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Frequency characteristics of $LiNbO_3/TiO_2+SrCO_3$ sol-gel composite ultrasonic transducer at high temperatures

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1. Introduction

In recent years, Non-Detective Testing (NDT) using ultrasonic waves has been widely used in the industrial field. However, conventional ultrasonic transducers require a backing material and couplant. So, they cannot be used in high-temperature environments. Therefore, we are developing an ultrasonic transducer that can be used at high temperatures using the sol-gel composite method.[1,2) In this method, a piezoelectric film can be fabricated directly on the object to be measured without a couplant. In addition, the sol-gel composite piezoelectric film has a large number of pores, which can replace the backing material, and can be used at a temperature that depends solely on the performance of the piezoelectric film.

In previous studies, various sol-gel composite ultrasonic transducers based on $Pb(Zr,Ti)O_3$, $Bi_4Ti_3O_{12}$, and $LiNbO_3$ (LN) have been fabricated.[3,4)] Among them, LN/TiO_2 (TO)+$SrCO_3$ (Sr) has shown great performance: lead-free, poling at low temperatures, and stable operation at 700 °C.[5)] In addition, 70 µm thickness LN/TO+Sr could be observed with a cylindrical defect up to 830 °C.[6)] However, although the Curie temperature of LN is approximately 1200 °C, the ultrasonic response decreases from approximately 700 °. It is probably due to the attenuation of the high-frequency component of the ultrasonic response and the degradation of the Pt electrode.

In this study, 100 µm LN/TO+Sr piezoelectric films were fabricated on the object to be measured to investigate the change in the frequency response and its effect on the ultrasonic response.

2. Sample Fabrication

First, LN piezoelectric powder and TO+Sr sol-gel solution were mixed using a ball mill machine for 24 hours. The mixed sol-gel composite solution was coated onto the object to be measured using an automatic spray machine. Subsequently, the piezoelectric films were dried (150 °C) and annealed (400 °C) for 5 minutes respectively. The process, from spray coating to annealing, was repeated 13 times to achieve a film thickness of 100 µm. In this study, Inconel blocks of 30 mm length, 30 mm width, and 25 mm thickness were used for the object to be measured. The block also had a cylindrical defect with a diameter of 2 mm and a depth of 20 mm.

The piezoelectric film was then poled using positive corona discharge. The fabricated samples were heated in an electric furnace at 200 °C for 10 minutes and then poled for 5 minutes. The DC power supply voltage used for poling was approximately 40 kV. The humidity during poling was maintained at less than 20 %. After poling the piezoelectric constant d_{33} was 4.8 pC/N.

Finally, the top electrode was fabricated using the sputtering method. Pt was used as the electrode material. The current during electrode fabrication was 20 mA, and sputtering was performed for 25 minutes. Ar was used for the atmosphere gas, and the atmospheric pressure was set to 1.3 Pa. The optical image of the completed sample is shown in **Fig. 1**.

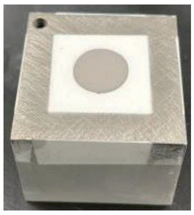

Fig. 1 Optical image of the completed LN/TO+Sr ultrasonic transducer

3. Experimental Method

The pulser/receiver, oscilloscope, and fabricated samples were connected and the samples were heated in an electric furnace. The ultrasonic response was measured at RT, 50 °C, and 100 °C in that order, up to 700 °C, and this measurement was repeated four times for the same sample. Subsequently, the same measurements were performed without an upper limit to determine the highest temperature at which the ultrasonic response of the cylindrical defect could be measured. The sensitivity of the ultrasonic response was calculated using Equation (1).

$$Sensitivity = -(20\log V_1/V_2 + P/R\ Gain) \quad (1)$$

The reference amplitude V_1 was set to 0.1 V, and V_2 was set to $V_{P\text{-}P}$ of the 1st reflected wave from the bottom of the substrate. Finally, a Fast Fourie Transform (FFT) was performed on the same waves, and the frequency response was measured.

