electric furnace's sensors

4. Conclusion

This study proposed on-site fabrication of sol-gel composite piezoelectric films to enable direct application to large objects such as power plants. In particular, we focused on the on-site firing of the films using the rubber heater. To evaluate the performance of sensors fabricated by directly attached firing of rubber heater, three samples prepared by rubber heater firing and those by electric furnace firing were compared.

From the results of the reflected waves, the sensors fabricated by the rubber heater had sufficient measurement capabilities. However, the d_{33} and sensitivity of the sensor by the rubber heater were lower than that of the electric furnace. One of the causes was that a sample with insufficient crystallization was produced due to bias in the surface temperatures of the rubber heaters. In the future, we expect to conduct a cross-sectional analysis using SEM to verify the results.

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