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$Pb(Zr, Ti)O_3/Pb(Zr, Ti)O_3$ Poling by pulse voltage

パルス電圧による$Pb(Zr, Ti)O_3/Pb(Zr, Ti)O_3$の分極に関する研究

Makie Hidaka[1‡], Minori Furukawa[1], and Makiko Kobayashi[1] ([1]Kumamoto Univ.)
日高　蒔恵[1‡]，古川　美徳[1]，小林　牧子[1] ([1]熊本大学)

1. Introduction

Piezoelectric sol-gel composite films were manufactured from sol-gel solutions and piezoelectric powders and have a potentila for a variety of devices. Piezoelectric sol-gel composites could be used as ultrasonic transducers for non-destructive testing. $Pb(Zr,Ti)O_3$ (PZT) material is usually used as ultrasonic transducers, because it has high piezoelectricity and relatively high Curie temperature. Recenrly, reproductivity of PZT/PZT samples had been improved drastically by automatic spray coating system.[1-2] There is a still demand for higher reproductivity for real applications.

Poling process is key process. Piezoelectric materials can show piezoelectric effect by poling process. Conventional poling process is performed by gradually applying DC voltage. However, this method takes a long time and may cause dielectric breakdown due to porosity of sol-gel composite. Therefore, corona discharge is commonly used for sol-gel composite, but it could cause performance deviation due to chaos phenomena.

To achieve the constant piezoelectricity and prevent dielectric breakdown, poling using a pulse voltage source was attempt in this research. As a pulse voltage source, pulser-receiver (P/R) was used.

2. PZT/PZT sample fabrication

PZT/PZT film was fabricated onto titanium substrates by sol-gel spray coating method. The area of the titanium substrate was 3cm square, and substrate thickness was 3mm. First, PZT sol-gel solution and PZT powder were mixed by ball milling machine for a day and after that, the mixture was sprayed onto titanium substrate with automatic spray coating machine. Then drying at room temperature was performed for 5minutes. Drying at 80℃ and 150℃ was performed on a hot plate for 5minutes. Then firing at 650℃ was performed in furnace for 5minutes. Spray and drying, firing process was repeated until target thickness. In this study target thickness was 50μm. In addition, silver paste was manually coated on the PZT/PZT film as the top electrode. Finally, pulse voltage poling was executed by P/R until the reflected echoes from the bottom surface of the substrate was observed in pulse-echo mode. A digital oscilloscope was used for data recording. Different poling directions were used for each sample to investigate the effects. Optical images of PZT/PZT samples are shown in **Fig. 1**.

(a)　　(b)

Fig.1 Optical images of (a) PZT/PZT with positive pulse, (b) PZT/PZT with negative pulse.

3. Experimental Results

The samples were set on a hot plate and ultrasonic measurement in pulse-echo mode was performed. The temperature increases from room temperature to 120 ° C in 10 ° C increments. After holding at each temperature for 5 minutes, the ultrasonic response was recorded by a digital oscilloscope. **Figs.2** and **3** show the results of ultrasonic measurements at room temperature. In Figs.2 and 3, the reflected echoes were clearly observed, however, attenuation ratios were different so it indicates center frequency difference. It is also noted that signal strengths were lower by 20dB compared with corona discharge poling.

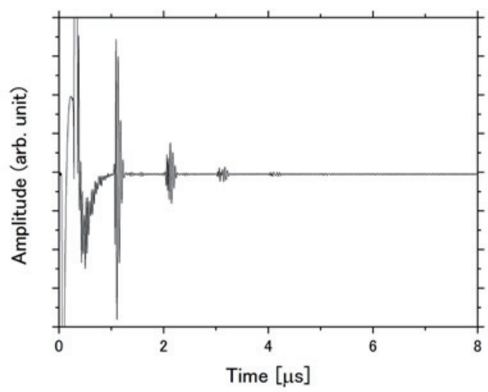

Fig. 2 Ultrasonic response poled by positive pulse voltage at room temperature.

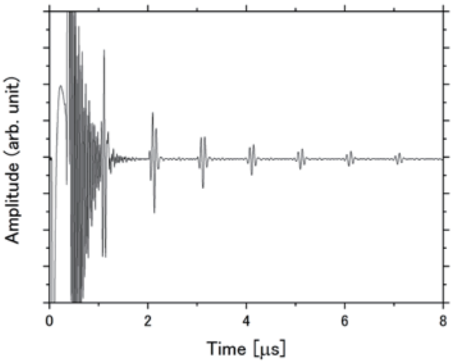

Fig. 3 Ultrasonic response poled by negative pulse voltage at room temperature.

