1Pa1-4

Ambience effect during poling of $Pb(Zr,Ti)O_3/Pb(Zr,Ti)O_3$

$Pb(Zr,Ti)O_3/Pb(Zr,Ti)O_3$ における分極雰囲気の影響

Ayaka Yamasaki[1‡], Makie Hidaka[1], Kei Nakatsuma[1] and Makiko Kobayashi[1]
([1]Kumamoto Univ.)
山﨑彩加 [1‡]，日高蒔恵 [1]，中妻啓 [1]，小林牧子 [1]([1]熊本大学)

1. Introduction

Non-Destructive Testing (NDT) is in increasing demand in the industrial field to detect small defects in the early stages. However, since some measurement points become hot, the conventional ultrasonic transducers could not withstand the high temperature and the plant had to be stopped before inspection.[1] Therefore, we developed a piezoelectric sol-gel composite material and fabricated a thick film, which was used as an ultrasonic transducer for NDT. Sol-gel composites are made by mixing sol-gel solution and piezoelectric powder. It is often used for NDT in high temperature environments because it has excellent high temperature durability and curved surface compatibility and does not require couplant or backing material. $Pb(Zr,Ti)O_3$ (PZT) materials are often used for ultrasonic transducers because of their high piezoelectric properties and relatively high Curie temperature.[2-4)] In this study, PZT/PZT ultrasonic transducers were fabricated by an automated spray method and polarized by negative corona discharge, because it is known that the thermal resistance of ultrasonic transducers is improved when polarized by negative corona discharge in PZT/PZT.[5] In the study of discharges in gases, especially corona phenomena in the atmosphere, it has been observed that the humidity of the air has a significant effect on the behavior of the corona. As the relative air humidity increases, the corona induced voltage rises to a certain limit, and then the corona induced voltage begins to decrease.[6] So, the atmosphere at the time of corona discharge might affect the performance of the sensor. Therefore, we investigated whether the humidity should be controlled by changing the conditions of the atmosphere during the poling.

2. PZT/PZT ultrasonic transducer fabrication

The PZT/PZT sol-gel composite was prepared by the sol-gel spray method. First, PZT piezoelectric powder and sol-gel solution were mixed in a ball mill machine for about a day. The prepared mixture was applied to a 30 mm x 30 mm x 3 mm titanium substrate by automatic spraying. It was then dried at 150°C for 5 minutes and fired at 650°C for 5 minutes. These spraying, drying, and baking processes were repeated until the target film thickness reached 50 µm. The sol-gel composite was then subjected to negative corona discharge in order to use it as an ultrasonic transducer. In order to investigate the effect of the ambient gas, poling was carried out with and without humidity control. When humidity was controlled, poling was performed with nitrogen and dry air, respectively. Finally, an upper electrode with a diameter of 1 cm was fabricated using a silver pen. The optical image of PZT/PZT on titanium substrate sample is shown in **Fig. 1**. Three samples were prepared for each condition. The piezoelectric constants d_{33} of each sample are shown in **Table I**.

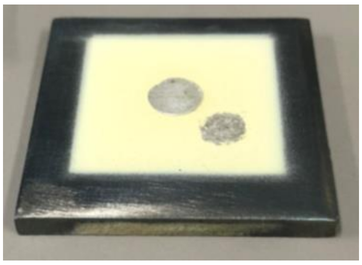

Fig 1. Optical image of LN/PZT film fabricated on 3mm thick titanium substrate.

Table I: The piezoelectric constants d_{33} [pC/N]

Sumple	High humidity	With nitrogen	With Dry air
1	0.0		
2	0.0		
3	0.0		
4		-41.1	
5		-38.2	
6		-45.0	
7			-38.1
8			-45.7
9			-45.2

3. Experimental results

The ultrasound measurements were performed in pulse-echo mode at room temperature. A negative pulse voltage was supplied to the PZT/PZT ultrasonic transducer from a pulsar/receiver machine (P/R). The results were recorded on a digital oscilloscope. The ultrasonic response of the sample without humidity control during poling is shown in **Fig. 2**. The humidity at the time was 60%. **Fig. 2** shows that the reflected wave was visible, but the scale was 50mV, which is much smaller than the 500mV of the sample described below. The waveform was not very clear. Also, measured d33 values were 0.0. **Figs. 3** and **4** show the ultrasonic response of the samples with the humidity with nitrogen and dry air during poling, respectively. In both cases, the humidity at the time of poling was around 11%. **Figs. 3** and **4** show that a clear ultrasonic response was obtained at room temperature. The piezoelectric constants for both nitrogen and dry air were -30~-50 [pC/N].

