Influence of Atmosphere on the Polarization of PbTiO₃/Pb(Zr, Ti)O₃

PbTiO₃/Pb(Zr, Ti)O₃の分極時雰囲気に関する研究 Yuki Matsuda^{1‡}, Kohei Hirakawa¹, and Makiko Kobayashi¹ (¹Kumamoto Univ.) 松田 悠希^{1‡}, 平川 康平¹, 小林 牧子¹ (¹熊本大学)

1. Introduction

In order to ensure the safety of power plant equipment, periodic inspections for wear of piping and other components are necessary, and as of now, many plants are manually inspected by workers. Especially in nuclear power plants, continuous monitoring rather than periodic inspections is desirable because a single accident can cause serious impact to the environment, the economy, and people's lives. Non-destructive testing (NDT) using ultrasonic transducers is one way to monitor wear and continuously detect sub-surface conditions. However, since some inspection points are subject to high temperatures, ultrasonic conventional transducers cannot withstand the high temperatures and the plant must be shut down before inspection can be conducted.⁽¹⁾ The thermal resistance of ultrasonic transducers has been improved by developing an ultrasonic transducer using the Sol-gel spray method.⁽²⁾ This is because it has a lower density and more vacancies than a conventional ultrasonic transducer, which suppresses depolarization at high temperatures. Depolarization can also be suppressed by poling PZT(Pb(Zr, Ti)O₃)/PZT with a positive corona discharge and reversing the applied voltage on P/R with a transformer⁽³⁾, or by poling PT/PZT at high temperatures to show a good ultrasonic response,⁽⁴⁾ or by poling PT(PbTiO₃)/PZT with a The polarization could also be suppressed by poling PT/PZT with positive corona discharge and attaching a transformer to the P/R to match the direction of the applied voltage with the direction of polarization.⁽⁵⁾ In this way, high temperature durability was discovered. his experiment examined the effect of humidity on the polarization process. During PT/PZT polarization, PZT reacts with moisture in the air, which may cause performance degradation. Therefore, during PT/PZT polarization, the polarizer was filled with nitrogen to keep the humidity below 20%. In addition, since PT has a high coercive electric field, it is necessary to heat the PT/PZT in a furnace at 400°C to polarize it. In this experiment, It seems like that the effect of moisture in the air might be small because of the heating during the polarization process. Therefore, this experiment was conducted at two different humidity levels, above 80% and below 20%, to compare the polarization and the ultrasonic

response at room temperature.

2. Sample Fabrication

PT/PZT Sol-gel composites were fabricated by the Sol-gel spray method. First, PT powder and PZT solution were mixed in a ball mill machine for more than 24 hours. After it was thoroughly mixed, it was sprayed onto the titanium substrate using an automatic spray machine. The dimensions of the titanium substrate were 30 mm, 30 mm, and 3 mm in length, width, and height. After spraying, they were dried in an electric furnace at 150°C and sintered at 400°C for 5 min each. The spraying, drying, and sintering processes were repeated until the thickness of the PT/PZT film reached 50µm. After deposition, the film was poled by corona discharge. During the poling, the humidity in the equipment was adjusted to more than 80% and 20% depending on the presence of nitrogen, and a negative corona discharge of 40 kV was applied for 5min. After that, the upper electrode was fabricated using a silver pencil. The optical image of the fabricated PT/PZT film is shown in Fig.1.



Fig.1 PT/PZT fabricated on a titanium substrate

3. Experimental Procedure

For the PT/PZT ultrasonic transducer, the polarization system was filled with nitrogen and the humidity was kept below 20% and above 80% during the polarization. The voltage application time was 5 minutes for both polarizations, and the room temperature was 27° C, so that the conditions were the same except for the humidity. The ultrasonic waveforms of each sample were recorded by pulse echo method using a digital oscilloscope. The gain of the pulsar receiver used in the measurement was 20dB for both samples. The

ultrasound response measurement diagram is shown in **Fig.2.**

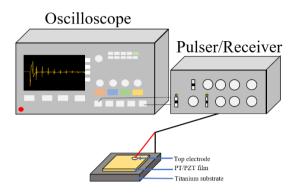


Fig.2 The ultrasound measurement setup.

4. Experimental Results

The ultrasonic responses at room temperature of samples poled at 20% humidity and samples poled at more than 80% humidity are shown in **Figs.3**. and **4**. The gain of the pulser receiver used to measure the ultrasonic response was measured at 20 dB for both samples. The d_{33} of the sample poled at 80% humidity was -1.3pC/N, and the d_{33} of the sample poled at 20% humidity was -5.5pC/N. From the measurement results, it can be said that the It was found that by using nitrogen to decrease the humidity during poling, the polarization could be humidity at room temperature done efficiently. In the future, other humidity control methods such as dry air will be used for similar experiments.

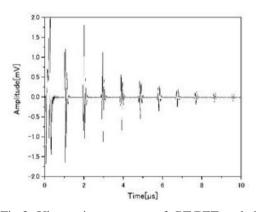


Fig.3 Ultrasonic response of PT/PZT poled at 20% humidity at room temperature.

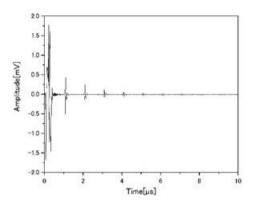


Fig.4 Ultrasonic response of PT/PZT poled at 80% humidity at room temperature

4. Conclusion

In this study, a PT/PZT ultrasonic transducer was used on a titanium substrate using the Sol-gel spray method. The ultrasonic response of the sample was measured in two patterns of humidity, below 20% and above 80%, by filling the polarizer with nitrogen during the polarization process. From the measurement results, it was found that the high humidity method clearly had a negative effect on the performance. And it was found that the surrounding environmental factors in fabricating the ultrasonic transducer, at least humidity, had a relationship in the polarization process. Therefore, in countries and regions where humidity is high, such as Japan, it is necessary to take into account the surrounding environment during the fabrication process and devise a method that is not affected by the climate and weather of the fabrication site. Although environmental factors other than humidity may also affect the performance of ultrasonic transducers, it can be said that we were able to discover one of the optimal environmental factors for fabrication in this experiment. In the future, the same experiments will be conducted using poling voltage, relationship with temperature during poling, and humidity control methods other than nitrogen.

References

- 1. K. Uchino, Ferroelectric devices, Marcel Dekker: New York, (2000) 145.
- 2. M. Kobayashi, T. R. Olding, M. Sayer, and C.-K. Jen: Ultrasonic **39** (2002) 675.
- M. Kobayashi, M. Furukawa: Jpn.DanseihaSoshiGijutu150Iinkai157 (2019) 31
- 4. T. Inoue and M. Kobayashi: Jpn. J. Appl. Phys. **53** 07KC11(2014)
- 5. K. Hirakawa, K. Nakatsuma and M. Kobayashi: Proc. 2020 USE