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Publisher's version / Version de l'éditeur:
https://doi.org/10.1109/ULTSYM.2006.213
Proceedings of the Ultrasonics Symposium (IEEE 2006), pp. 816-819, 2006-10-03

Fabrication and Characterization of Thick Film Piezoelectric Ultrasonic Transducers

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Abstract— **Lead-zirconate-titanate fine powders mixed with chemical solutions have been used to fabricate 50µm thick films. The fabrication methods include sol-gel spray, tape casting and screen printing. The substrates were 75µm thick stainless steel membrane. The sol-gel spray technique included multiple coatings and each coating involved spray and heat treatment. Each layer was between 5 and 15µm thick. For tape casting and screen printing, only one layer of 50µm was directly cast and printed onto the substrate. Then films were treated in the furnace up to 650°C for 5 to 30minutes in this study. These films became piezoelectric through a corona poling technique. The relative dielectric constant of the sol-gel sprayed films was around 130 and those of the tape casting and screen printing ones were lower than 100. The capability of these films as piezoelectric flexible ultrasonic transducers was confirmed in the pulse-echo mode at several MHz for non-destructive testing at room temperature and 150°C.**

Keywords-component; flexible transducer; nondestrauctive testing; high temperature; sol-gel spray; tape casting; screen printing

I. Introduction

Many vehicles for transportation and utilities in various engineering industries desire to have their life-span extended. Nondestructive testing (NDT) [1-4] of these vehicles and utilities are commonly performed to identify, characterize and assess defects and damages so that proper repairs can be performed. Ultrasonic techniques are routinely used for such NDT purposes because of their subsurface inspection capabilities, fast inspection speed, simplicity and ease of operation. In many applications, ultrasonic inspections may need to be applied to curved surfaces or complex geometries. It is known that conventional planar ultrasonic transducers (UTs) show poor inspection performance on curved surfaces [1,3]. Flexible UTs are desired for such NDT applications. The flexibility may conform the flexible UTs with the curved shapes of the sample so that the transmitted ultrasonic energy can be maximized for improved diagnoses [3]. Furthermore, numerous industrial applications require high temperature (HT) operation of UTs to perform in-situ characterization of materials, real-time process diagnostics, and NDT [4,5]. Thus, the research interest here is to develop flexible UTs for potential NDT applications on substrates with simple and complex shapes at room and high temperature.

In this investigation, flexible UTs, consisting of a 75µm thick stainless steel (SS) foil, a 50µm thick piezoelectric film and a top electrode [6], will be made. Three techniques, sol-gel spray [7], tape casting [8], and screen printing [9] will be used to fabricate the piezoelectric film. Ultrasonic performances of the flexible UT made by the above three techniques will be compared at room temperature and 150°C.

II. Fabrication

A. Sol-gel Spray

The detailed fabrication process of sol-gel spray technique can be found in the previous publication [7]. Piezoelectric powders, lead-zirconate-titanate (PZT) in this study, were purchased. Fine and submicron size powders were dispersed into PZT sol-gel solution by the ball milling method to achieve the paint for spray. An airbrush was then used to spray the PZT/PZT sol-gel composite directly onto 34mm width, 34mm length, and 75µm thick SS foils. Paper masks were used to serve as the 26mm×26mm shadow mask during the spray coating. After each coating, thermal treatments such as drying, firing and annealing up to 650°C were carried out. Multiple layers were made in order to reach desired 50µm film thickness.

B. Tape Casting and Screen Printing

Alcohol based solvent and organic binder was first added to the purchased PZT powders. Then the ball milling was operated to achieve the appropriate viscosity as slurry of tape casting and/or screen printing. SS foils of 75µm thick, 25mm wide and 52mm long were used as substrates for tape casting.

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Rectangular masks of 23mm×45mm square patterns were cut from 120μm thick masking tapes and placed onto the above mentioned SS foils. PZT slurry was then applied to the exposed SS surfaces. A straight edge was held at around 20° angle and manually dragged along the long side of the mask. The PZT slurry was also screen printed in a mesh, screen onto the same substrate with the same mask as tape casting. The masked metal strips were fixed slightly below a screen. The PZT slurry was applied on top of the screen. A blunt plastic knife was pressed on the screen and then dragged manually.

