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Poling conditions of $PbTiO_3/TiO_2$

$PbTiO_3/TiO_2$ の分極条件に関する研究

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1. Introduction

Previous studies have shown that $PbTiO_3$ (PT) is sensitive but difficult to polarize[1-5]. Polarization is less likely to occur at room temperature than at high temperatures. PT/PZT had high sensitivity when it was polarized at high temperatures. However, as mentioned before, the sensitivity was low when oy was poled at room temperature. It was caused by high coercive field. In past study, experiments were carried out using TiO_2, a sol-gel solution with high resistivity, with $Bi_4Ti_3O_{12}$ powders, and efficient poling was succesfully carried out at room temperature.[6] Therefore, it was expected that PT powder phase would be poled efficiently at room temperature when TiO_2 was used as a sol-gel solution. The material properties of the $Pb(Zr,Ti)O_3$ (PZT), PT, and TiO_2 are shown in **Table I.** 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 PT based sol-gel composite could be poled at room tempetature efficiently. In this experiment, PT/TiO_2 sol-gel composite films were fabricated by firing at 400°C, poled at room temperature and high temperature, and ultrasnic performance was compared.

Table I. Material properties of each piezoelectric powder

	PZT	PT	TiO_2
Curie temperature (K)	573	763	
Dielectric constant	2100	100	150
d_{33} (10^{-12} m/V)	410	47	
g_{33} (10^{-3} Vm/N)	22	53	

2. Sample fabrication

PT/TiO_2 sol-gel composite films were fabricated by sol-gel splay technique. The PT/TiO_2 sample fabrication process is shown in **Fig. 1**. First, PT powders and TiO_2 sol-gel solution were mixed appropriately and the mixture was milled for more than 24 hours to obtain an appropriate viscosity for spray coating process and ensure mixture uniformity. Then, the mixture was sprayed on titanium substrates by an air brush. The titanium substrate had a length of 30 mm, a width of 30 mm, and a thickness of 3 mm. This substrate was chosen because of low thermal capacitance and high temperature durability. After spray coating, the samples were dried at 150°C in an electrical oven and sintered at 400°C in an electrical furnace for 5 minutes, respectively. The spray coating process and the thermal processes were repeated until the film thickness of PT/TiO_2 reaches 50μm. Coating process were divided to suppress internal stress which could cause cracks. After film fabrication, poling was operated by corona discharge. The distance between the electrodes is 2.5cm.The output voltage of the power supply was 31kV at room temperature and 32kV at high temperature. Corona discharge poling concept is shown in **Fig. 2**. Optical image of PT/TiO_2 film onto titanium substrate is shown in **Figs. 3** and **4**. In Fig. 3, the sample was poled at room temperature, and in Fig. 4, the sample was poled at high temperature, 40°C 0. There was no significant difference between the two samples by appearance.

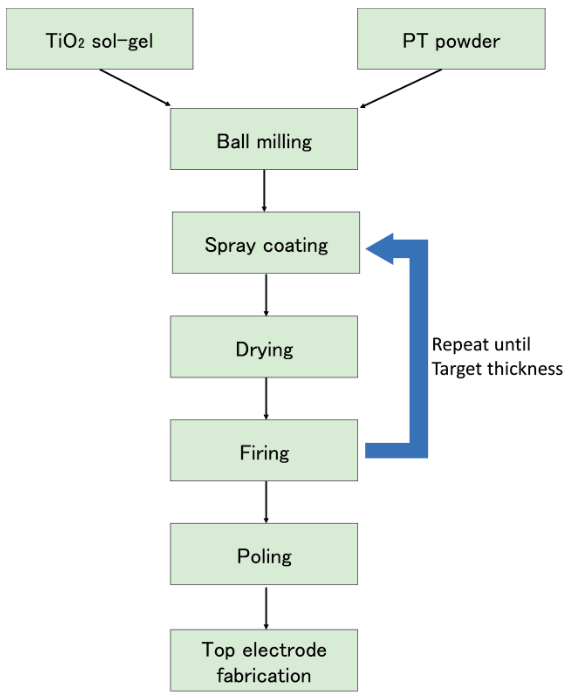

Fig. 1. Fabrication process of PT/TiO_2 sample.

