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Poling Optimization of $Pb(Zr,Ti)O_3/Al_2O_3$ Sol-gel Composite

$Pb(Zr,Ti)O_3/Al_2O_3$ ゾルゲル複合体の分極に関する研究

Daichi Maeda[1‡], Tomoya Yamamoto[1], Kazuki Okada[1], Makiko Kobayashi[1] ([1]Kumamoto Univ.)
前田　大地[1‡]，山本　智也[1]，岡田　一希[1]，小林　牧子[1]([1]熊本大)

1. Introduction

Non-destructive testing (NDT) widely used for several fields to detect early stage small defects before fatal failure. Various NDT methods have been developed and ultrasound NDT is one of the common methods because it is possible to measure thinning of the structure and to detect defects and cracks inside the structure, which is difficult by external image diagnosis. Sol-gel composite ultrasonic transducers have been developed for on-line NDT monitoring and there are several advantages[1-5]. First, backing material is unnecessary because there are small pores inside the sol-gel composite film and it reduces ringing effect. Second, since the sol-gel composite film has small pores as described above, it has high thermal shock durability. Third, since the oxide layer is formed by the heating process between the sol-gel composite and the substrate, the coupling agent is unnecessary since it has good acoustic coupling with the substrate.

In past study, $Pb(Zr,Ti)O_3$ (PZT)/Al_2O_3 sol-gel composite transducer was developed[5]. At that time, poling was operated by directly applying DC voltage at high temperature, such as 150°C. However, dielectric breakdown tends to occur during poling process so that only low voltage could be applied. Therefore, the poling voltage could not be sufficiently high. Ultrasonic performance at elevated temperatures was not investigated yet. In this research, PZT/Al_2O_3 sol-gel composite transducers were poled by corona discharge to prevent dielectric breakdown and ultrasonic performance at various temperatures was studied.

2. Fabrication process of PZT/ Al_2O_3 films

Samples were manufactured by sol-gel spray technique. First, Al_2O_3 sol-gel solution was self-manufactured. PZT/Al_2O_3 sol-gel composite was prepared by mixing PZT piezoelectric powders and Al_2O_3 sol-gel solution by a ball mill machine for about 1 day. Sol-gel composite films were coated on 3-mm-thick titanium substrates by spray method[1-4]. Then, drying at 150°C and firing at 650°C were carried out for 5 min each, respectively. The spray-coating process and heating process were repeated until the target thickness was achieved. This research was repeated about 5 times to obtain 50μm. When the target film thickness was reached, poling was carried out at room temperature for 10 min. Poling using corona discharge is to avoid dielectric breakdown. The output voltage and the output current were about 19 kV and about 0.10 mA, respectively. A top electrode was manufactured by silver pen. Optical image of PZT/Al_2O_3 sample is shown in **Fig. 1**.

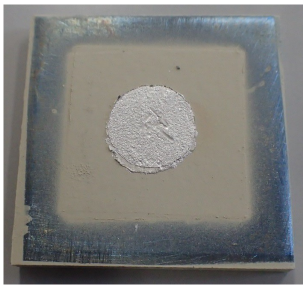

Fig. 1. Optical image of PZT/Al_2O_3 sample on a 3-mm thick titanium substrate.

3. Experimental Results

To determine thermal durability, maximum temperature test was carried out for PZT/Al_2O_3 sol-gel composite sample. The sample was set onto a hot plate and ultrasonic measurements in pulse-echo mode were operated. Temperatures ranged from room temperature to 190 °C in increments of 20 °C, and from 190 °C to 300 °C in 10 °C increments. After 5min holding time at each temperature, ultrasonic response was recorded by a digital oscilloscope. Ultrasonic measurement results at room temperature and 300°C are shown in **Figs. 2** and **3**, respectively. Clear multiple echoes were observed for both measurements, even though signal-to-noise ratio was deteriorated in Fig. 3, due to depoling of PZT. Ultrasonic response of PZT/Al_2O_3 ultrasonic transducer was able to confirm multiple signals even at 300°C. The reason of dead zone change could be electrical impedance mismatching caused by PZT/Al_2O_3 and measurement instruments.