After tape casting or screen printing, thermal treatments up to 650°C were operated. It is noted that the films without further sintering at temperature higher than 1000°C were used as piezoelectric films in order to assure the flexibility and to avoid microstructure change of SS foils. Such approach is the main difference between the reported tape casting [8] and screen printing [9] techniques and our approach presented here. Figs. 1 and 2 show the PZT films onto 75μm SS foil fabricated by tape casting and screen printing, respectively. By these two methods, 50μm thick piezoelectric films were fabricated only by one coating. It was found that these PZT films, especially made by screen printing, had poor mechanical strength and chemical resistance. Chemical treatments will be added for tape casting and screen printing process to improve the mechanical strength and chemical resistance but without increasing process temperature.

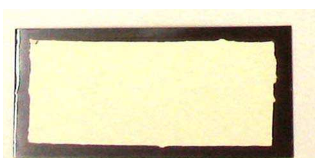


Figure 1. Photograph of a PZT film coated onto 75μm thick SS foil by tape casting.

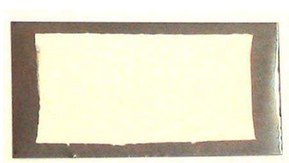


Figure 2. Photograph of a PZT film coated onto 75μm thick SS foil by screen printing.

C. Poling

Films of 50μm thickness were then electrically poled using a corona discharging technique [10]. For corona poling, the temperature of the SS foil was heated to around 120°C and a high positive voltage supplied from a 28kV DC power supply was fed into a thin and sharp needle located several centimeters above the 50μm thick PZT/PZT film coated on the SS foil, which serves as the ground electrode. The corona poling method was chosen because it could pole the piezoelectric powder mixed with low dielectric constant material efficiently over a large area. It should be mentioned that the traditional poling using two (top and bottom) electrodes were also attempted. But all the samples made by tape casting and screen printing were electrically short circuited after such traditional poling. Empirically it was found that during the traditional poling of piezoelectric composite consisting of PZT powders and low dielectric constant material, the leakage current was relatively high, and dielectric breakdown often occurs in short period. In this study PZT/PZT sol-gel sprayed films were poled both by corona and traditional poling methods. The sample poled by corona poling had about 15dB higher signal strength than that poled by traditional poling.

D. Top Electrode Fabrication

The top electrode of each UT was fabricated at room temperature. In this study, silver colloid was painted or sprayed onto piezoelectric films with paper mask. The thicknesses of the painted and sprayed silver top electrode were about 20μm and 2μm, respectively, with fairly good uniformity. For films made by tape casting and screen printing, the entire structure was sandwiched by polyimide films so that it can overcome the weak mechanical/chemical strength. In addition, the polyimide film was chosen because it can work at temperatures more than 350°C. Copper strips were used for electrical connection; one connected to the top silver colloid electrode and the other to the SS substrate as bottom electrode of the flexible UT. The PZT UT made by tape casting with electric wires and polyimide protection films is shown in Fig. 3.

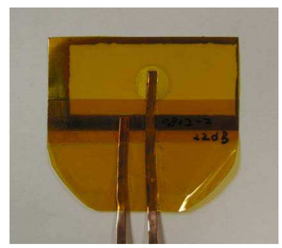


Figure 3. Picture of PZT flexible UT made by tape casting with electric wires and polyimide protection.

III. CHARACTERIZATION

The capacitances of every film were measured by a Hewlett Packard 4192A LF Impedance Analyzer at 1kHz in order to calculate the relative dielectric constant. The diameter of the top electrode was 9mm for each sample. The relative dielectric constants of 50μm thick flexible UT made by tape casting, screen printing, and sol-gel spray technique were about 55, 20, and 130, respectively. The dielectric constants of PZT bulk powder and sol-gel are 1800 and 300, respectively. The lower dielectric constants of flexible PZT UT made by sol-gel spray, tape casting and screen printing than that of bulk PZT may be higher porosity and existence of bonding material. It is noted that the bonding material of sol-gel spray technique is PZT sol-gel and it may result in higher dielectric constant than tape casting and screen printing because the dielectric constants of organic residue used in tape casting and screen printing are much lower than that of PZT sol-gel.

Longitudinal piezoelectric constant, d_{33}, and transverse piezoelectric constant, d_{31}, of the PZT/PZT composite film by sol-gel spray technique were obtained using a laser interferometer and an optical interferometer, respectively. The values of d_{33} and d_{31} were calculated as 30×10^{-12} m/V and -

26×10^{-12} m/V, respectively. It is expected that the PZT film made by tape casting may show similar results because of their similar ultrasonic performances which will be mentioned at later sections.

Figs. 4-6 show the SEM images of the PZT film made by sol-gel spray, tape casting, and screen printing, respectively. It is indicated in Figs. 3-5 that the grain size is less than 1μm and the film was not dense. Due to the porosity in the piezoelectric film, thin metallic membrane substrate and thin top electrodes, all UTs made by tape casting, screen printing, and sol-gel spray technique achieved some flexibility. This porosity also shows the good agreement with the low dielectric constant results.

