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Development of $Pb(Zr,Ti)O_3/TiO_2$ Ultrasonic Transducer

$Pb(Zr,Ti)O_3/TiO_2$ 超音波トランスデューサに関する研究

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1. Introduction

High temperature ultrasonic transducers using have been investigated in the field of non-destructive testing (NDT) for safety assuarance. It is difficult to apply commercial ultrasonic transducer in this application, mainly due to lack of high temperature durability caused by backing material and couplant. Sol-gel composite ultrasonic transducers have been developed to elminate backing material and couplant problems.[1-5)]PZT/PZT shows good ultrasonic performance up to 200°C. [6)] However, PZT has a high dielectric constant and PZT/PZT piezoelectricity deteriorates when sintering temperature is lower than crystaliczation of PZT sol-gel phase. In past PZT based sol-gel studies, firing was carried out at 650°C. It would be concerned for on-site fabrication since it is difficult to raise up substrate temperature in such a high degree. In addition, fabrication temperature controle of flexible sensor is somewhat difficult.[7)] Therefore, low manufacturing temperature of PZT-based sol-gel composite has been desired.

In this experiment, TiO_2 was used as a sol-gel solution. Amorphous TiO_2 has a high resistivity, and according to our experiments, amorphous TiO_2 was chemically sysnthesized around 400°C. Therefore, it was thought that low firing temperature around 400°C of PZT based sol-gel composite could be achieved with maintaing high piezoelectricity. PZT powder with relatively low dielectric constant was used as shown in **Table I**. In this experiment, PZT/TiO_2 sol-gel composite film was fabricated by firing 400°C and ultrasonic performance was investigated at various temperature.

Table I Characteristics of PZT powders

ε_r	k_{33} (%)	d_{33} (pC/N)	Q_m
1300	70	290	2000

2. Sample fabrication

PZT/TiO_2 sol-gel composite was made by sol-gel spray technique. First, PZT powders and TiO_2 sol-gel solution were prepared. TiO_2 sol-gel solutions was self-manufactured. PZT powders are commercially available. The mixtures of PZT powders and TiO_2 sol-gel solution were ball milled. Then, the mixtures were sprayed onto titanium substrate by manual splay technique. The dimensions of titanium substrate were 30mm × 30mm × 3mm. This substrate was chosen due to low thermal capacitance and high temperature durability. After spray coating, the sample was dried at 150°C for 5 min. After drying, the sample was fired at 400°C for 5 min as well. Those spray coating process and thermal process were repeated until film thickness reached 50μm. After film fabrication, poling was operated by corona discharge at room temperature. The output voltage of the power supply was 21.5 kV. Optical image of PZT/TiO_2 film onto titanium substrate is shown in **Fig. 1**. Film thickness of PZT/TiO_2 fired at 400°C was measured by a micrometer and the value were 48μm. Piezoelectric constant d_{33} was measured by ZJ-3B piezo d33 meter and the value was 4.4pC/N. After these processes, high temperature silver paste was fabricated on the film as a top electrode. The top electrode diameter was about 1cm. For drying silver paste, thermal process at around 100°C were carried out for 2 h.

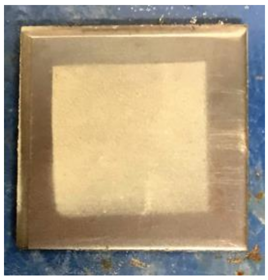

Fig. 1 Optical image of PZT/TiO_2 film with 400°C firing temperature on 3-mm thick titanium substrate.

3. Experimental results

Ultrasonic responses of the PZT/TiO_2 sample in pulse-echo mode were recorded from room temperature to 200°C. The substrates were set onto hot plate and raised the temperature in increments of 10°C. After 5 min holding temperature, the ultrasonic response was measured and the data was recorded by a digital oscilloscope. The ultrasonic measurement result at 200°C is shown in **Fig. 2**. From Fig. 2, multiple echoes can be observed and high signal to noise ratio (SNR) were obtained even at 200°C. From this fact, it was found that PZT/ TiO_2 with low temperature firing of 400°C could function as an ultrasonic transducer sufficiently. 1st reflected echo was hidden due to electrical impedance mismatch. The peeling of silver top electrode happened during high temperature measurement test and the electrical impedance mismatch could be caused by area reduction due to partially peeling of top electrode. Over thickness of silver paste was considerable reason.

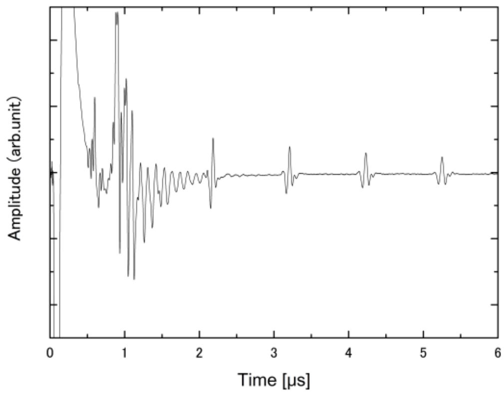

Fig. 2 Ultrasonic response of PZT/TiO_2 film at 200°C.

The result of fast fourier transform (FFT) of 2nd reflected echo at 200°C is shown in **Fig. 3**. From Fig. 3, the center frequency was 12.2MHz, and 6dB bandwidth was about 16.4MHz. It was found that PZT/TiO_2 sample has high frequency and broadband characteristic.

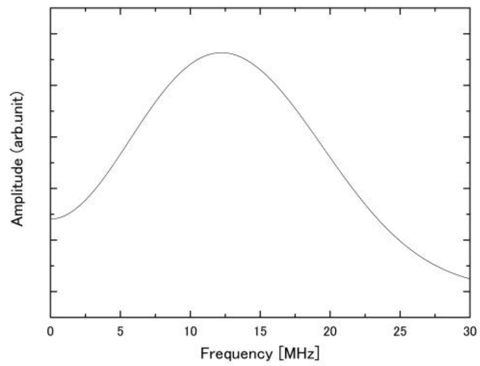

Fig. 3 FFT result of ultrasonic response of PZT/TiO_2 film at 200°C.

In order to determine maximum operation temperature, the temperature was continuously increased by 10°C until there was no ultrasonic response. Although the SNR was significantly deteriorated, it was possible to observe multiple reflected echoes up to 290°C. Therefore, PZT/TiO_2 sol-gel composite with 400°C firing temperature has sufficiently high temperature durability as PZT based material.

4. Conclusion

48μm thick PZT/TiO_2 sol-gel composite was fabricated on a 3mm thick titanium substrate to determine low fabrication temperature possibility. The sample was fired at 400°C and the piezoelectric constant d_{33} was 4.4pC/N. Pulse-echo mode ultrasonic measurement was carried out from room temperature to the temperature where waveform cannot be observed. At 200°C, the waveform could be clearly observed with 12.2MHz center frequency and 16.4MHz 6dB bandwidth, respectively. Ultrasonic response could be confirmed up to 290°C. From this study, PZT/TiO_2 demonstrated sufficiently high performance even maximum fabrication temperature was 200°C. Further research is required for comparison of traditional PZT/PZT.

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