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4. Result

Fig. 2 shows the ultrasonic response in the 5th thermal cycle test. The ultrasonic response from the defect could be observed up to 1050 °C, although the SNR decreased compared with that at RT owing to a decrease in sensitivity. The shape of the ultrasonic response also changed. At over 1050 °C, ultrasonic response from the defect could not be observed. **Fig. 3** shows the sensitivity transition of the ultrasonic response from the 1st to the 5th cycle in the thermal cycling test. The sensitivity transition was consistent up to 700 °C, indicating stable operation. However, in the 5th cycle, the sensitivity decreases at 800 °C.

A comparison of the ultrasonic response with previous studies showed improved sensitivity.[6] It is considered that the increased film thickness changed the impedance matching with the measurement equipment and the attenuation of the ultrasonic response by the substrate, resulting in maximum sensitivity at 800 °C.

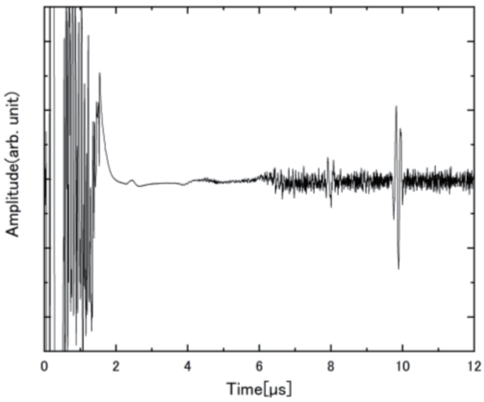

Fig. 2 Ultrasonic response at 1050 ℃ (5th cycle)

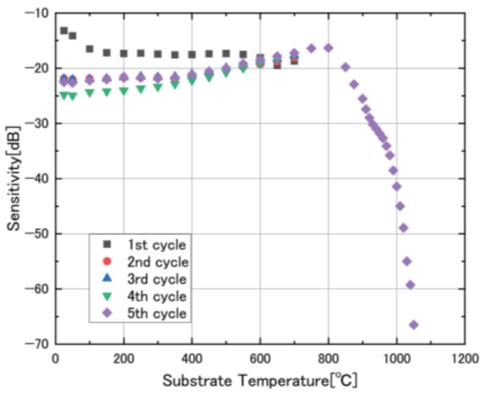

Fig. 3 Sensitivity transition (1st - 5th cycle)

Table I FFT characteristics (5th cycle)

Temp.	f_L[MHz]	f_H[MHz]	f_C[MHz]	BW[MHz]	FBW
RT	5.4	8.3	6.9	2.9	0.43
500	5.5	8.3	6.9	2.8	0.41
700	5.4	8.1	6.7	2.7	0.41
900	5.9	8.9	7.4	3.1	0.42
1050	5.0	10.2	7.6	5.2	0.69

Table I shows the FFT results obtained at various temperatures. Under 700 °C, there were no significant changes in the center frequency (f_C), high (f_H), and low (f_L) cut-off frequency. However, after 900 °C, the -6 dB bandwidth increased, indicating an increase in the fractional bandwidth (FBW). A similar phenomenon was reported at the same temperature in a previous study, suggesting an effect of the Pt electrode. However, when the film thickness was 50 μm, the f_C was approximately 10 MHz, whereas in this study, it was reduced to approximately 7 MHz under 700 °C.[6]

5. Conclusion

In this study, 100 μm LN/TO+Sr ultrasonic transducers were fabricated on Inconel blocks to investigate the change in the frequency response of the ultrasonic response with increasing film thickness. As a result, ultrasonic responses from the defects were observed up to 1050 °C. The sensitivity of the ultrasonic response reached a maximum of 800 °C owing to the change in impedance with increasing film thickness.

The FFT results showed no significant changes in the f_C and -6 dB bandwidth under 700 °C. However, over 900 °C, the shape of the ultrasonic response changed and the -6 dB bandwidth and FBW increased. The f_C was approximately 7 MHz, which was smaller than in previous studies. Therefore, the ultrasonic response could be observed at 1050 °C. In contrast, the sensitivity decreases over 800 °C, and a lower frequency ultrasonic response is required.

In addition, the FFT results possibly included the effect of the Pt electrode. Future research will remove this effect and improve sensitivity by optimizing electrode surface area and increasing film thickness.

References

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