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Poling of $Pb(Zr,Ti)O_3$/$Pb(Zr,Ti)O_3$ by negative corona discharge

負のコロナ放電による $Pb(Zr,Ti)O_3$/$Pb(Zr,Ti)O_3$ の分極

Minori Furukawa[1†],Shohei Nozawa[1],Takumi Hara[1],Kei Nakatsuma[1],and Makiko Kobayashi[1]([1]Kumamoto Univ.)

古川美徳 [1†],野澤勝平 [1],原拓未 [1],中妻啓 [1],小林牧子 [1] ([1]熊本大)

1. Introduction

The sol-gel composite is a technique for producing a crack-free thick film by mixing a powder and a sol-gel solution and performing a heat treatment. Ultrasonic transducers based on sol-gel composites are expected to be put to practical use for industrial applications due to their excellent heat resistance, curved surface compatibility, and the absence of a backing material. PZT/PZT using $Pb(Zr,Ti)O_3$ (PZT) is a frequently used material because of its easy poling and high piezoelectric properties.[1-2] PZT has a pure Curie point of about 350°C, but recently it has been confirmed that an ultrasonic response can be obtained even when PZT /PZT is 400°C.[3] Since the Curie point of the PZT powder used at this time is about 300°C, it is expected that by using a sol-gel composite, the Curie point is increased and as a result, the heat resistance is improved. However, when the hot plate temperature was 400°C, the signal strength and signal-to-noise ratio(SNR) deteriorated significantly, making it far from practical use. This is thought to be due to acceleration of depoling because the applied voltage of the pulsar receiver is opposite to the polarity during poling. The pulsar receiver applies a negative pulse voltage and applies a voltage in the direction opposite to the normal poling direction. This is because a general piezoelectric material is dense and can be stretched, but it is difficult to shrink. However, the sol-gel composite has fine pores distributed in the material. Due to this porosity, the sol-gel composite can also shrink. Therefore, it is possible to perform poling with the same polarity as the applied voltage of the pulsar receiver. Therefore, in this study, PZT/PZT films were polled by conventional positive corona discharge and negative corona discharge, and the comparison was made.

2. Fabrication of PZT/PZT films

Spray coating technology has been selected as a manufacturing method because it can serve many industrial applications due to its curved surface compatibility. The PZT powder was mixed with a laboratory-made PZT sol-gel solution. After ball milling, the mixture was sprayed onto a titanium substrate with dimensions of 3mm thickness, 30mm length and 30mm width. In this experiment, an automatic coating machine was used in the spray coating process to reduce individual differences. Heat treatment at 80°C with a hot plate, 150°C with a hot plate, and 650°C with a furnace followed by a drying process and an annealing process. Spray coating and heat treatment were repeated until the film thickness reached the target film thickness of 40μm.After achieving the target film thickness, poling was performed by positive and negative corona discharge, and the upper electrode was made of silver paste.

3. Experimental results

Ultrasonic measurements were operated at room temperature in pulse-echo mode. Negative pulsed voltage was supplied from a pulsar/receiver machine (P/R) to a PZT/PZT ultrasonic transducers. The results were recorded by a digital oscilloscope. **Figs. 1-2** show the ultrasound response in the time domain at room temperature of each sample and **Figs. 3-4** show the FFT results of the first wave in Figs. 1-2, respectively. No significant difference was observed.

Maximum operating temperature tests were performed on each PZT/PZT ultrasonic transducer to determine poling direction effect. The samples were placed on a hot plate and the temperature of the hot plate was changed by 50°C from room temperature to 200°C, and when the temperature was further raised, the temperature was raised by 10°C. After holding for 5 minutes at each temperature, an ultrasonic waveform was recorded. In order to determine the temperature effect quantitatively, sensitivity was calculated as following equation;

$$Sensitivity = -\left(20\log\frac{V_1}{V_2} + Gain\ of\ \mathrm{P/R}\right) \quad (1)$$

where V_1 is the reference amplitude, in this experiment 0.1 Vp-p, and V_2 is the Vp-p of the second reflected echo from the bottom of the titanium substrate. Because P/R stands for pulser/receiver, this equation calculates the true required gain of the pulser/receiver to achieve 0.1V.

Then the value was multiplied by -1 to aid in your own understanding. **Fig. 5** shows the sensitivity of each sample from room temperature to 420 °C. As a result, PZT/ PZT poled by negative corona discharge showed higher thermal durability than PZT/PZT poled by positive corona discharge.

