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Fabrication and Characterizations of $PbZr_xTi_{1-x}O_3$ (PZT) Ultrasonic Sensing Chips

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ABSTRACT $PbZr_xTi_{1-x}O_3$ (PZT) ultrasonic sensing chips were fabricated by ball milling, electrical discharge and spraying on iron sheets. Various PZT growth conditions, including the thickness of the iron sheet and the coating layers, were investigated. Ultrasonic sensing measurements indicated that PZT chips of sufficient thickness and with 6 or 9 coating layers exhibited ultrasonic sensing capability. Further, multiple material and electrical characterizations revealed that while sufficient thickness and low leakage were required, uniform growth of the film and a grain size of approximately 150 nm were preferable for ultrasonic sensing. Their compact size, rapid response, and low cost make PZT-based ultrasonic sensors promising for future ultrasonic detection application.

INDEX TERMS $PbZr_xTi_{1-x}O_3$, ultrasonic sensor, morphology, grain size, iron sheet.

I. INTRODUCTION

Ultrasonic transducers are widely used in medical imaging [1], fractal architecture analysis [2], and oil transport leakage detection owing to their precise detection capabilities [3]. Furthermore, ultrasonic sensing can adapt to different environments such as the human body, the atmosphere [4], and reinforced concrete [5] with its penetrating ability, directivity, and easy detection [6]. As manufacturing technologies have advanced, the micro-machined ultrasonic transducer (MUT) has replaced the traditional piezoelectric ceramic ultrasonic transducer. The MUT is a new type of supersonic transducer using a microelectromechanical system (MEMS) of microelectronics and micromechanical structures [7] capable of operating in extreme conditions. Among modern MUTs, piezoelectric micromachined ultrasonic transducers (PMUTs) have attracted growing attention due to their compact size, light weight, low power consumption and high sensitivity [8]. The thin film structure layer of the PMUT, with its manufacturing processes of material preparation, thin film deposition, and heat treatment, is the key component for ultrasonic wave monitoring [7], [9]. Therefore, it is worthwhile to explore different materials with distinct properties in order to optimize ultrasonic sensing capability [8].

Based on previous studies [10], [11], thin films can be used for humidity, gas, and optical sensors. However, fabricating a thin film with acoustic impedance, good piezoelectric characteristics, and adaptability to the substrate is a challenging achievement. In this research, thin film-based sensors were used to detect ultrasonic acoustic waves. High-voltage electric coefficients and high relative permittivity are also required conditions for good piezoelectric materials [12]. Among these, lead zirconate titanate (PZT) is an especially important sensing film material, with a wide range of applications in MEMS system devices [13]–[15]. PZT film

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is usually deposited by printing[16] or sputtering [8], and after careful grinding the thickness of the film can reach 5-50 μm [17]. Differences in thickness may affect the orientation of the internal crystals and the surface structure, which is crucial to ultrasonic sensor performance [18].

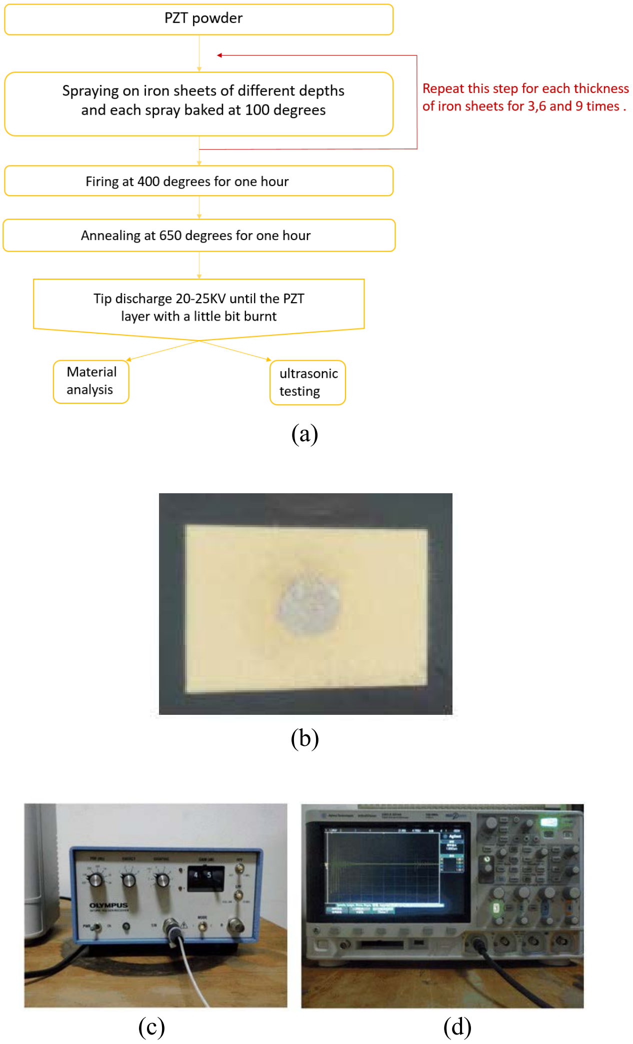
This study differs from conventional manufacturing methods, as PZT films of various thicknesses used in this study were fabricated utlizing a process of ball grinding, electrical discharge, and spraying. In our previous studies, the effects of UV light [7] and poling voltage [19] have been investigated on the film quality of PZT films, while in this study, the thickness of a Ti substrate and the number of coated layers were examined. Different from previous studies [20]–[22], multiple material, electrical characterizations, and ultrasonic detection were performed [23], [24]. Results indicate that PZT films with certain surface microstructures and material properties could achieve ultrasonic sensing [25]. In addition, the characteristics, advantages, and disadvantages of different samples were analyzed. The current-voltage (I-V) curves with leakage current and breakdown voltage related to the ultrasonic performance, were also measured [26], [27]. Our findings indicate that owing to their compact size, easy fabrication, and good ultrasonic sensing performance [13], PZT films with appropriate substrate thickness and layer deposition show promise for future industrial ultrasonic transducers.

