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Influence on Sol-Gel Composite Properties caused by Piezoelectric Powder Phase with Different Dielectric Constants

異なる誘電率の圧電粉体相によるゾルゲル複合体全体特性への影響

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1. Introduction

Sol-gel composite materials was developed in order to fabricate thick film without crack. [1)] Sol-gel composite materials could be one of the piezoelectric material canditates for high temperature ultrasonic transducers, because of high tempearature durability, relatively low center frequency such as 2-20MHz, reasonable signal strength, and high signal-to-noise ratio (SNR). [1-4)]

Sol-gel composite material is made by 2 row materials: sol-gel solution and piezoelectric powder, and each phases could affect the whole composite properties. In these days, in order to obtain the highest signal strength at specific operation temperature, it is desired to develop new sol-gel composite materials optimised for narrow specific temperature. Therefore, it becomes more important to know the effects caused by each phase.

It was suspected from experimental results that the dielectric constant relationship between sol-gel solution and piezoelectric powder was important. In the past research, several different powders, such as lead zirconate titanate (PZT) powders, bismuth titanate powders, and lithium niobate powders, were mixed with same PZT sol-gel solution, and each composite material showed different operation temperatures and different signal strength. [4)] However, it is difficult to determine the dielectric constant effect of piezoelectric powder phase from those experiments, since 3 kinds of piezoelectric powders have very different characteristics. In this research, dielectric constant effect of powder phase was investigated using 2 kinds of PZT powders, very similar properties except dielectric constant.

2. Materials and Experimental procedures

2 kinds of PZT powders, P-7 and P-7B from Murata Manufacturing Co., Ltd., were prepared for this research by. The material properties of each powder are shown in **Table I**. It is noted that P-7B has twice higher dielectric constant than P-7. Concerning piezoelectric constant d, P-7B has higher values than P-7, whereas P-7 has higher Curie point. There is no significant difference in other properties such as electromechanical coupling coefficients k and mechanical quality factor Q_m.

Table I Material properties used in this experiment

Material Property	P-7	P-7B
ε_{33} (relative)	2100	4720
k_{33} (%)	71	68
k_t (%)	51	47
d_{33} (10^{-12}m/V)	410	603
Q_m	80	70
T_C (°C)	300	180

Spray coating technique was chosen as fabrication method, since it could be useful for many industrial applications because of curved surface suitability.[3-4)] These 2 kinds of powders were mixed with PZT sol-gel solution. After ball milling, the mixture was sprayed onto steel substrates with dimensions of 4.2mm thickness, ~50mm length, and ~50mm width. Thermal treatments, drying process and annealing process at 150°C by a hot plate and 650°C by a furnace, were followed. Spray coating and thermal treatments were repeated 5 times. Poling was operated after film fabrication process in order to achieve piezoelectricity of the film.

Film thickness was measured by a micrometer. Silver top electrodes were made by a conductive pen. Capacitance was measured by a LCR meter in order to calculate dielectric constant. Ultrasonic responses with different top electrode size were measured for performance comparison purpose between 2 kinds of sol-gel composite materials, P-7/PZT and P-7B/PZT.

3. Results and Discussions

Optical images of each material were shown in **Fig. 1**. Except film color difference due to piezoelectric powders, no significant difference was observed. Film thickness of P-7/PZT and P-7B/PZT were ~70μm and ~50μm, respectively. Film thickness difference was caused by spray coating

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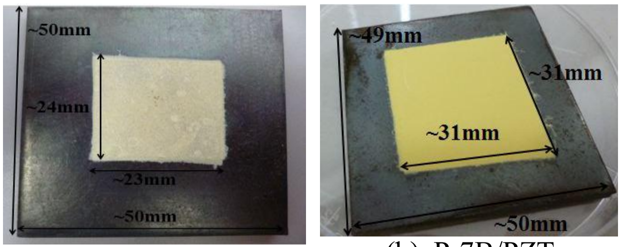
process. The calculated relative dielectric constants were ~270 and ~230, respectively. This difference was much smaller than that of row piezoelectric powders. Other components of sol-gel composite materials, air and PZT sol-gel phase, have also significant effect to determine the dielectric constant of sol-gel composite materials.



(a) P-7/PZT (b) P-7B/PZT

Fig.1 Optical images of the samples.

