Bi₄Ti₃O₁₂ Based Thick Film Fabrication by Stencil Printing

ステンシル印刷法による Bi₄Ti₃O₁₂ ベース厚膜の作製に関する 研究

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1. Introduction

Ultrasonic non-destructive testing (NDT) is widely used in various industries such as power plants because of sub-surface defect/flaw detection capability and cost effectiveness. Ultrasonic transducers made by sol-gel composite materials could be good canditates due to curved surface suitability, high tempearature durability, relatively low center frequency such as 2-20MHz, reasonable signal strength, and high signal-to-noise ratio (SNR).¹⁾ However, by conventional spray coating technique, in order to achieve desired frequency, it requires multipul spray coating and thermal treatments, and in some case, it could be a concerned issue, especially for on-site fabrication under controlled area.

Stencil printing is fast and simple method and it could be possible to fabricate thick film by only one coating process.²⁻³⁾ The similar materials used for conventional sol-gel composites could be used for stencil printing, though it requires slight modification since suitable viscosity for spray coating and stencil printing is different. In the previous study, PZT/PZT thick films were succesfully fabricated by stencil pring, however, the signal strength was much lower than that by traditional spray coating method.⁴⁾ It was suspected that since PZT piezoelectric powder phase has high dielectric constant, even higher than that of PZT sol-gel phase, so that even slight decrease of PZT sol-gel phase dielectric constant could affect the ultrasonic performance significantly.

In this study, $Bi_4Ti_3O_{12}$ (BiT)/PZT thick film fabrication by stencil printing was attempted. In addition to BiT/PZT was one of the good canditates for long term ultrasonic NDT above 300°C, it was thought that BiT/PZT would receive less influence of decrease of PZT sol-gel phase dielectric constant, sicne dielectric constant of BiT powder phase was lower than that of that of PZT sol-gel phase.

2. Fabrication process

Plastic stencil masks with 100µm thick were prepared and used for patterning facility, curved surface suitability and low cost. Mask pattern was small circle with ~6mm diameter. Mask pattern was made by laser cutting. Films were fabricated on steel substrates with dimensions of 4.2mm thickness, ~50mm length, and ~50mm width, titanium substrates with dimensions of ~3mm thickness, ~30mm length, and ~30mm width,, and steel pipes with dimensions of ~4mm thickness, ~40mm outer diameter. Steel substrates were chosen because it was common material for industrial structures, whereas titanium substrates were also used for high temperature test.

After stencil masks and substrates were prepared, the stencil mask was placed on a substrate and covered with paint material, i.e. mixture of BiT powder and PZT sol-gel solution, by a squeegee. After stencil printing process, similar thermal treatments with traditional sol-gel composite materials, such as drying process at 150°C by a hot plate, annealing process at 650°C by a furnace, were followed, whereas drying process and annealing process were carried out by hair dryer and gas torch for the samples on steel pipes in order to simulate on-site fabrication. After thermal treatments, poling and top electrode fabrication processes were followed. Film thickness was measured by a micrometer. Ultrasonic responses were attempted to be measured for performance comparison purpose.

3. Experimental Results

3.1. Stencil printing of BiT/PZT on flat surfaces

First, BiT/PZT thick films were fabricated onto steel and titanium plates. An optical image of the sample was shown in **Fig. 1**. Film thickness of samples was varied between $\sim 120-150\mu m$. Ultrasonic response of the films were attempted to be monitored and clear reflected echoes were observed as shown in **Fig. 2** and **Fig. 3** from steel and titanium substrate, respectively. Center frequency of the samples onto steel plate and

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titanium plate was ~9.5MHz and ~4.6MHz, respectively. Since more gain was required for titanium substrates so that lower center frequency of titanium substrate sample was caused by attenuation. It should be mentioned that almost same signal strength by traditional spray coating process was achieved, even though fabrication time was much shorter because of no repetition of coating process and thermal treatments. Further investigation was required for high temperature performance and thermal cycle durability.



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



Fig.2 Ultrasonic response on ~4.2mm thick steel substrate.



Fig.3 Ultrasonic response on ~3mm thick titanium substrate.

3.2. Stencil printing of BiT/PZT on curved surface

BiT/PZT thick films were fabricated onto steel pipes as well. An optical image of the sample was shown in **Fig. 4**. The thick film was successfully fabricated onto pipe with high curvature such as ~40mm outer diameter. Ultrasonic response of the films was shown in **Fig. 5**. Center frequency was ~7.0MHz. It should be noticed that almost same signal strength as flat substrate was achieved, even though during thermal treatments, hair dryer and gas torch were used instead of hot plate and furnace, in order to demonstrate on-site fabrication capability. Those results indicated that on-site BiT/PZT fabrication could be practical by stencil printing approach.



Fig.4 BiT/PZT film optical images made by stencil printing onto ~40mm outer diameter steel pipe.



Fig.5 Ultrasonic response on ~4mm thick steel pipe substrate.

4. Conclusions

BiT/PZT thick films were fabricated onto flat and curved surfaces of steel and titanium substrates. 100µm over thickness was achieved by only one stencil printing process. Signal strength was significantly improved than previous stencil printing study and almost same signal strength by traditional spray coating process was obtained. Fabrication onto high curvature such as ~40mm outer diameter was successfully performed without conventional furnace so that BiT/PZT ultrasonic transducer by stencil printing became more realistic approach for on-site fabrication.

References

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