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Polarization Condition Optimization of $CaBi_4Ti_4O_{15}$/ $Pb(Zr,Ti)O_3$ Sol-Gel Composite membrane using pulsed power supply

パルス電源を用いた CBT/PZT ゾルゲル複合体膜の分極条件最適化に関する研究

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1. Introduction

Safety of social infrastructure is an important issue. Especially in chemical plants, there are many high temperature structures and the demand for non-destructive inspection (NDI) in operating condition to avoid serious accidents. One of the main NDI methods is ultrasound NDI using an ultrasonic transducer. $Pb(Zr,Ti)O_3$ (PZT) material is usually used as ultrasonic transducers because of high piezoelectricity and relatively high Curie temperature. However, Since chemical plants in operation may reach high temperatures such as 400°C, it is still difficult to use in operation condition, thus it is normally inspected at room temperature during shutdown and development of ultrasonic transducers that can be used at high temperatures has been desired.

High-temperature ultrasonic transducers using sol-gel composite materials have been developed to improve the thermal resistance.[1-4)] Mn-doped $CaBi_4Ti_4O_{15}$ (CBT) powder and PZT sol-gel solution combination, CBT/PZT exhibited continuous high temperature durability up to 600°C[3)]. Since Mn-doped CBT has a high coercive field, it needs to be poled at high temperature, and heating and cooling time are necessary for poling process. If Mn-doped CBT/PZT can be poled at room temperature (RT), it is expected to reduce manufacturing cost, make on-site fabrication easier, and improve practicality.

In past studies, RT poling of CBT/PZT using pulse voltage discharge attemped[4),] and as a result, RT poling of CBT/PZT itself was succeeded. However, the signal-to-noise ratio (SNR) at high temperature was not satisfactory. It was thought that the poling condition was not optimized yet. In this study, the poling needle shape differenct was investigated empirically.

2. Fabrication of Mn-doped CBT/PZT films

Mn-doped CBT/PZT sol-gel composite films were fabricated on titanium substrates with 3mm thickness, 30mm width, and 30mm length by a sol-gel spray technique. The PZT sol-gel solution and the Mn-doped CBT piezoelectric powder were mixed. In order to achieve adequate viscosity of the mixture, ball milling was carried out for more than 1 day. After spraying the mixture on titanium substrates, the samples were dried at 150°C for 5 minutes and then sintered at 650°C for 5 minutes. The spray coating and heating process were repeated until the target film thickness was achieved. In this study, the target thickness was set as 50μm. Thereafter, platinum top electrode with 1cm diameter were manufactured using commercially available platinum paste. **Fig. 1** shows an optical image of the Mn-doped CBT/PZT film with platinum paste top electrode on the 3mm thick titanium substrate.

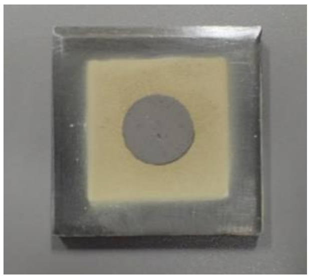

Fig. 1 50-μm-thick Mn-doped CBT/PZT film with 1cm diameter platinum paste top electrode on 3mm thick titanium substrate.

3. Poling process

In this research, an attempt was made to optimize the RT polling condition. By using a newly developed pulse voltage source device, arc discharge was suppressed by supplying higher voltage and lower current than previous machine. Two types of needles as shown in **Fig. 2** were used for RT poling process. Traditional one in left has a flat end whereas the new needle has higher curvature radius. The distance between the needle and the film surface was 2.0cm and 40kV peak pulse voltage was supplied.

Fig. 2 The shape of poling needle, conventional needle (left) and new needle (right).

4. Experimental results

In order to evaluate the SNR at high temperature, ultrasonic measurements were carried out between RT and 600 °C in pulse echo mode. The samples poled by pulse voltage discharge with different needles were put into a furnace. Platinum wires were used as electrical cable and ceramic weight was used to maintain the electrical connection between the cables and the top electrode and ground. Measurement data was recorded by a digital oscilloscope. Ultrasonic response at 600 °C of CBT/PZT using the conventional poling needle is shown in **Fig. 3**.[5] As it was mentioned above, SNR was significantly deteriorated. Ultrasonic response at 600 °C of Mn-doped CBT/PZT using the new poling needle is shown in **Fig. 4**. SNR was much higher than previous result and it was confirmed that high curvature radius of poling needle was effective to pole sol-gel composite with high coercive field powder phase.

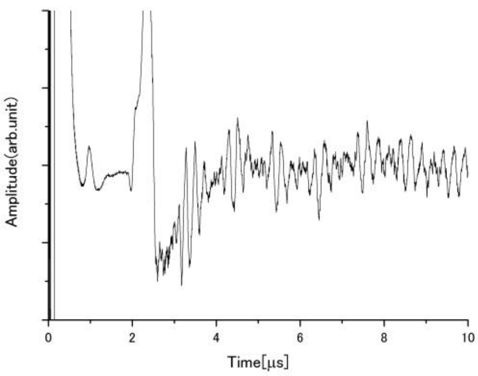

Fig.3 Ultrasonic response of CBT/PZT transducer on 3-mm thick titanium substrate at 600 °C poled with the conventional needle.

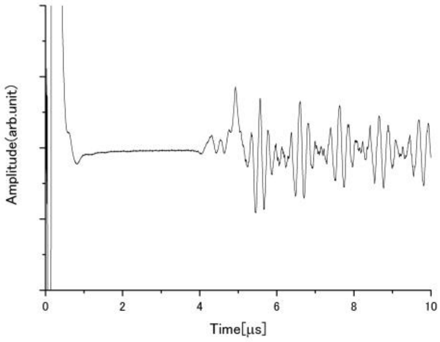

Fig. 4 Ultrasonic response of CBT/PZT transducer on 3-mm thick titanium substrate at 600 °C poled with the higher curvature radius needle

5. Conclusions

Poling optimization of Mn-doped CBT/PZT by pulse voltage source was attempt to improve SNR at 600 ° C. It was found that the curvature radius of the poling needle is also very important parameter for poling. Further experiments will be carried out to improve the signal strength by optimizing poling conditions, such as the needle curvature radius, the distance between the needle and the platinum top electrode, and poling environments in the near future.

References

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