Ultrasonic Characteristics of $PbTiO_3/Pb(Zr,Ti)O_3$ at High Temperature

$PbTiO_3/Pb(Zr,Ti)O_3$ の高温下における超音波特性

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1. Introduction

Periodic inspections are necessary for the safety of industrial facilities. For example, a single accident at a nuclear power plant may cause long-term damage to the environment, economy, and livelihoods.so continuous monitoring during operation is desirable. Non-Destructive-Testing (NDT) using ultrasonic transducers is a method to monitor wear and detect sub-surface defects. However, due to the high temperatures at some of the measurement points, conventional ultrasonic transducers cannot withstand the high temperatures, so it is necessary to shut down the plant before performing the inspection.(1) Therefore, An ultrasonic transducer based on the sol-gel spray method, which can shrink because of the presence of voids in the sol-gel composite film, and can be used in a high temperature environment without any couplant or backing material, has been developed.(2) Since conventional ultrasonic transducers are dense and can be stretched, but not shrunk, we use a pulsar receiver (P/R) that is polarized by a positive voltage and a negative voltage is applied to the transducer. Therefore, at high temperatures, depolarization is promoted by the opposite polarization voltage and the applied voltage of the P/R, making measurements difficult, but the Negative voltage can be applied due to the presence of vacancies, and this problem can be solved. In a previous study, we used positive corona discharge in PZT ($Pb(Zr,Ti)O_3$)/PZT and reversed the applied voltage of P/R by using a transformer to suppress depolarization and improve the high-temperature durability(3). PT/PZT showed a good ultrasonic response due to high temperature polarization(4).In this study, PT ($PbTiO_3$)/PZT was poled by positive corona discharge and a transformer was attached to the P/R to align the direction of the applied voltage with the direction of the polarization and to obtain high temperature durability of the P/R.

2. Sample Fabrication

The PT/PZT sol-gel composite method was were fabricated by the sol-gel spray method. First, the PT powder and PZT sol-gel solution were properly mixed and the mixture was ground in a ball mill machine for more than 24 hours. The well-mixed solution was then sprayed onto the titanium substrate using an automatic spray coating machine. The dimensions of the titanium substrate are 30mm × 30mm × 3mm in length, width and height. The titanium substrate was selected because of its low heat capacity and excellent high-temperature durability. After spraying, they were dried in an electric furnace at 150°C and sintered at 400°C for 5 minutes each. The spray and heat treatment processes were repeated until the film thickness of PT/PZT was 50μm. After deposition, the film was polarized by corona discharge. First, the PT/PZT was heated to 400°C and held for 5 minutes. Thereafter, a positive corona discharge of 40 kV was applied for 5 minutes. Then, the upper electrode of 10 mm in diameter was made on the sample using silver paste and dried at 100°C for 2 hours. Optical image of PT/PZT film onto titanium substrate is shown **Fig.1**.

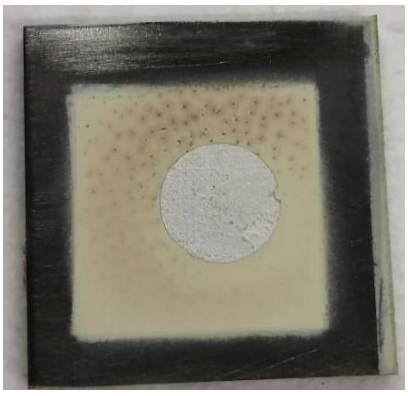

Fig.1 Optical image of PT/PZT film on 3mm thick titanium substrate.

3. Experimental Results

Maximum temperature tests were carried out on PT/PZT sol-gel composite films on titanium substrates. The two samples were set in an electric furnace and electrically bonded using platinum wire as the wire and the weight of the ceramic. The temperature of the furnace was varied from room temperature (RT) to 50°C. The samples were held at each temperature for 5 minutes. After being held at each temperature for 5 minutes, the ultrasonic waveforms were recorded with a digital oscilloscope. In one of the samples, a transformer was attached to the P/R and the polarity was

reversed to record the ultrasonic response. **Fig.2**. and **Fig.3**. show the ultrasonic response at RT with transformer and without a transformer.