Typical ultrasonic response of P-7/PZT and P-7B/PZT were shown in **Fig. 2** and **Fig. 3**, respectively. Clear reflected echoes from bottom surface of the substrates were confirmed and it indicated that both samples showed broadband characteristic and high SNR. P-7B/PZT seems to have higher frequency component than P-7/PZT, and it resulted from lower film thickness.

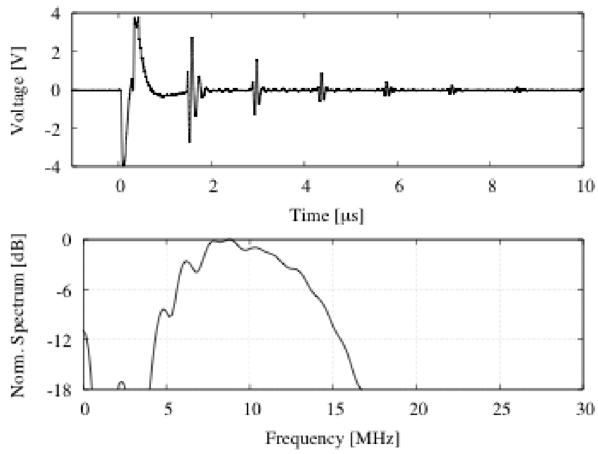


Fig.2 Ultrasonic response of P-7/PZT; upper) in time domain, bottom) in frequency domain.

Ultrasonic responses with variable top electrode size were recorded to compare ultrasonic performance of those materials. Used gain to obtain $6V_{p\text{-}p}$ for each top electrode size and polynomial fitting curves were shown in **Fig. 4**, respectively. The tendency was very similar. It is noted that signal strength of P-7/PZT was higher than P-7B/PZT. It might be caused from high dielectric constant of P-7B. Since dielectric constant of PZT sol-gel is lower than bulk ceramics (~200)[2], electrical field might not be sufficiently applied to P-7B during poling and operation. However, the number of the sample was too small to make a conclusion, and it could be improved if poling conditions are modified.

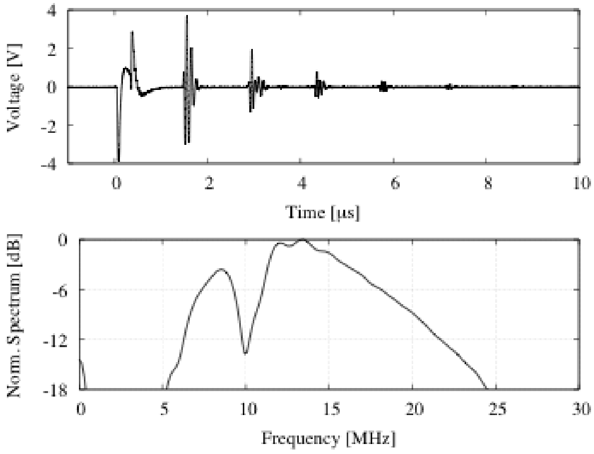


Fig.3 Ultrasonic response of P-7B/PZT; upper) in time domain, bottom) in frequency domain.

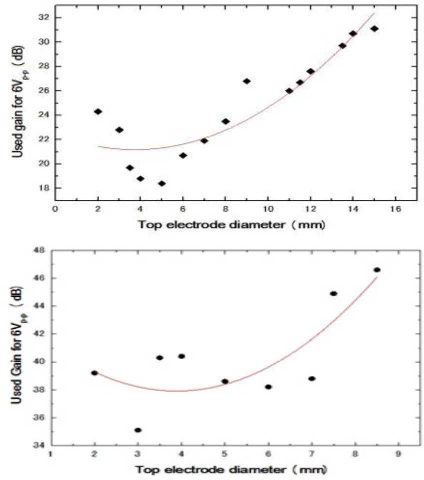


Fig. 4 Top electrode dependence of signal strength of upper) P-7/PZT bottom) P-7B/PZT

4. Conclusions

In order to investigate dielectric constant effect of powder phase, 2 kinds of sol-gel composite film were fabricated using different dielectric constant PZT powders. High dielectric constant powder might deteriorate signal strength of ultrasonic transducers made by sol-gel composite.

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