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Selective Sintering of Sol-Gel Composite Films by Commercial Microwave Oven

家庭用電子レンジによるゾルゲル複合体圧電膜の選択的焼成に関する研究

Makiko Kobayashi[1†] and Makoto Matsumoto[1] ([1]Grad. School Sci. Technol., Kumamoto Univ.)
小林牧子 [1†]，松本真 [1] ([1]熊本大院　自然科学研究科)

1. Introduction

Ultrasonic imaging (UI) can observe the internal object without damaging. In UI, high-frequency of the transducer is required for high resolution. Sol-gel composite film could be good material for high frequency ultrasonic transducer and to achieve the high frequency ultrasonic transducer by sol-gel composite method, thickness and porosity control is required and it is preferable to fabricate thin spherical concave surface substrate for acoustic wave focus. However, by traditional sintering method, such as furnace, the deformation of the thin substrate was not avoidable. Microwave sintering could be a good method to heat dielectric material homogeneously and less thermal damage for the substrate. However, the experimental system of microwave sintering in the past was relatively complex and installation cost was high.

In this study, PZT/PZT transducer fabrication by sol-gel composite method using the microwave sintering by a domestic microwave oven was attempted. In additon, the performance of the fabricated transduers were evaluated.

2. Fabrication process

Ultrasonic transducers made by PZT/PZT sol-gel composite films were manufactured. The mixture of PZT sol-gel solution and PZT piezoelectric powder were ball milled for more than 24 hours. Two kinds of PZT powders with different dielectric constant were used. For convenience, PZT-1 and PZT-2 indicated the PZT powder with the lower dielectric constant and with higher dielectric constant, respectively, as shown in **Table I**. Then the mixtures were sprayed onto the substrates. After spray coating, drying process at 150℃ was carried out. Conventional sintering process was carried out in an electric furnace at 650℃. In this experiment, sintering process was operated by a domestic microwave oven with 700W for 10 min. Those spray coating process and thermal process were repeated four times in this experiment. After poling at room temperature, ~2mm diameter top electrodes were fabricated onto piezoelectric films.

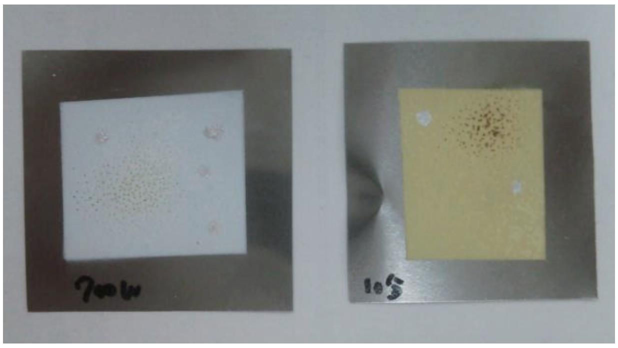
Table I. Characteristics of PZT

	ε_r	d_{33}[pC/N]	Q_m	k_{33}(%)	k_t(%)
PZT-1	1800	400	75	70	62
PZT-2	4720	603	70	68	47

3. Experimental result

3.1. The dielectric constant comparison

First, PZT-1/PZT and PZT-2/PZT thick films were fabricated with microwave sintering onto stainless substrates, respectively. Stainless substrate had ~50μm thick, ~300mm length, and ~300mm width. The optical images of the sample were shown in **Fig.1**. The films with reasonable smoothness were obtained. Discoloration and deformation of the substrate was not observed, which usually occurred by an electrical furnace sintering at 650℃.



(a)PZT-1/PZT (b)PZT-2/PZT

Fig.1: PZT/PZT samples onto stainless substrates sintered by a domestic microwave oven.

The piezoelectric film thickness of samples was ~20μm measured by micrometer. Piezoelectric constants d_{33} of PZT-1/PZT sample and PZT-2/PZT sample were measured by ZJ-3EN Piezo d33 meter supplied by Institute of Acoustic, Chinese Academy of Science and the values were ~1.5pC/N and ~14.3pC/N, respectively. It seems that high dielectric constant PZT powder assisted microwave sintering by dielectric loss. Ultrasonic monitoring of 3mm thick titanium plate was attempted with couplant, however it was impossible to obtain multiple reflected echoes. It might happen because of high frequency of fabricated transducer.

