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# Fabrication of $LiNbO_3$-based Sol-Gel Composite at Low-Temperature

低温下における $LiNbO_3$ ベースゾルゲル複合体の作製


Naoki Kambayashi[1‡], Hiroaki Akatsuka[1], Kohei Hirakawa[1] , Kei Nakatsuma[1], and Makiko Kobayashi[1] ([1]Kumamoto Univ.)

神林尚樹 [1‡]，赤塚大晃 [1]， 平川康平 [1]，中妻啓 [1]，小林牧子 [1]（[1]熊本大学）


## 1. Introduction

Ultrasound is often used as a means of non-destructive testing. Piezoelectric ceramics are commonly used to generate ultrasonic waves. In order to perform NDT in a high temperature environment, piezoelectric ceramics, couplant, and backing material that can withstand the high temperature are required. The piezoelectric ceramics fabricated by the sol-gel composite method do not require a couplant or backing material, and the piezoelectric ceramics themselves have high temperature durability.

In the previous studies using $LiNbO_3$ (LN) as powder, $Pb(Zr,Ti)O_3$ (PZT) and $Bi_4Ti_3O_{12}$ (BiT) were used as sol-gel solution.However, each of these fabrication processes requires high temperature treatment above 650°C, which increases the difficulty of fabrication. [1-2]. In addition, there was a report on the use of $TiO_2$ (TO) sol-gel solution for BiT-based transducers to enable low temperature fabrication, and the addition of Sr to the sol-gel solution improved the performance at low temperature fabrication. [3-4].

In this study, the heat treatment was performed below 200°C in the fabrication of LN/TO+Sr sol-gel composites. Fabrication of piezoelectric ceramics on Inconel substrate by repeated spraying of solution, drying and calcined. Next, thermal cycling tests and high temeture test were conducted to measure ultrasonic response.

## 2. Fabrication of $LiNbO_3$-based transducer

The LN/TO+Sr piezoelectric ceramics were made of LN piezoelectric powder and TO+Sr sol-gel solution. These two were mixed using a ball mill for about 24 hours. After mixing, the solution was sprayed onto a 3 mm Inconel substrate and dried at 150°C and calcined at 200°C for each 5 minutes. This process is repeated until a film thickness of 50μm is reached. For the poling process, after heating the sample at 200°C, the poling was done using corona discharge. Finally, electrodes are formed on the sample surface using platinum paste. **Fig. 1** shows an image of the sample after the electrodes were made.

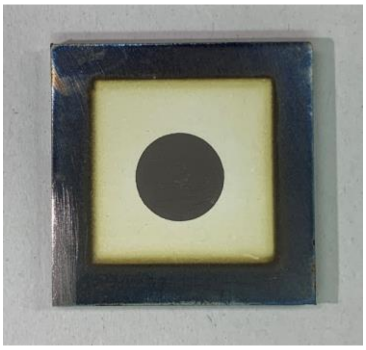

Fig. 1 Image of LN/TO+Sr ceramic with platinum paste on 3mm thick Inconel substarate

## 3. Experimental results

A thermal cycling test of LN/TO+Sr was conducted. The LN/TO+Sr was heated from room temperature to 700°C, and the ultrasonic response was measured with a digital oscilloscope at every 100°C rise, while the substrate temperature was monitored with thermocouples. After the temperature rises to 700°C, cool the substrate until it returns to room temperature sufficiently, and then conduct the next test. This is done three times. The temperature of the furnace when it reaches 700°C each cycle is 810°C, 820°C, and 820°C. Fig. 2 shows the ultrasonic response at 700°C for the third cycle, and Fig. 3 shows sensitivity obtained from the cycling test. Sensitivity(SENS) was calculated by the following equation

$$\mathrm{SENS} = -\left(20\log_{10}\frac{V_2}{V_1} + GAIN \text{ of } \mathrm{P/R}\right) \qquad (1)$$

where $V_1$ is the amplitude of the reference ultrasonic waveform, which in this experiment is 0.1V. $V_2$ is the amplitude of the second reflection echo from the bottom surface. Sensitivity after 600°C in the 2nd cycle is greatly attenuated. The reason is the filter is more applied because the noise becomes large.

Next, the high temperature test was conducted. **Fig. 4** shows sensitivity obtained high temperature test**. Fig. 5** shows the response substrate temperature is 896°C and furnace temperature is 980°C. And it showed the same heat resistance as LN/BiT. The result is discontinuous from the sensitivity exceeding 800°C in **Fig. 4**. It is because the setting of the pulsar receiver was changed.

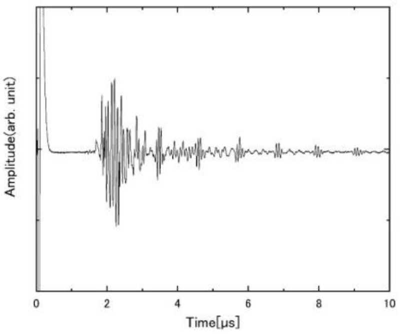


Fig. 2 Ultrasonic response of LN/TO+Sr at substrate temperature 702℃, furnace temperature 820℃ in the 3rd cycle

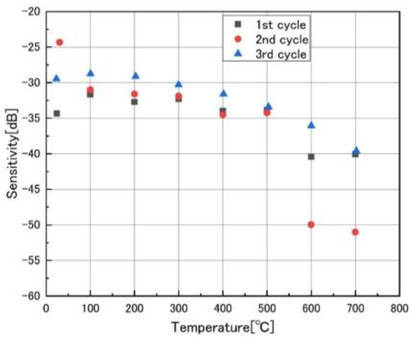


Fig. 3 Sensitivity of LN/TO+Sr in cycling test

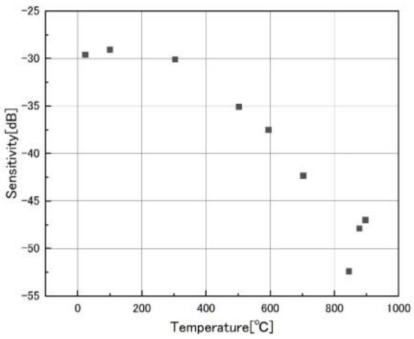


Fig. 4 Sensitivity of LN/TO+Sr up to substrate temperature 896℃

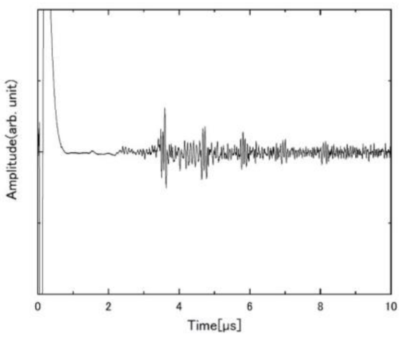


Fig. 5 Ultrasonic response of LN/TO+Sr at substrate temperature of 896℃.

**4.Conclusion**

In this study, LN/TO+Sr sol-gel composites were fabricated on Inconel substrates. In the fabrication process, the calcining temperature and the poling process were conducted at 200°C. Through the high temperature test and thermal cycling test, it was shown that the LN/TO+Sr sol-gel composite could be fabricated at lower temperature than LN/PZT and LN/BiT. It showed the same heat resistance as LN/BiT. In the next study, it is necessary to confirm whether the performance can be maintained in the long term under high temperature conditions. And the fabrication of LN-based transducers under room temperature conditions will also be studied.

**References**


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