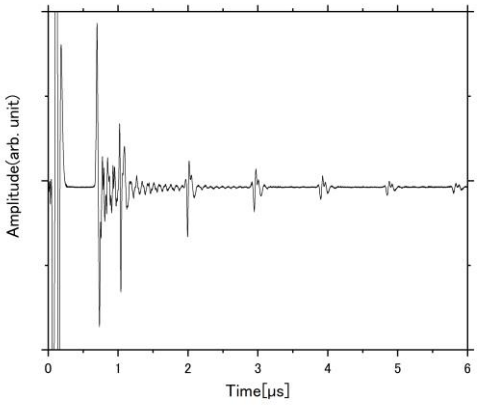

Fig.2 Ultrasonic response at RT without transformer.

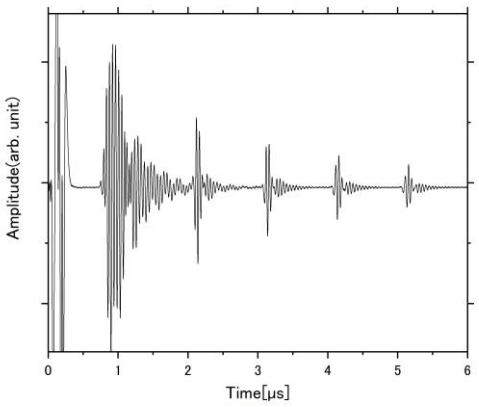

Fig.3 Ultrasonic response at RT with transformer.

Next, sensitivity was calculated for quantitative evaluation. The sensitivity was calculated according to Equation (1).

$$\text{Sensitivity} = -(20\log\frac{V_1}{V_2} + P/R\ Gain) \qquad (1)$$

where V_1 is the reference amplitude, which in this experiment is $0.1V_{p\text{-}p}$, and V_2 is the third reflected wave $V_{p\text{-}p}$ from the lower surface of the titanium substrate. This equation calculates the true P/R gain required to achieve $0.1V_{p\text{-}p}$ for the third reflected wave. Multiplying by -1 helps the visual understanding of the graph. The sensitivity of the PT/PZT ultrasonic transducers at various temperatures is shown in **Fig.4**. Both samples showed a response up to 600°C, but after that, the waveform could not be observed due to depolarization. There was no difference in the shape of the graphs, and the sensitivity decreased with increasing temperature in the same trend. This result is considered to be due to individual differences in the samples and insufficient polarization.

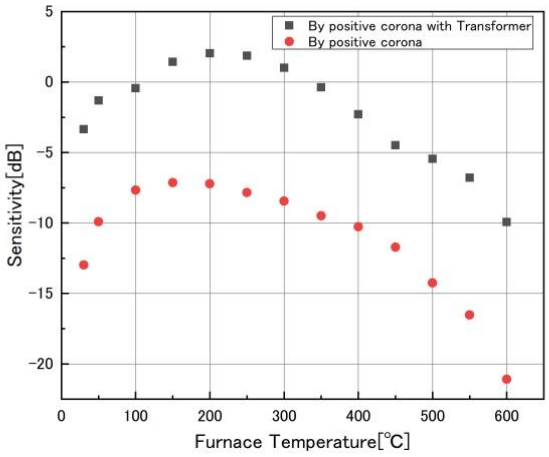

Fig.4 Sensitivity of PT/PZT film at various temperatures during maximum temperature test

4. Conclusiton

In this experiment, the PT/PZT ultrasonic transducer was fabricated by the sol-gel spray method. The objective of this study was to improve the high temperature durability. A transformer was attached to the P/R and the direction of the voltage applied to the P/R was aligned with that of the polarization. The measurements were made in the electric furnace until the waveform could not be observed from RT. As a result, the waveforms of both samples could be observed up to 600°C and the sensitivity decreased with the same trend. This result is considered to be inadequate polarization, and plan to re-examine the polarization conditions and perform the experiment again.

References

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