Fast Fourier transform (FFT) results of second reflected echoes for the samples poled by positive and negative pulses is shown in **Figs.4** and **5**, respectively. The center frequency poled by positive pulse was 22.5 MHz and the 6dB bandwidth was about 14.1MHz, whereas the center frequency poled negative pulse was 13.0MHz and the 6dB bandwidth was about 11.3MHz. Fig.4 shows sample poled by positive pulse has high frequency. Fig.5 show sample poled by negative pulse has low frequency and broadband characteristic.

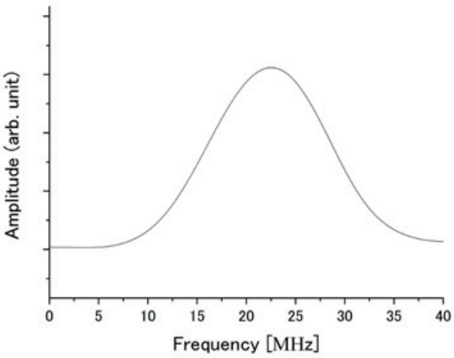

Fig. 4 FFT result poled by positive pulse voltage.

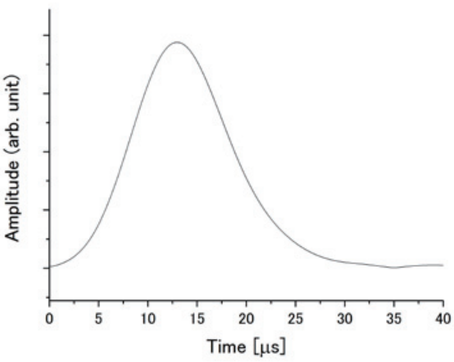

Fig. 5 FFT result poled by negative pulse voltage.

The temperature dependences of PZT/PZT samples are shown in **Figs.6** and **7**. Fig.6 and Fig.7 show that the sensitivity of PZT/PZT sample poled by negative pulse is more stable. The repeatability will be investigated by multiple sensors with more reliable top electrodes to make final conclusions, including center frequency tendency.

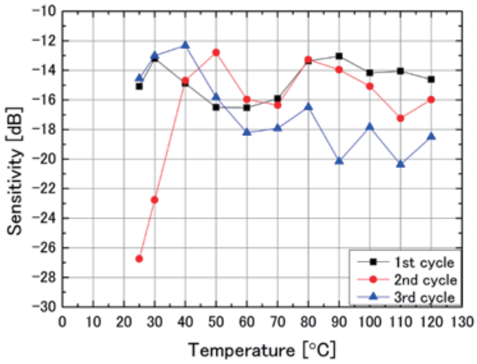

Fig. 6 Sensitivity of PZT/PZT sample poled by positive pulse at each temperature.

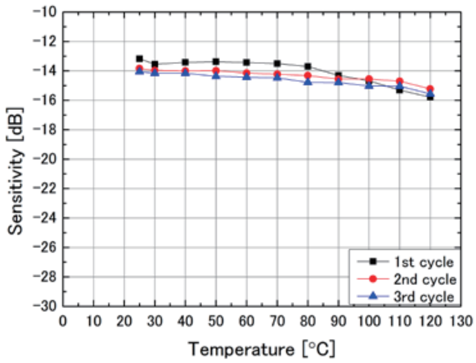

Fig. 7 Sensitivity of PZT/PZT sample poled by negative pulse at each temperature.

4. Conclusions

PZT/PZT sol-gel composite film was fabricated on a titanium substrate by sol-gel spray coating method and positive and negative pulse voltages were directly applied for poling by P/R. The sample poled by negative pulse voltage had lower center frequency and broad bandwidth. The cycle test was performed for 3 cycles from RT to 120°C. It was found that PZT/PZT poled by negative pulse voltage showed more temperature stability. In order to improve reproducibility in the future, it is necessary to create a higher quality upper electrode.

References

1. Y. Kiyota, H. Makino, K. Nakatsuma and M. Kobayashi: Proc. Symp. USE (2018).
2. . Kiyota, K. Nakatsuma, and M. Kobayashi: Jpn. Proc. Symp. USE (2017).