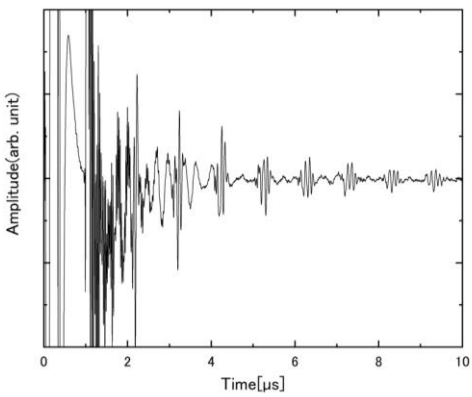

Fig. 2 Ultrasonic response of by negative corona poling without humidity control.

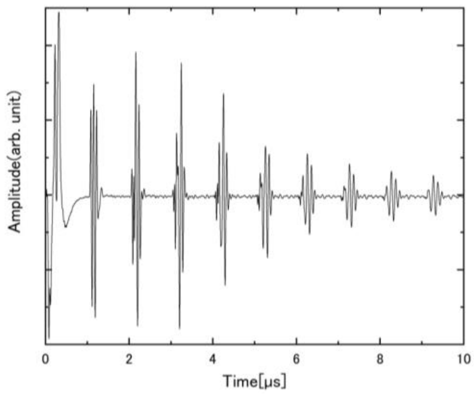

Fig. 3 Ultrasonic response of by negative corona poling with humidity control by nitrogen.

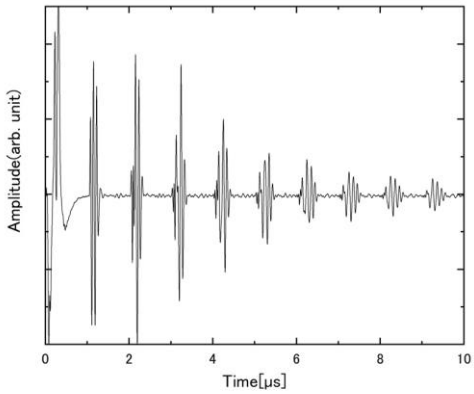

Fig. 4 Ultrasonic response of by negative corona poling without humidity control by dry air.

4. Conclusions

PZT/PZT sol-gel composites were prepared on titanium substrate by automatic spraying method. Poling was performed by negative corona discharge. The results were compared with three different atmosphere gases. When the humidity was not controlled, the ultrasonic response was weak and measured d33 values were 0.0. When the humidity was controlled with nitrogen and dry air, a clear ultrasonic response was obtained and the piezoelectric constant was -30~-50 [pC/N]. From the above results, it was found that in PZT/PZT sol-gel composites, it is necessary to control the humidity during poling, but the atmosphere gas can be either nitrogen or dry air.

References

1. K.Uchino, Ferroelectric devices, Marcel Dekker:New York, (2000) 145.

2. K. Kimoto, M. Matsumoto, T. Kaneko, and M. Kobayashi: Jpn. J. Appl. Phys. 55 (2016) 07KB04

3. S. Fujimoto, T. Namihira, K. Iwata, and M. Kobayashi: Jpn. J. Appl. Phys. 54 (2015) 07HB04

4. M. Hidaka, T. Hara, K. Nakatsuma, and M. Kobayashi: Proceedings of Symposium on Ultrasonic Electronics, Vol. 41 (2020).

5. T. Hara, M. Furukawa, S. Nozawa, K. Nakatsuma, and M. Kobayashi: Jpn. J. Appl. Phys. 59 (2020) SKKC10

6. L. Fouad and Elhazek: Journal of Electrostatics 35 (1995) 21-30