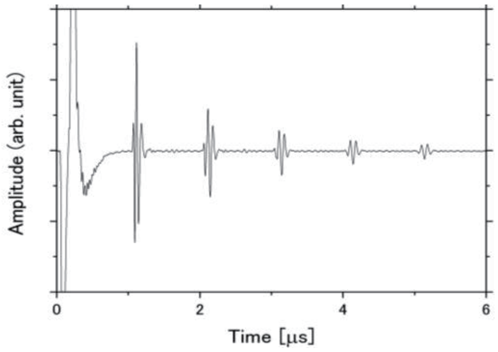

Fig. 1 Ultrasonic response of PZT/PZT transducers fabricated by positive corona poling on 3mm thick titanium substrate at room temperature

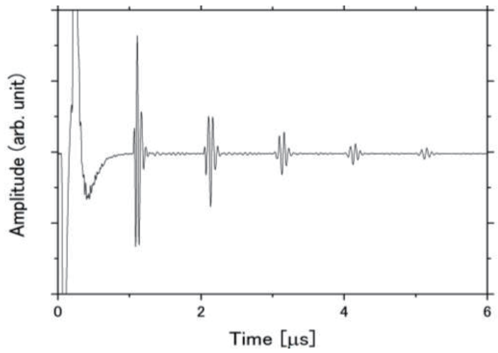

Fig. 2 Ultrasonic response of PZT/PZT transducers fabricated by negative corona poling on 3mm thick titanium substrate at room temperature

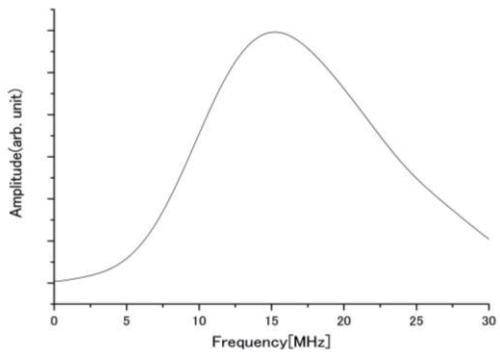

Fig. 3 First reflected wave FFT result of pulse echo waveform at room temperature of PZT / PZT film poled by positive corona discharge

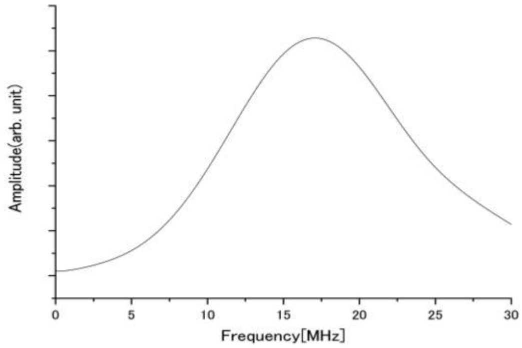

Fig. 4 First reflected wave FFT result of pulse echo waveform at room temperature of PZT / PZT film poled by negative corona discharge

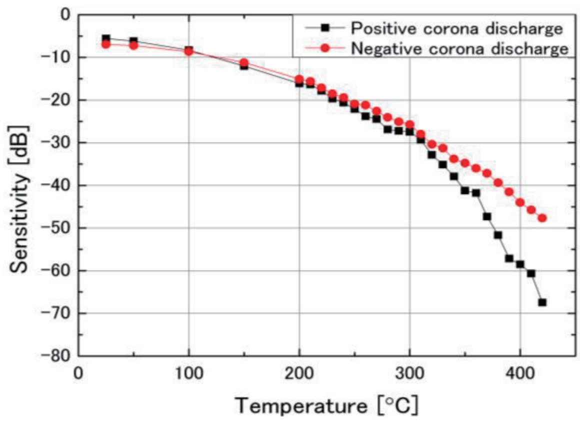

Fig. 5 Sensitivity temperature dependency comparison between positive corona discharge and negative corona discharge

5. Conclusions

To improve thermal durability of PZT/PZT, negative corona discharge was used for poling and ultrasonic performance were compared with that by poled by traditional positive corona discharge. As a result, PZT/PZT poled by negative corona discharge showed superior performance above 300°C than PZT/PZT poled by traditional positive corona discharge. Further experiments will be carried out by replacing top electrode material to more stable one.

References

1. M. Kobayashi and C.-K. Jen: Ultrasonic transducers: materials design and application, Ed. by K. Nakamura and S. Ueha, Woodhead Publishing (2012) 408.
2. K. Kimoto, M. Matsumoto, T. Kaneko, and M. Kobayashi: Jpn. J. Appl. Phys. **55** (2016) 07KB04.
3. M. Kobayashi, T. Kibe, and H. Nagata: Sensors, (2017).
4. S. Fujimoto, T. Namihira, K. Iwata, and M. Kobayashi: Jpn. J. Appl. Phys. **54** (2015) 07HB04.