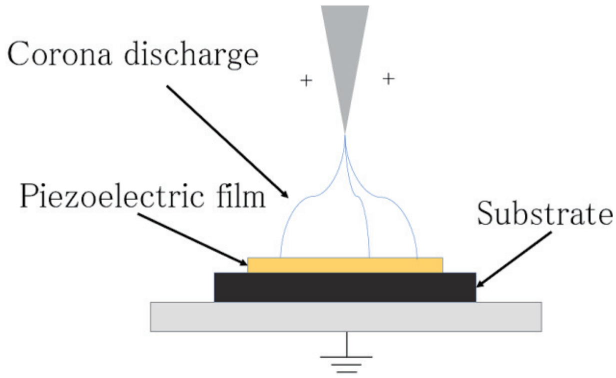

Fig. 2. Corona discharge poling concept.

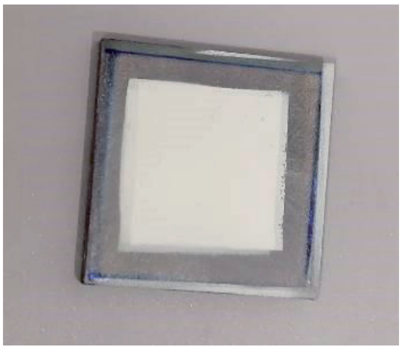

Fig. 3. Optical image of PT/TiO_2 sample fabricated on 3mm thick titanium substrate and poled at RT.

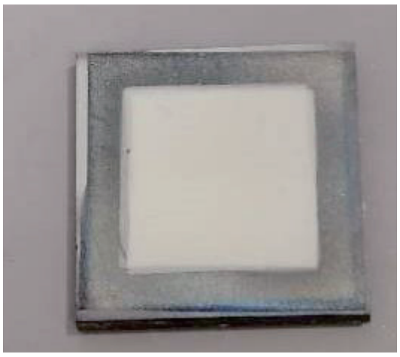

Fig. 4. Optical image of PT/TiO_2 sample fabricated on 3mm thick titanium substrate and poled at 400°C.

3. Experimental results

The piezoelectric constant d_{33} was measured by a ZJ3B piezo d33 meter and the values were 1.4pC/N for room temperature poling and 9.4pC/N at high temperature poling, respectively. The sample poled at high temperature showed higher value than the one poled at room temperature. It indicated that poling degree became lower when the sample was poled at room temperature.

Ultrasonic measurement was carried out in pulse-echo mode, as shown in **Fig. 5**. A digital oscilloscope was used to record measurement results. As a result, signal amplitude of the sample poled at room temperature was about 30dB lower than the sample poled at high temperature. This result was matched with d_{33} measurement results.

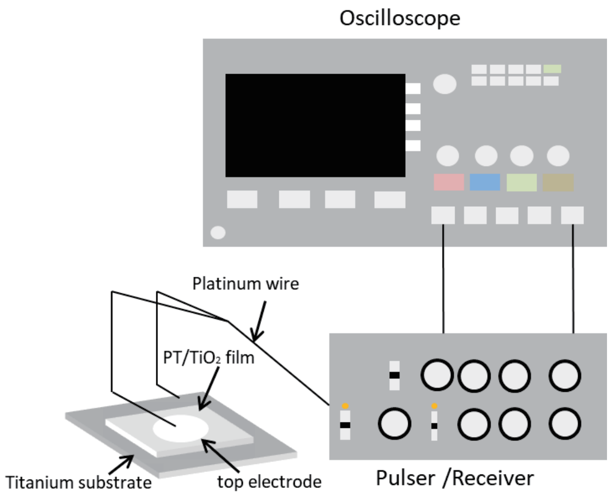

Fig. 5. The Ultrasonic measurement setup.

5. Conclusion

PT/TiO_2 films were fabricated and poled at room temperature and high temperature. As a result, the sample poled at room temperature showed lower d_{33} value and about 30dB lower signal amplitude than the sample poled at high temperature. In the future, it is necessary to improve film quality by automatic spray coating and confirm the reproducibility of this tendency.

References

1. T.Kibe, K.Kimoto, M.Kobayashi, Proc.IEEE Int. Ultrason.Symp.(2016) 980.
2. K.Kiyofuji,K.Kimoto,M.Kobayashi,Proc.Symp. Ultrasonic Electronics, (2016) 2P11.
3. Y.Inada,T.Inoue,M.Kobayashi,H.Nagata,and T.Takenaka:Jpn.J.Appl.Phys.53(2013)07KB10.
4. M.Kobayashi, C.K. Jen, J.F. Bussiere, and K.T. Wu: NDT & E Int. 42 (2009) 157.
5. D.Fu,K.Suzuki,andK.Kato: Jpn.J.Appl.Phys **42** (1994)
6. S. Nozawa, T. Yamamoto and M. Kobayashi: IEEE Inter. Ultrason. Symp. Proc, (2018).