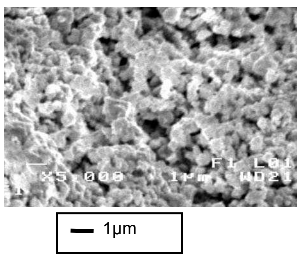


Figure 4. SEM image of PZT/PZT film made by sol-gel spray

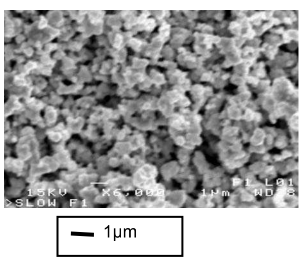


Figure 5. SEM image of PZT film made by tape casting

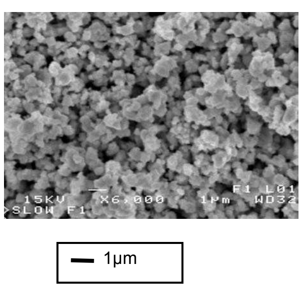


Figure 6. SEM image of PZT film made by screen printing

In order to compare the performance of the flexible UTs without polyimide protection layer at room temperature using sol-gel spray technique, tape casting and screen printing, the UTs were pressed onto a 13.8mm thick SS plate at room temperature. The ultrasonic couplant was placed between the probing side of the SS foil and SS plate. Figs. 7 (a) and (b) show the transducer response made by sol-gel spray technique in time and frequency domains, respectively. L_2, L_4 and L_6 are the first, second, and third round trip echoes through the thickness of the SS plate. The frequency at peak amplitude, the 6dB bandwidth and SNR of the L_2 echo are determined to be 6.1MHz, about 5.8MHz and 25dB, respectively. Figs. 8 (a) and (b) show the transducer response made by tape casting in time and frequency domains respectively, in pulse-echo mode at room temperature. The center frequency, the 6dB bandwidth and SNR of the L_2 echo are determined to be 3.7MHz, 3.3MHz and 20dB, respectively. Figs. 9 (a) and (b) show the transducer response made by screen printing in time and frequency domains respectively, in pulse-echo mode at room temperature. The frequency at peak amplitude, the 6dB bandwidth and SNR of the L_2 echo are determined to be 3.4MHz, 3.2MHz and 20dB, respectively. It was found out that the signal strengths of the UTs made by sol-gel spray technique and tape casting were about 20dB higher than that of the UT made by screen printing. The low capability of the screen printed UT may be related to the high porosity and high existence of organic residue. In our observation, the low signal strength and the broadband characteristic indicate that the film will suffer high ultrasonic loss. As it is mentioned before, further research of chemical treatment of the film may be required to improve the performance.

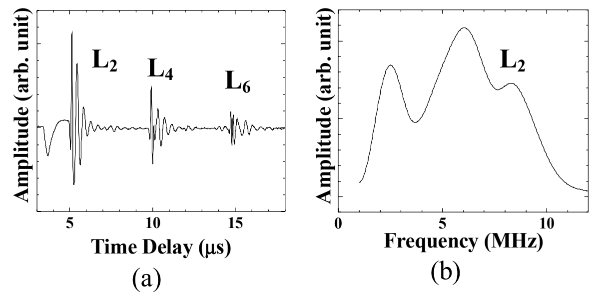


Figure 7. Room temperature response of flexible UT made by sol-gel spray technique in (a) time and (b) frequency domain for a 13.8mm thick SS plate.

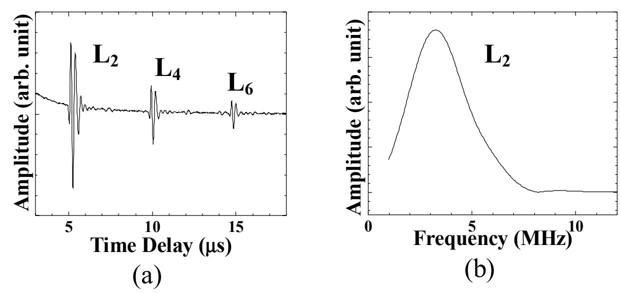


Figure 8. Room temperature response of flexible UT made by tape casting in (a) time and (b) frequency domain for a 13.8mm thick SS plate.