The application of this material can readily be applied to ultrasonic sensing devices, and the ultrasonic signal has a close relationship with physical and rheological material properties. Therefore, this ultrasonic technique is a promising candidate for the nondestructive and non-intrusive method of real-time process diagnosis [28]. Ultrasonic technique has been applied for diagnosis of injection molding, co-injection molding, microfluidic devices fabrication [21], [29], [30].

II. EXPERIMENTAL DETAILS

The detailed procedures of the experiment are shown in Fig. 1(a). PZT powder (QA, Eleceram Technoogy Co., Ltd., Taiwan) with an average size of 0.6 μm was used by a ball milling method to form a PZT solvent. Samples of various conditions were prepared by spraying on iron sheets with different thicknesses for 3, 6, and 9 times. A commercial air gun was used to spray the PZT solvent directly onto the samples as follows: First, each kind of iron sheet (S: 0.104 mm, M: 0.490 mm, L: 0.996 mm) was sprayed with formed solvent for 3, 6, and 9 times. Next, the sprayed samples were dried by a hot plate with a temperature of 100 °C, fired at 400 °C for one hour, and then annealed at 650 °C for one hour. Finally, the sample was electrically discharged with a poling machine (RA5-30, Matsusada Precision Inc., Japan) at 20-25 kV until the PZT coating showed a burnt color. This process created piezoelectric material containing electron pairs to enhance directivity.

Figure 1(b) shows the fabricated sample with a silver paste as an electrode in the center. The pulser/receiver shown in Fig. 1C (5072PR, Olympus, Japan) is a broadband, negative spike pulser and broadband receiver which can be applied in a reflection or transmission mode. For this research, the reflection mode was utilized for measuring the ultrasonic signal. Figure 1(d) shows the digital oscilloscope (DSO-X2014A, Agilent Technologies, USA) used for ultrasonic signal recording.



(a)

(b)

(c) (d)

FIGURE 1. **(a) PZT ultrasonic sensing chips fabrication processes.; (b)The fabricated sample; (c) The utilized pulser/receiver; (d) The utilized digital oscilloscope for recording the ultrasonic signals during the experiments.**

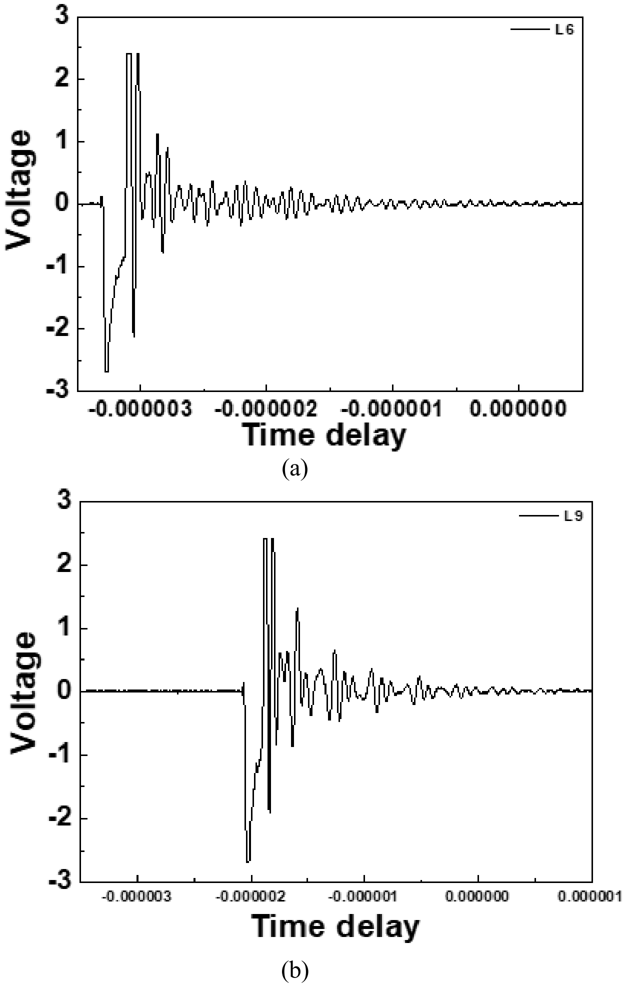
After the PZT films were fabricated, material analysis techniques including Scanning Electron Microscope and Energy-dispersive X-ray Spectroscopy (EDS) (Hitachi S-3000H, Japan), Atomic Force Microscope (AFM) (Bruker Dimension Icon), X-ray Diffraction (XRD, Rigaku PC-2000, USA) were performed. In addition, electrical measurements and ultrasonic testing were also conducted. The fabrication condition of the samples are listed in Table 1.