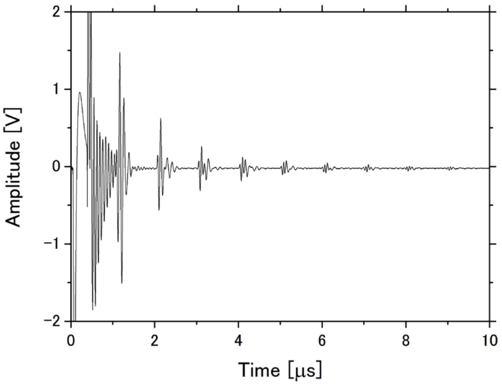

Fig. 2. Ultrasonic response of PZT/Al_2O_3 sample fabricated on 3-mm-thick titanium substrate at room temperature.

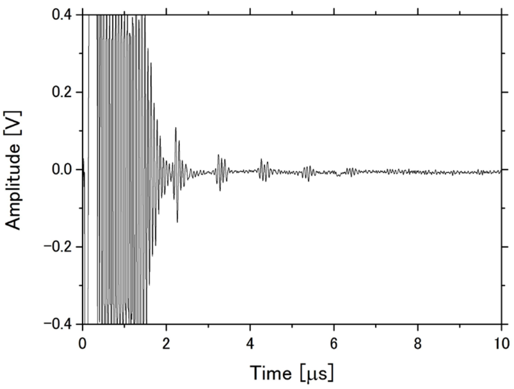

Fig. 3. Ultrasonic response of PZT/Al_2O_3 sample fabricated on 3-mm-thick titanium substrate at 300°C.

The sensitivity of maximum temperature test is shown in **Fig.4**. The equation of the sensitivity is given as

$$\text{Sensitivity} = -(20\log_{10}V_1/V_2 + \text{gain of P/R})\ (\text{dB}) \quad (1)$$

Where V_1 is the ideal amplitude, 0.1 (V) in this experiment, V_2 is the amplitude (V) of the second reflected echo from the bottom surface of the substrate. From Fig.4, the sensitivity gradually decreased as temperature increased. Since Al_2O_3 sol-gel phase does not have piezoelectricity, so it seems that PZT powder phase was depoled by temperature. Thermal cycle tests will be carried out to confirm whether depoling is reversible or irreversible. Other poling conditions by corona discharge will be also carried out to determine PZT/$A_{l2}O_3$ potential.

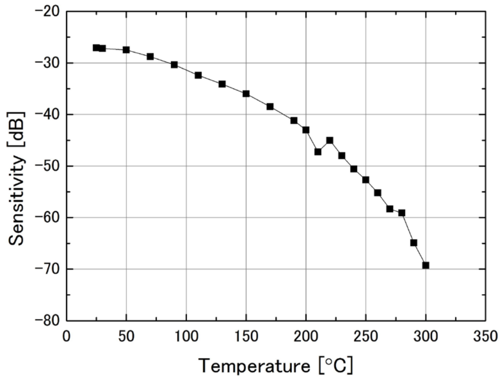

Fig. 4. Temperature dependence of the sensitivity of PZT/Al_2O_3.

4. Conclusions

PZT/Al_2O_3 sol-gel composite samples were fabricated on 3-mm-thick titanium substrates for poling optimization experiment. In this time, PZT/Al_2O_3 were poled not by traditional DC poling but corona discharge poling at room temperature. Compared with past experimental results, signal strength of PZT/Al_2O_3 was improved by corona discharge poling even though poling was executed at room temperature. The maximum operation temperature test was carried out and the ultrasonic performance of the PZT/Al_2O_3 was confirmed with reasonable SNR even at 300°C. PZT/Al_2O_3 could show the possibility of use at a temperature of 300 °C.

References

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