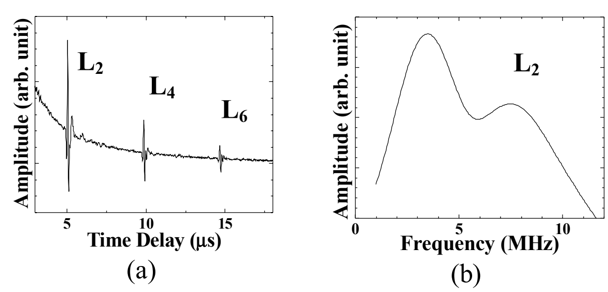
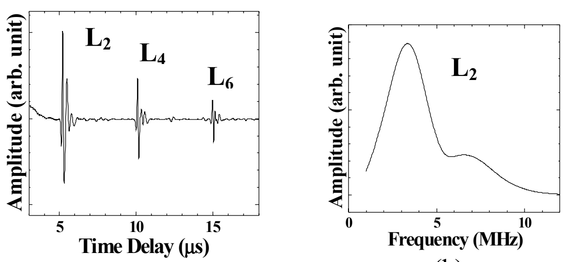


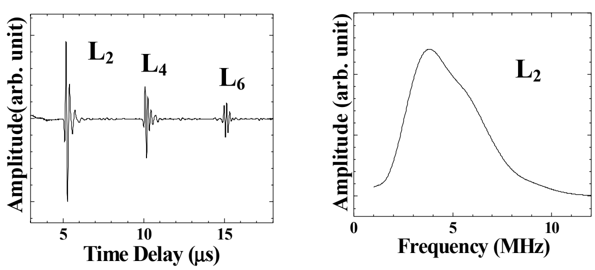
Figure 9. Room temperature response of flexible UT made by screen printing in (a) time and (b) frequency domain for a 13.8mm thick SS plate.

In order to demonstrate the performance of the flexible UT made by sol-gel spray technique and tape casting at elevated temperature with polyimide protection layer, the transducers, tape casting one was shown in Fig. 3, were pressed onto the same SS plate, which was used in the previous experiment but heated up to 150°C here. High temperature oil couplant was placed between the probing side of the SS foil and SS plate. Figs. 10 (a) and (b) show the transducer response made by sol-gel spray technique, in time and frequency domains respectively, in pulse-echo mode at 150°C. The center frequency, the 6dB band width and SNR of the L_2 echo are determined to be 4.6 MHz, 4.1MHz and 25dB, respectively. Figs. 11 (a) and (b) show the transducer response made by tape casting, in time and frequency domains respectively, in pulse-echo mode at 150°C. The center frequency, the 6dB band width and SNR of the L_2 echo are determined to be 4.4MHz, 3.7MHz and 25dB, respectively. Both UTs were about 5dB weaker than the measurement results at room temperature. These experimental results showed that ultrasonic performance of the PZT UT made by tape casting was similar to that made by sol-gel spray.



(a) (b)

Figure 10. At 150°C response of flexible UT made by sol-gel spray technique in (a) time and (b) frequency domain for a 13.8 mm thick SS plate.



(a) (b)

Figure 11. At 150°C response of flexible UT made by tape casting in (a) time and (b) frequency domain for a 13.8mm thick SS plate.

IV. CONCLUSIONS

50μm thick film flexible UTs were fabricated by sol-gel spray, tape casting and screen printing techniques. The substrates used were 75μm thick SS foils. The sol-gel spray technique included multiple coatings and each coating involved spray and heat treatment. It required commonly 4 or-5 layers to reach 50μm thickness. For tape casting and screen printing, only one layer of 50μm was directly cast and printed onto the substrate and then treated by the heat. All heat treatments were carried out in the furnace up to 650°C for 5 to 30 minutes. It is noted that in this investigation PZT films were fired at 650°C instead of fully sintered ones above 1000°C reported in [8,9] in order to ensure the flexibility of the flexible UTs and avoid the microstructure change of SS foil substrate. These films were made piezoelectric through a corona poling technique. It was found that the corona poling provided efficient poling without damaging the films.

The relative dielectric constant of the sprayed thick film was around 130 and those of the tape casting and screen printing ones were lower than 100. The reason for the lower dielectric constants of the UTs may be porosity and the existence of other phases such as PZT sol-gel or organic residue. Porosity was also confirmed by SEM pictures. In ultrasonic measurements it was found out that the signal strength of the flexible UTs made by sol-gel spray and tape casting were 20dB stronger than that fabricated by the screen printing. At room temperature and 150°C, the UT made by tape casting showed comparable signal strength to that made by sol-gel spray technique.

ACKNOWLEDGMENT

The authors thank to Y. Ono, J.F. Moison and Q. Liu for their technical assistance.

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