Piezoelectric Characteristic Sustaining Temperature of Pb(Zr,Ti)O₃/Pb(Zr,Ti)O₃

Pb(Zr,Ti)O₃/Pb(Zr,Ti)O₃における圧電特性持続温度に関する研究

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1. Introduction

A piezoelectric element has a property called a piezoelectric effect, which is a property of generating voltage from mechanical pressure and generating mechanical pressure from voltage. One of the methods of using a piezoelectric element is non-destructive testing (NDT) using an ultrasonic transducer, which are used to find cracks and corrosion in power plants or chemical plants. A conventional ultrasonic transducer uses a couplant or a backing material having low thermal shock. Therefore, inspections of industrial facilities operating at high temperatures have been performed with the operation stopped. In this case, since the defect cannot be noticed in real time, the safety becomes low, and since the operation of the factory is stopped, an economic loss occurs. The sol-gel composite is made by mixing piezoelectric powder and sol-gel solution, contains pores, and requires no couplant or backing material, so it can be used for NDT in high-temperature environments.

Pb(Zr,Ti)O₃ (PZT) materials are commonly used as ultrasonic transducers because of their high piezoelectricity and relatively high Curie temperature. In the past, research was conducted on PZT/PZT ultrasonic transducers manufactured by the manual spray method. It was confirmed that the Curie temperature of PZT was about 350°C, while the piezoelectric property remained even above 350°C, causing the Curie temperature to shift.¹ It has also been confirmed that the heat resistance of PZT/PZT ultrasonic transducers is improved by aligning the directions of the poling voltage and the applied voltage during measurement.² In this research, PZT/PZT ultrasonic transducers are fabricated by the automatic spray method, and the influence of the electric field direction is taken into consideration, and it is verified how long the piezoelectric characteristics last.

2. PZT/PZT ultrasonic transducer fabrication

First, the piezoelectric powder and the sol-gel solution are mixed by a ball mill machine for about one day. After that, spray coating on a 3cm × 3cm titanium substrate with an automatic spray device. Subsequently, drying at 150°C and firing at 650°C are performed. The procedure of spraying, drying and firing is repeated until the target film thickness is reached. The target film thickness in this study was 50 μm. Subsequently, a poling treatment is performed in order to use the sol-gel composite as an ultrasonic transducer. In this experiment, poling was performed by positive corona discharge. After that, an upper electrode having a diameter of 10 mm is made of silver paste. In this research, three PZT/PZT ultrasonic transducers were fabricated. The piezoelectric constant $d_{33}$ of sample 1 was 56.8 [pC/N], the $d_{33}$ of sample 2 was 52.0 [pC/N], and the $d_{33}$ of sample 3 was 54.3 [pC/N].

3. Experimental results and discussion

The sample was set on a hot plate and ultrasonic measurement was performed. The temperature is increased by 40°C from room temperature to 550°C which is the maximum temperature of the hot plate. After holding for 5 minutes at each temperature, the ultrasonic response was recorded with a digital oscilloscope. In this research, three kinds of connection methods were executed at the time of measurement. One is the forward connection method. The second method is to connect the upper electrode to the ground in reverse. The third method is to connect the pulser receiver (P/R) and the ultrasonic transducer by inserting a transformer between them to invert the input from the P/R. Using these, a maximum temperature test was performed to confirm the piezoelectric sustaining temperature of the PZT/PZT film. Also, since P/R applies a negative pulse voltage, the measured voltage becomes negative when normally connected. To quantitatively evaluate the thermal durability, the sensitivity $S$ is calculated by the following formula. $V_1$ is the reference amplitude, 0.1 Vp-p in this experiment. $V_2$ is Vp-p of the third reflected echo from the bottom of the titanium substrate.

$$S = -(20 \log_{10} \frac{V_1}{V_2} + \text{gain of P/R})$$
Fig. 1, 2 and 3 show the ultrasonic response during forward connection, reverse connection and transformer connection. All three waveforms are when the hot plate temperature is 520°C. From Fig. 1, 2 and 3, ultrasonic response was confirmed even at 520°C. Furthermore, in the case of forward connection and transformer connection, a clear ultrasonic response could be confirmed up to 550°C. In the case of reverse connection, could not be confirmed at 550°C due to much noise. This is considered to be due to deterioration of the upper electrode during repeated tests.

Fig. 1 Ultrasonic response at hot plate temperature of 520°C with forward connection

Fig. 2 Ultrasonic response at hot plate temperature of 520°C with reverse connection

Fig. 3 Ultrasonic response at hot plate temperature of 520°C with transformer connection

Fig. 4 shows the sensitivity obtained from the ultrasonic response. The reverse connection and transformer connection PZT/PZT, in which the directions of the measurement voltage and the poling voltage are aligned, showed higher sensitivity than the conventional forward connection method.

4. Conclusion

The PZT/PZT ultrasonic transducer was prepared by the automatic spray method, and the sustaining temperature of the piezoelectric characteristics was confirmed. Each of the ultrasonic transducers measured the maximum temperature test with three connection methods. The ultrasonic response of PZT/PZT by the forward connection method and the connection method using a transformer could be confirmed up to 550°C at the hot plate temperature. In the case of the reverse connection method, a clear ultrasonic response was shown up to 520°C, but at 550°C no clear waveform could be confirmed due to severe noise. In addition, the reverse connection and the transformer connection, in which the electric field directions of the polarization voltage and the measured voltage were the same, showed higher sensitivity than the forward connection PZT/PZT.

In this study, it was found that even if the temperature is changed using a hot plate and the temperature exceeds 500°C, it has piezoelectricity. In the future, it is necessary to put the sample in the furnace and check the accurate substrate temperature using a thermocouple.

References