Development of miniature ultrasonic probe with PZT film deposited by hydrothermal method

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Abstract — Recently, the PZT poly-crystalline film deposited on titanium substrate by hydrothermal method has been studied actively for development of a small actuator and ultrasonic sensor in our laboratory. The PZT poly-crystalline films were deposited on a titanium substrate in KOH solution used as mineralizer which included Pb ion, Ti ion, and Zr ion under high temperature (about 150°C) and high pressure (about 4 kPa). We reported on the needle type miniature hydrophone and the ultrasonic transducer with hydrothermally synthesized PZT poly-crystalline films in 2005 IEEE International Ultrasonics Symposium in Rotterdam. It has confirmed that the hydrothermally synthesized PZT poly-crystalline films showed wide band frequency characteristics in MHz band of the sensitivities. It is thought that miniature ultrasonic probe with wide frequency band characteristics of sensitivities can be fabricated by using the hydrothermally synthesized PZT poly-crystalline films. The miniature ultrasonic probes were fabricated by hydrothermally deposited PZT poly-crystalline film on an end of the titanium wire with diameter of 0.6 mm and length of 50 mm. The performances of trialy fabricated ultrasound probes were measured. Ultrasonic imaging experiments using a target in water were performed with the trialy fabricated ultrasonic probe with hydrothermally synthesized PZT films. As a result, it was confirmed that this probe has a center frequency of 16 MHz, a bandwidth of 80%, and distance resolution more than 80 micrometers. We will report about the structure and performance of the fabricated ultrasound probe with hydrothermally synthesized PZT poly-crystalline film and the results of imaging experiments under water.

Keywords—hydrothermal method; PZT thick film; miniature ultrasonic probe;

I. INTRODUCTION

Recently, miniature and high frequency ultrasonic probes have been developed actively in medical field. The piezoelectric ceramics or the piezoelectric polymer films are used as piezoelectric materials for a miniature high frequency ultrasonic probe. A piezoelectric polymer film shows wide band frequency characteristics but it has the problem of low transmitting sensitivity. The piezoelectric ceramics show high transmitting sensitivity but they have the problems of narrow band frequency characteristics, the brittle material etc. Thus, we paid attention to the deposition technology of the PZT polycrystalline films by the hydrothermal method. The PZT polycrystalline film deposited by hydrothermal method shows piezoelectricity without polluting process and it is easy to form the PZT poly-crystalline films on the substrates with tiny or complex shape. We reported needle-type miniature hydrophones and the ultrasonic transducers with hydrothermally synthesized PZT poly-crystalline films. We could confirm that the hydrothermally synthesized PZT poly-crystalline films showed wide band frequency characteristics of sensitivities in the megahertz band. It is thought that miniature ultrasonic probes with wide frequency band characteristics using hydrothermally synthesized PZT poly-crystalline films. As basics of fabrication of miniature ultrasonic probes, the miniature ultrasonic probe with single element transducer was fabricated and the performances of fabricated ultrasonic probes like transmitted and received characteristics, resolution, etc. were evaluated.

II. METHODS

A. Hydrothermal Method for PZT Deposition

Hydrothermal method is the method of material synthesis or the method for crystal growth in water under high temperature and high pressure. Water exists as liquid under high pressure even if the temperature is above the boiling point of water at atmospheric condition. The reaction impossible to be occurred in atmospheric pressure can be occurred under the above hydrothermal situation. Although it is known that hydrothermally deposited PZT piezoelectric films have various merits, this method is not used widely because of instability and low yield rate. Therefore, we modified the apparatus for hydrothermal synthesis of piezoelectric materials and synthesizing procedure to allow stable deposition of PZT films [1]. Figure 1 shows an apparatus for hydrothermal method. The apparatus was used to develop the miniature ultrasonic probe by hydrothermal method. Aqueous solutions with precursor materials including metal ions of Ti⁴⁺, Zr⁴⁺, Pb²⁺ are mixed with a mineralizer of KOH solution in a teflon coated tank of the apparatus. They are stirred with rotating Teflon blade. PZT poly-crystalline films were deposited hydrothermally on an end of titanium wire with diameter of 0.6 mm for the fabrication of miniature ultrasonic probe. The titanium wires are held directly on the rotating teflon stirrer blade. The PZT poly-crystal films can be deposited on the titanium wires by stirring aqueous solution.
with precursor materials and mineralizer using the rotating Teflon blade under high temperature (120-160 degree centigrade) and high pressure (about 400 kPa). Stirring aqueous solutions and mineralizer using the rotating blade with the titanium substrate is typical feature of our hydrothermal apparatus. We can deposit more stable PZT piezoelectric films within much shorter time with this apparatus than the conventional apparatus. PZT polycrystalline films can be deposited hydrothermally on an end of Ti wire with diameter of 0.6 mm for fabrication of miniature ultrasonic probe. The crystal nucleation process was accomplished at 140°C and the crystal growth process was accomplished at 160°C[4]. One cycle of the crystal nucleation process and five cycle of the crystal growth process were accomplished respectively. Other conditions except temperature were same in both processes of the hydrothermal method. The deposition conditions of PZT polycrystalline films on the Ti wires for fabrication of the miniature ultrasonic probe are shown in Table 1. Figure 2 shows SEM image of the PZT piezoelectric poly-crystalline film deposited on the end face of the titanium wire by hydrothermal synthesis.

