Low frequency ultrasonic transducer fabrication in sol-gel composites

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Once polarized

1. Introduction

In recent years, Non-Destructive Testing (NDT) using ultrasonic waves has been widely used in the industrial fields. However, conventional ultrasonic transducers cannot be used in high-temperature environments because they require backing material and couplant. Therefore, we have been developing ultrasonic transducers that can be used in high-temperature environments using the sol-gel composite method.^{1,2}) Since the sol-gel composite piezoelectric film has a large number of pores, it can be used in place of a backing material and can be used at a temperature that depend on the performance of the piezoelectric film.

In previous studies, various sol-gel composite ultrasonic transducers based on Pb(Zr,Ti)O₃, Bi₄Ti₃O₁₂, and LiNbO₃ (LN) have been fabricated.^{3,4)} Among them, LN/TiO₂ (TO)+SrCO₃ (Sr) is lead-free, poling at low temperatures, and stable operation at 700°C.⁵⁾ In NDT, sensors with a center frequency of about 5MHz are generally required. However, the 50 µm thickness LN/TO+Sr had a center frequency of approximately 11MHz.^{5,6)} In order to lower the frequency, the film thickness was increased to 100µm and its evaluation was conducted. However, the 100µm sample had poor sensitivity and distorted the ultrasonic response, thereby failing to obtain an accurate center frequency.

In this study, we focused on the number of polarizations. Increasing the number of polarization times may increase the degree of polarization and improve sensitivity. LN/TO+Sr piezoelectric films of 100 μ m thickness were fabricated in the object, which were polarized twice, one at 50 μ m and the other at 100 μ m. The frequency characteristics and ultrasonic response were then observed.

2. Sample Fabrication

First, LN piezoelectric powder and TO+Sr solgel solution were mixed for 24h using a ball mill machine. The mixed sol-gel composite solution was applied to the object to be measured using an automatic sprayer at humidity below 20%. The piezoelectric film was then dried at 150°C for 5min and annealed at 400°C for 5min. The process from spray application to annealing was repeated until the target film thickness was reached. In this study, an Inconel plate of 30mm length, 30mm width, and 3mm thickness ware used for the measurement.

The piezoelectric film was then polled using positive corona discharge. The fabricated samples were heated in an electric furnace at 200°C for 10min and then polled for 5min. The humidity during poling was maintained below 20%. **Fig. 1** shows the change in the fabrication process according to the different number of polarizations.



Fig.1 Fabrication process (one or two poling)

Finally, the top electrode was fabricated by sputtering. Pt was used as the electrode material. The current during electrode fabrication was 20mA, and sputtering was performed for 25min. Ar was used as the atmosphere gas, and the atmospheric pressure was 1.3Pa. In this experiment, the waveform of the ultrasonic response was observed using silver paste instead of the top electrode, because the top electrode of the ultrasonic transducer with two separate polarizations could not be fabricated successfully. **Fig. 2** shows the Optical image of the completed LN/TO+Sr ultrasonic transducer with two poling process.



Fig.2 Optical image of the complete LN/TO+Sr ultrasonic transducer with two poling process

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3. Experiment Method

The ultrasonic response was observed by connecting the pulser/receiver, oscilloscope, and fabricated samples. The sensitivity of the ultrasonic response was calculated using Equation (1).

$$Sensitivity = -(20logV_1/V_2 + P/RGain) \quad (1)$$

The reference amplitude V_1 was set to 0.1V, and V_2 was set to V_{P-P} of the first reflected wave from the bottom of the substrate.

4. Result

Fig. 3 shows the ultrasonic response at RT of an ultrasonic transducer with only one polarization and **Fig. 4** shows the ultrasonic response of a sample with two polarizations. The one poling sample had a pulser/receiver gain of 40dB, whereas the two poling sample had a gain of 25dB.

The one poling sample had a sensitivity of -20.96dB at RT, whereas the two poling sample had a sensitivity of -15.85dB. Despite the fact that the top electrode could not be made properly and silver paste was used instead of the Pt electrode, the two poling sample showed improved sensitivity and a clear ultrasonic response. It is possible that the performance of the ultrasonic transducer could be improved by polarizing it twice. The two poling one did not initially produce a clear ultrasonic response. To solidify the top electrode, the temperature was raised to 1000°C for 5h for annealing. As a result, the top electrode could not hold up and silver paste had to be used. However, it shows the high-temperature durability of the LN/TO+Sr ultrasonic transducer, which can be used at temperatures as high as 1000°C.



Fig.3 Ultrasonic response of $100\mu m$ thick LN/TO+Sr fabricated on 3mm thick Inconel substrate with one poling process



Fig.4 Ultrasonic response of 100µm thick LN/TO+Sr fabricated on 3mm thick Inconel substrate with two poling process

5. Conclusion

In this experiment, 100μ m LN/TO+Sr ultrasonic transducers were fabricated with one and two poling process. As a result, the ultrasonic response of the two poling sample was confirmed. In addition, the waveform could be obtained even with silver paste, and the transducer was able to endure 1000°C. This indicates that the performance may be improved by dividing the polarization. In the future, samples with two poling process will be fabricated again with a new substrate, taking into consideration the decrease in sensitivity due to degradation of the substrate. We will then evaluate the changes that occur with different numbers of polarizations based on sensitivity and FFT results.

References

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