

## Study of High Temperature Ultrasonic Transducer in LiNbO<sub>3</sub> Based Sol-gel Composite

LiNbO<sub>3</sub> ゴルゲル複合体における超音波トランスデューサに関する研究

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### 1. Introduction

Non-destructive testing (NDT) widely used for industrial fields to detect early stage small defects before fatal failure. One of the main nondestructive inspection methods is ultrasound using an ultrasonic transducer. Compared with conventional ultrasonic sensors, no coupling agent nor backing material is required, so ultrasonic NDT can be performed at high temperature during operation. Non-destructive inspection during high-temperature operation has been desired to assure safety and reduce economical loss during shut-down.

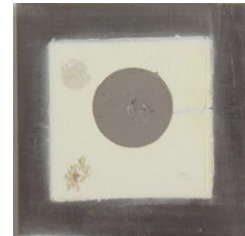
In the past studies, the high temperature performance of LiNbO<sub>3</sub>/Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> (LN/BiT) composites was showed a clear reflected wave at 700°C, it showed the possibility of nondestructive inspection at high temperature<sup>[1-2]</sup>. However, LN/BiT required a high temperature of 900°C during poling process of sample fabrication. Therefore, it was very difficult to manufacture. There are several reports that LN/Pb(Zr,Ti)O<sub>3</sub> (LN/PZT) was able to conduct poling process at 550°C and showed a good SNR<sup>[3-5]</sup>. Since the LN/PZT sol-gel composite was coated by the manual spray method, individual differences were created in the manufactured samples, and their samples didn't compare with LN/BiT.

In this study, LN/PZT ultrasonic transducers by sol-gel automatic spray technique on 3mm thick titanium substrates were fabricated and conducted the thermal cycle testing and long-term test in order to carry out sensitivity comparison between LN/BiT and LN/PZT. At a substrate temperature of 700°C, the thermal cycle test was performed 3 times, and the long-term durability test was performed for 13 days.

### 2. Fabrication sol-gel composite samples

LN/PZT samples were manufactured by sol-gel spray technique. LN/PZT sol-gel composite was prepared by mixing LN piezoelectric powders and PZT sol-gel solution by a ball mill machine for

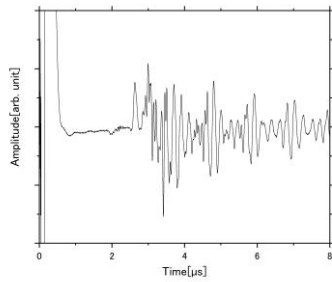
about 1day. The thoroughly mixed solution was coated onto a 3mm thick titanium substrate by spray method. The spray-coated sample was dried at 150°C and calcined at 650°C for 5minutes each. These processes were repeated to produce a LN/PZT sample having a film thickness of 50μm. After their processes, LN/PZT sample was subjected to poling treatment. Poling was performed by applying a corona discharge to a sample from 575°C to room temperature conditions and on a heated substrate to make it hard to cool. Finally, an electrode having a diameter of 1 cm was formed on the sample by using platinum paste. The optical image of LN/PZT on titanium substrate sample is shown in **Fig. 1**.



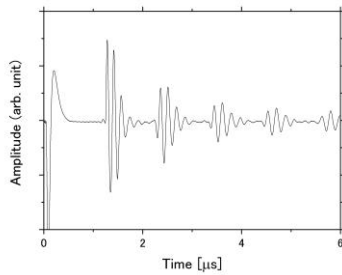
**Fig.1** Optical image of LN/PZT film fabricated on 3mm thick titanium substrate.

### 3. Experimental results

First, three thermal cycles of LN/PZT were performed. The ultrasonic response was measured at each temperature while increasing the substrate temperature that was measured by thermocouple from room temperature by 700°C. After 5min holding time at each temperature, ultrasonic response was recorded by a digital oscilloscope up to 700°C. The ultrasonic response of LN/PZT at 700°C in the third cycle is shown in **Fig. 2**. **Fig. 3** shows the LN/BiT signal waveform at 700°C in third cycle. From Fig. 2 and Fig. 3, the ultrasonic response is clearer for LN/BiT.



**Fig.2** Ultrasonic response of LN/PZT on titanium substrate at 700°C in the third cycle

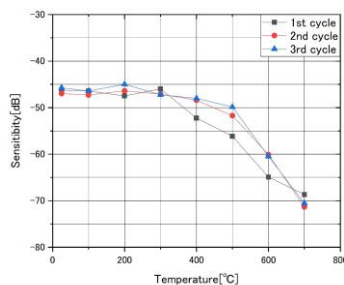


**Fig.3** Ultrasonic response of LN/BiT on Inconel substrate at 700°C in the third cycle

**Fig. 4** shows sensitivity comparison results of the thermal cycle test LN/PZT. The sensitivity was calculated as following;

$$\text{Sensitivity} = - (20 \log_{10} V_1 / V_2 + \text{gain of P/R}) \text{ (dB)} \quad (1)$$

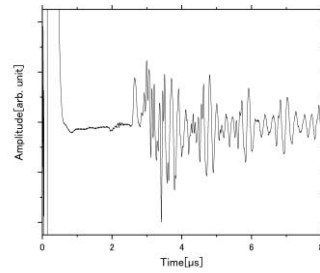
where  $V_1$  is the ideal amplitude, 0.1 (V) in this experiment,  $V_2$  is the amplitude (V) of the second reflected echo from the bottom surface of the substrate. From Fig.4, the sensitivity was reduced.



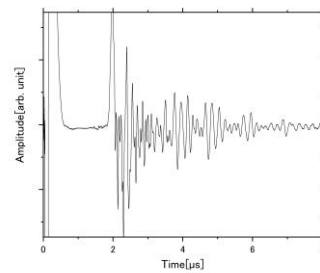
**Fig.4** Thermal cycle test results of LN/PZT.

Next, a long-term durability test of LN/PZT was performed at 700°C for 13 days. The ultrasonic response that heating starts at 700°C is shown in **Fig. 5**. Ultrasonic response continued heating for 13 days is shown in **Fig. 6**. Through long-term durability testing, LN/PZT was showed the deterioration of signal and low sensitivity. It is

considered that the titanium substrate deteriorated.



**Fig.5** Ultrasonic response of LN/PZT at 700°C after 0h.



**Fig.6** Ultrasonic response of LN/PZT continued heating at 700°C after 13 days.

#### 4. Conclusions

LN/PZT sol-gel composites were fabricated on 3mm thick titanium. Throughout the thermal cycle test and long-term testing, LN/PZT was showed the deterioration of signal and low sensitivity. LN/PZT was not able to obtain sufficient SNR and was not showed sufficient sensitivity at high temperature. It is considered that the titanium substrate deteriorates at high temperature and affects the signal. From these, it is necessary to manufacture the LN/PZT sol-gel composite by the automatic spray method on the Inconel substrate that does not cause the deterioration of the substrate and measure its accurate performance.

#### References

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