Table 1 Hydrothermally synthesizing conditions of PZT polycrystalline films on the Ti wires for fabrication of the miniature ultrasonic probe

<table>
<thead>
<tr>
<th>Material</th>
<th>Concentration</th>
<th>Quantity</th>
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<tbody>
<tr>
<td>ZrOCl₂·8H₂O</td>
<td>0.25M</td>
<td>60ml</td>
</tr>
<tr>
<td>TiO₂</td>
<td>-</td>
<td>1.0g</td>
</tr>
<tr>
<td>Pb(NO₃)₂</td>
<td>0.5M</td>
<td>100ml</td>
</tr>
<tr>
<td>KOH</td>
<td>4M</td>
<td>200ml</td>
</tr>
</tbody>
</table>

B. Assembly of miniature ultrasonic probe

Ti wire with hydrothermally deposited PZT film is connected to the inner conductive line (signal line) of a coaxial cable, and an insulating layer of acrylic resin is coated to the side surface of Ti wire and the inner conductive line. The conductive resin was coated all over the surface of miniature ultrasonic probe for electrostatic shield and ground, and it was connected with the outer conductive line (ground line) of a coaxial cable. Figure 3 and 4 show the schematic diagram and photograph of the fabricated miniature ultrasonic probe, respectively.
C. Performance estimation of fabricated miniature ultrasonic probe

The styrene foam reflector was used for confirmation of transmitted and received of ultrasonic pulse. The styrene foam reflector was located at the distance of 4 mm from the acoustic radiation surface of the miniature ultrasonic probe in water. The pulsar receiver [Model 5800 Panametrics] was used to drive the fabricated miniature ultrasonic probe and to amplify the output signal from the fabricated miniature ultrasonic probe. The burst wave with peak to peak voltage of 250 Vp-p, frequency 20 MHz, and 1.5 cycles was applied to the fabricated ultrasonic probe with the pulsar receiver. The output signal from the ultrasonic probe was amplified using a pre-amplifier with gain of 60 dB in the pulsar receiver. The distance resolution was calculated from the transmission and reception wave form of the ultrasonic wave and the fractional bandwidth was calculated from the frequency spectrum of the receiving wave form. Furthermore, the ultrasonic beam profile transmitted from a miniature ultrasonic probe was measured. Aluminum wire with diameter of 25 µm was used as a string target. The diameter of aluminum wire was thinner than the wave length (75 µm) of ultrasonic wave at 20 MHz from the fabricated miniature ultrasonic probe. The beam profile was measured at the distances of 4 mm and 10 mm between the string target and the fabricated ultrasonic probe by scanning the probe horizontally. Figure 5 shows the beam profile measuring system.

III. RESULTS

The ultrasonic wave transmission-and-reception experiments were performed in water using the fabricated miniature ultrasonic probe. Figure 6 shows the result. Received waveforms were observed at 4.6 µs after transmission. Figure 7 shows the received waveform. We could observe the received ultrasonic waveform with 2.5 cycles in burst. It was found from Fig. 7 that the distance resolution of the fabricated ultrasonic probe was about 110 µm. Figure 8 shows the frequency spectrum calculated from the waveform in Fig. 7 by FFT. The fractional bandwidth was calculated from Fig. 8. As results, center frequency was 16 MHz, fractional bandwidth was 80 %. It has confirmed from the results that the fabricated ultrasonic probe had wideband frequency characteristics.
Next, the beam profile of the miniature ultrasonic probe was measured. Figure 9 shows the measurement result. Beam width data were calculated from the beam profile measured at distances of 4 mm and 10 mm between the acoustic surface of the fabricated ultrasonic probe and the string target. Beam width was estimated as 0.55 mm at distance of 4 mm from the ultrasonic probe, and 1.45 mm at distance of 10 mm. The calculated value of the beam width was calculated as the diameter of 0.6 mm, the driving frequency of 20 MHz, 4 mm and 10 mm of distances from a probe. As a result, the beam width was about 0.71 mm at distance of 4 mm, and was about 1.78 mm at distance of 10 mm. The beam width of the measured value and the calculated value is shown in Table 2. It has confirmed that the measured and calculated value of a beam width showed a near value.

Table 2 Comparison of measurement value and calculated value of beam width

<table>
<thead>
<tr>
<th>Distance of ultrasonic probe and string target</th>
<th>measurement value</th>
<th>calculated value</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 mm</td>
<td>550 µm</td>
<td>710 µm</td>
</tr>
<tr>
<td>10 mm</td>
<td>1450 µm</td>
<td>1780 µm</td>
</tr>
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</table>

IV. CONCLUSIONS

The miniature ultrasonic probe was fabricated using the PZT poly-crystalline film deposited on the end face of a titanium wire by a hydrothermal synthesis. The performances of fabricated miniature ultrasonic probes were measured. Transmission and reception ultrasonic wave by miniature ultrasonic probe were observed in the water. The band width of the miniature ultrasonic probe was 89.7 %, and distance resolution was 110 µm. The beam width was 0.55 mm of distance of 4 mm between the string target and a miniature ultrasonic probe.

REFERENCES