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Room Temperature Poling of Sol-Gel Composite Materials with High Coercive Field Piezoelectric Powder Phase

高抗電界圧電粉体相を用いたゾルゲル複合材料の室温分極に関する研究

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1. Introduction

Sol-gel composite ultrasonic transducers have been developed to improve high-temperature durability. [1-2] Sol-gel composites are composed of a ferroelectric powder and a dielectric sol-gel-derived material. Sol-gel composites are composed of three phases, a piezoelectric powder phase, a dielectric sol-gel phase, and an air phase. A sol-gel composite has three main advantages.[3)] First, no backing material is necessary because tiny pores are manufactured inside the sol-gel composite film, which cause ultrasonic scattering and reduce the ringing effect. Second, a couplant is also unnecessary because a sol-gel composite has good acoustic coupling with the substrate since an oxide layer is created between the substrate and the piezoelectric film by a thermal process during film fabrication. Third, the sol-gel composite film has high thermal shock resistance because there are many micro holes inside the film.

In past study, $PbTiO_3$ (PT)/ $Pb(Zr,Ti)O_3$ (PZT), made by PT powders, piezoelectric powder phase with high coercive field and PZT sol-gel solution, demonstrated superior sensitivity to PZT/PZT from room temperature to 360 °C. [4)] However, PT/PZT required poling at high-temperature due to high coercive field of PT in order to obtain high piezoelectricity. Therefore, it took a long time for poling process and it could be an issue for on-site fabriation. If PT/PZT can be poled at RT, manufacturing cost is reduced, and on-site fabrication becomes more realistic. In this research, new poling method at RT was attempt for PT/PZT and results between PT/PZT poled by new poling method at room temperature and PT/PZT poled by traditional method at high-temperature were compared.

2. Fabrication of PT/PZT films

For PT/PZT film fabrication, the mixture of PZT sol-gel solution and PT powder were ball milled for more than one day. Then it was sprayed onto a 3mm thick titanium substrate. After spray coating, drying process at 150°C for 5min, and firing process at 650°C for 5min were operated. In this research, those spray coating process and thermal process were repeated twice. PT/PZT film thickness was 15μm.

3. High and Room Temperature Poling

First, traditional poling at high-temperature was operated by adding positive corona discharge after heating the substrate by a hot plate. Corona poling utilizes corona discharge, which surpress dielectric breakdown which often occured when DC voltage was applied directly to the sol-gel composite film. In this experiment, DC voltage 30kV was used and the sample was poled for 15 minutes after heating up to 400°C. Next, the new poling method at RT was performed by pulse voltage source. In this experiment, pulse repetition frequency was 20kHz, pulse width was 7μs, voltage was 20kV and poling time was 15sec. Optical images of each sample after poling were shown in **Fig. 1**. The part of the PT/PZT film poled at RT had brown spots, which did not occurred for the sample poled at traditional corona poling at high-temperature.

Fig. 1 PT/PZT film optical images poled at (a) high temperature and (b) at room temperature.

4. Experimental results

First, Ultrasonic responses of each PT/PZT films were monitored **Figure 2** shows ultrasonic response of PT/PZT poled by traditional corona poling at high temperature. Multiple reflected echoes from the bottom surface of the titanium substrate were confirmed. Piezoelectric constant d_{33} of that sample was 7.8 pC/N. **Figure 3** shows ultrasonic response of PT/PZT poled by pulse voltage at RT. Multiple reflected echoes were confirmed as well. Then, Piezoelectric constant d_{33} of PT/PZT was 12.9 pC/N. From those results, there was no significance difference between PT/PZT samples poled at high-temperature and RT.

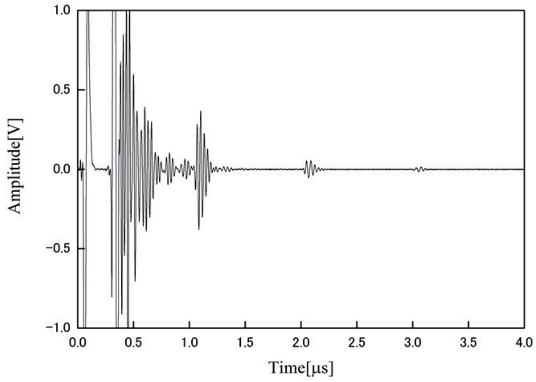

Fig.2 Ultrasonic response of PT/PZT poled at high temperature on ~3mm thick titanium substrate

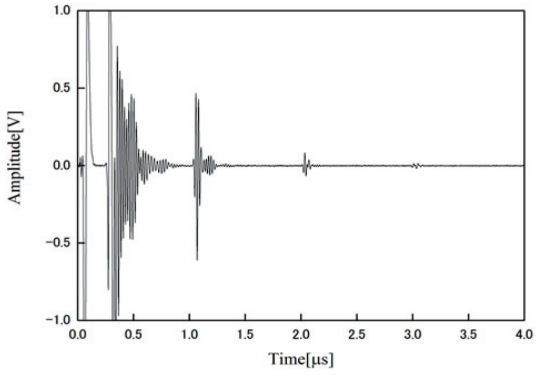

Fig.3 Ultrasonic response of PT/PZT poled at room temperature on ~3mm thick titanium substrate

Next, Fast Fourier Transform (FFT) results of second reflected echoes in **Figs. 2** and **3** were shown in **Figs. 4** and **5,** respectively. Center frequency of the sample poled by high-temperature poling and RT poling was about ~32MHz and ~27MHz respectively. 6dB bandwidth was ~12 MHz. From those results, PT/PZT sol-gel composite poled by new poling method at RT showed reasonable ultrasonic response, as good as that of poled by traditional corona poling at high-temperature.

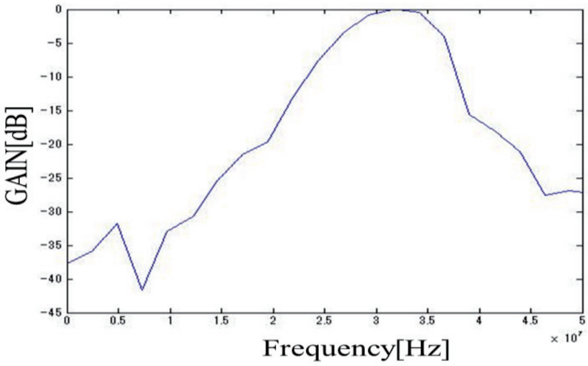

Fig.4 FFT of second wave of Fig.2

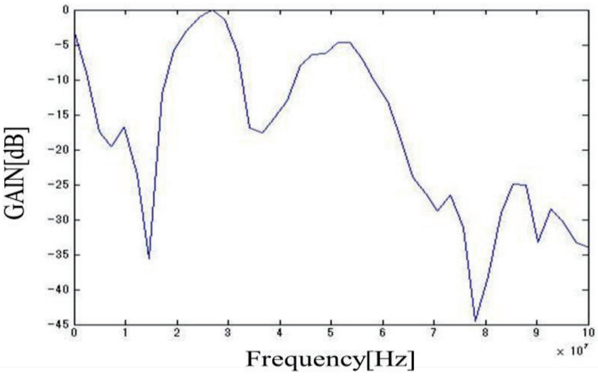

Fig.5 FFT of second wave of Fig.3

5. Conclusions

New poling method for PT/PZT at RT was attempt using pulse voltage source. That sample showed reasonable piezoelectric properties and ultrasonic response, as good as that of the sample poled by traditional corona poling at high-temperature. Therefore, PT/PZT can be poled at RT in a short time with resonable device perfirmance with using pulse voltage source.

References

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