

# Thickness Control of Piezoelectric Film Made by Stencil Printing

ステンシル印刷法による圧電膜の膜厚制御に関する研究

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## 1. Introduction

In various industries such as power plants, ultrasonic non-destructive testing (NDT) is widely used because of cost effectiveness and sub-surface defect/flaw detection capability. Piezoelectric films made of sol-gel composites could be useful as ultrasonic transducers due to high temperature durability, reasonable signal strength, curved surface suitability, relatively low center frequency such as 2-50MHz, and high signal-to-noise ratio (SNR).<sup>1)</sup> However, traditional spray technique required multiple spray coating and thermal treatments until desired film thickness was obtained in order to achieve desired frequency without crack. If thickness limitation by one coating process augmented, fabrication time could be reduced so that it would be more appropriate for industrial applications.

Stencil printing is fast and simple method and it could be possible to fabricate thick films by only one coating process.<sup>2-3)</sup> When a sol-gel solution was used for stencil printing, it became more condensed than that for spray technique because suitable viscosity for stencil printing was higher. In the previous studies, Pb(Zr,Ti)O<sub>3</sub> (PZT)/PZT, Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> (BiT)/PZT, and PbTiO<sub>3</sub> (PT)/PZT films were successfully fabricated by stencil printing.<sup>4)</sup> By only one stencil printing process, film thickness and center frequency were obtained ~70-80μm and ~10-23MHz, respectively, however, it was necessary to develop more low-frequency ultrasonic transducers in order to inspect under severe high temperature situations. Therefore, thickness control of sol-gel composite film fabrication by stencil printing was desired.

In this study, thickness increase of PZT/PZT film fabricated by stencil printing was attempted. After the fabrication, the sample was heated by a hot plate and ultrasonic performance, such as reflected echoes, and sensitivity at various temperatures were compared and discussed.

## 2. Fabrication process

Stencil mask was fabricated by superimposing three conventional 60μm thick sheets in order to increase the film thickness. Polyethylene terephthalate sheet was used as mask material due to patterning facility and low cost. The mask pattern was a circle with ~15 mm diameter. The mask pattern was made by a circle cutter. Piezoelectric film was fabricated onto titanium substrate with dimensions of ~3mm thickness, ~30mm length, and ~30mm width. Titanium substrate was selected because of high temperature durability. 3mm thick substrate was chosen because of heating facility by a hot plate and black zone investigation.

After the stencil mask and the substrate were prepared, the stencil mask was placed onto a substrate and covered with paint material, i.e. mixture of PZT powder and PZT sol-gel solution, by a squeegee. A metal squeegee was used in order to supply high and uniform force onto paint. After stencil printing process, drying process at 150°C for 5 minutes by a hot plate and annealing process at 650°C for 5 minutes by a furnace were followed. These heating processes were similar to spray technique. After thermal treatments, a poling process was carried out. Poling was executed by positive corona discharge at atmospheric pressure. The corona discharge was used because it could apply high electrical fields without dielectric breakdown. Finally, top electrode fabrication process was followed.

## 3. Experimental results

### 3.1. Film characterization

An optical image of the sample was shown in **Fig. 1**. Film thickness of the sample was ~160μm measured by micrometer. The  $d_{33}$  value was ~49pC/N measured by ZJ-3EN Piezo d33 meter. The frequency characteristic of ultrasonic response of the sample was shown in **Fig. 2**. Center frequency was ~3.7MHz, therefore, frequency characteristic of the sample was relatively low.



Fig. 1 PZT/PZT film optical image made by stencil printing onto ~3mm thick titanium substrate.

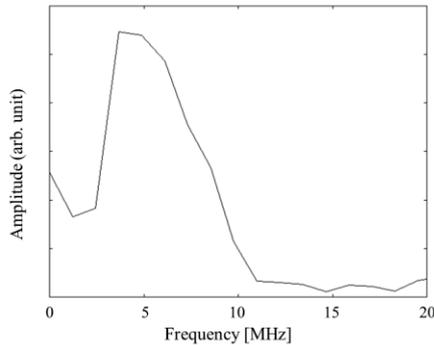


Fig. 2 Frequency characteristic of ultrasonic response of PZT/PZT film made by stencil printing.

### 3.2. Thermal cycle test

Thermal cycle test from room temperature to 100°C was carried out three times in order to investigate durability and ultrasonic performance at high temperature. The sample was heated by hot plate, and the ultrasonic response was measured by pulse-echo mode. During the test, film surface temperature was measured by thermography.

Ultrasonic response of PZT/PZT film onto 3mm thick titanium substrate fabricated by stencil printing at the end of thermal cycle test was showed in Fig. 3. Throughout three thermal cycles, the sample presented clear reflected echoes with high SNR.

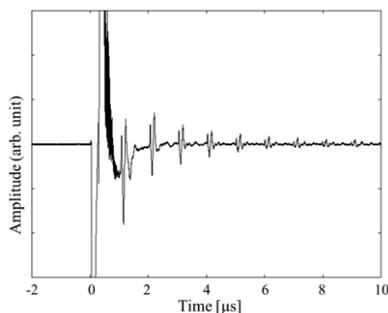


Fig. 3 Ultrasonic response of PZT/PZT film made by stencil printing at 100°C in 3rd thermal cycle.

Temperature dependency of sensitivity during thermal cycle test for PZT/PZT film fabricated by stencil printing was shown in Fig. 4. In this study,

the sensitivity was determined as the negative value of the true gain of pulser/receiver to obtain 1 Vp-p for second reflected echoes. The sensitivity was set as negative value for intuitive comprehension. The sensitivity of the sample between second and third cycles was lower than that of first cycle because of depoling effect during first cycle. PZT powder phase and PZT sol-gel phase were suffered by depoling effect. The sensitivity was nearly identical between second and third cycles, and therefore high temperature durability and high enough adhesion strength of the sample by stencil printing was demonstrated up to 100°C in spite of increased film thickness.

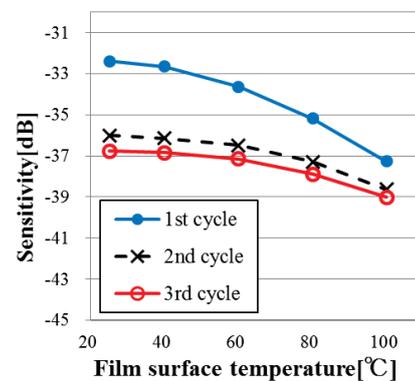


Fig. 4 Temperature dependency of sensitivity during thermal cycle test for PZT/PZT film fabricated by stencil printing.

### 4. Conclusions

PZT/PZT film was fabricated onto 3mm thick titanium substrate. ~160μm thickness was obtained by only one stencil printing process. Center frequency was ~3.7MHz so that frequency characteristic of the sample was relatively low. During thermal cycle test, high temperature durability and high enough adhesion strength was demonstrated because sensitivity was stable up to 100°C. Therefore, PZT/PZT film which was increased the thickness made by stencil printing could be a good piezoelectric material for low cost ultrasonic transducers.

### References

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