3.2. Fabrication onto titanium substrate

In order to avoid couplant, PZT-2/PZT thick film was fabricated onto a titanium substrate directly. Titanium substrate had ~3mm thick, ~300mm length, and ~300mm width. The optical image of the PZT-2/PZT samples with microwave sintering is shown in **Fig.2**. The films with reasonable smoothness were obtained as before. The piezoelectric film thickness was ~25μm. Piezoelectric constant d_{33} of titanium substrate sample was ~3.5pC/N. Ultrasonic response of PZT-2/PZT sample onto titanium substrate was observed as shown in **Fig.3** from titanium substrate. Clear multiple echoes from back surface of titanium substrate were observed with reasonable signal to noise ratio.

Fig.2: PZT-2/PZT sample onto a titanium substrate sintered by a domestic microwave oven.

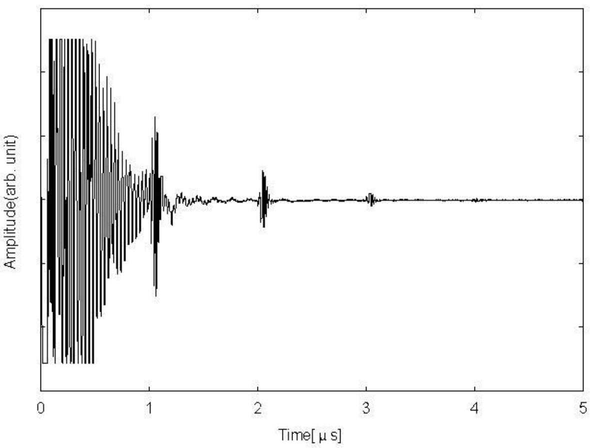


Fig.3: Ultrasonic response of PZT-2/PZT sample onto ~3mm titanium substrate

FFT of the second echo was carried out and the result is shown in **Fig.4**. The center frequency of the sample onto a titanium substrate was 43MHz. From this result, PZT-2/PZT sample with microwave sintering had a high frequency characteristic, as expected, with reasonable broad bandwidth.

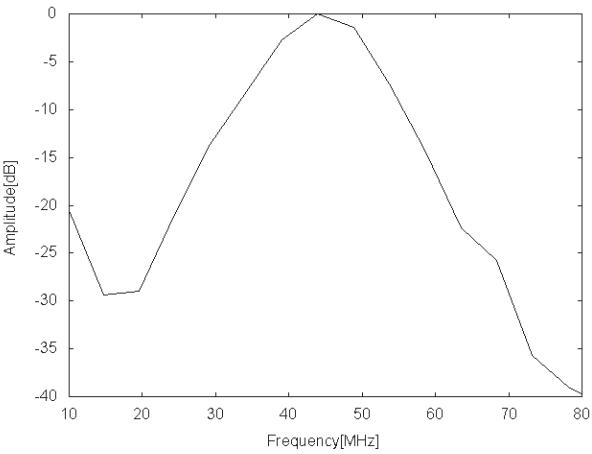


Fig.4: Frequency characteristic of PZT-2/PZT sample onto titanium substrate

4. Conclusion

PZT/PZT thick films were fabricated onto stainless and titanium substrates with domestic microwave oven sintering. It seems that microwave sintering with higher dielectric constant PZT powder was efficiently carried out. Piezoelectric constants d_{33} of titanium substrate and stainless substrate were ~3.5pC/N and ~14.3pC/N, respectively. Ultrasonic response of PZT-2/PZT sample onto titanium substrate was observed. Center frequency of the sample onto titanium plate was 43MHz and bandwidth was large. Therefore it could be concluded that microwave sintering by domestic microwave oven was successfully operated.

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kobayashi@cs.kumamoto-u.ac.jp