III. RESULTS AND DISCUSSION

By examining the ultrasonic properties of the samples, clear ultrasonic signals could be found from L6 and L9, as shown in Fig. 2(a) and (b). However, no other samples exhibited

TABLE 1. Fabrication conditions of all the samples.

No.	Iron thickness	Spraying times
S3	0.104 mm	3
S6	0.104 mm	6
S9	0.104 mm	9
M3	0.490 mm	3
M6	0.490 mm	6
M9	0.490 mm	9
L3	0.996 mm	3
L6	0.996 mm	6
L9	0.996 mm	9



(a)

(b)

FIGURE 2. Ultrasonic response of (a) L6 and (b) L9.

ultrasonic signals. To explore the films capable of generating ultrasonic signals compared with those without signals, multiple material analyses and electrical measurements were performed. The microscopy images, as shown in Fig. 3, reveal that uniformly-grown PZT crystals could be found in the L6 and L9 samples while randomly grown crystals could be found in some other samples. This finding indicates that crystalline uniformity might be preferable for PZT ultrasonic sensing.

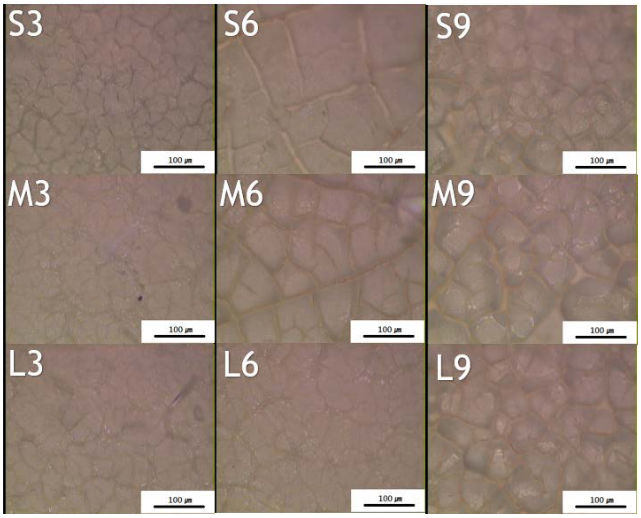


FIGURE 3. Microscopic optical images of all samples.

To zoom in on the nanostructures of all the samples, top-view SEM images are shown in Fig. 4. In the SEM images of S3, S6, and S9, grains of various sizes (200 nm to 20 nm) can be observed. Furthermore, small grains with sizes between 20 nm and 50 nm each can be found in all the S samples. These small grains had many boundaries, which may worsen ultrasonic signal transmission. In the SEM images of M3, M6, and M9, small grains with a size around 20 nm can also be seen. Additionally, grains larger than 150 nm can also be found. Uniform growth was one of the factors for good ultrasonic wave sensing; however, the M samples also showed non-uniform growth. As shown in an SEM image of the L3 sample, large grains of 200 nm and small grains of 20 nm can be seen. Owing to the small grains and random-sized growth, the sample may not be good for ultrasonic sensing. In contrast, the L6 and L9 SEM images did have more uniformly grown grains of around 150 nm in size. Our findings indicate that a grain size of around 150 nm and uniform growth should be key factors for ultrasonic detectors. To further confirm the presence of PZT film, EDX analysis was conducted on all the samples, as shown in Fig. 4, showing the presence of Pb, Zr, Ti, C and O. In addition, Ti concentration in L6 and L9 was much lower than all the other samples, indicative of better PZT film composition. Moreover, cross-sectional SEM images of the three L samples (L3, L6, and L9) shown in Fig. 5 show much more uniform growth in the cross section of L6 and L9 compared to that of L3.

To further characterize the nine samples, AFM and XRD were performed. AFM images and measurements, as shown in Fig. 6, indicate that L6 and L9 had smaller roughness values compared with most of the other samples. Although S6 and S9 presented small roughness values, the iron plate was too thin (0.104mm), and that may influence electrical properties.

Further, XRD patterns reveal the crystalline phases of all the samples, as shown in Fig. 7. All the samples exhibited similar XRD profiles and a strong (110) peak. While L6 had

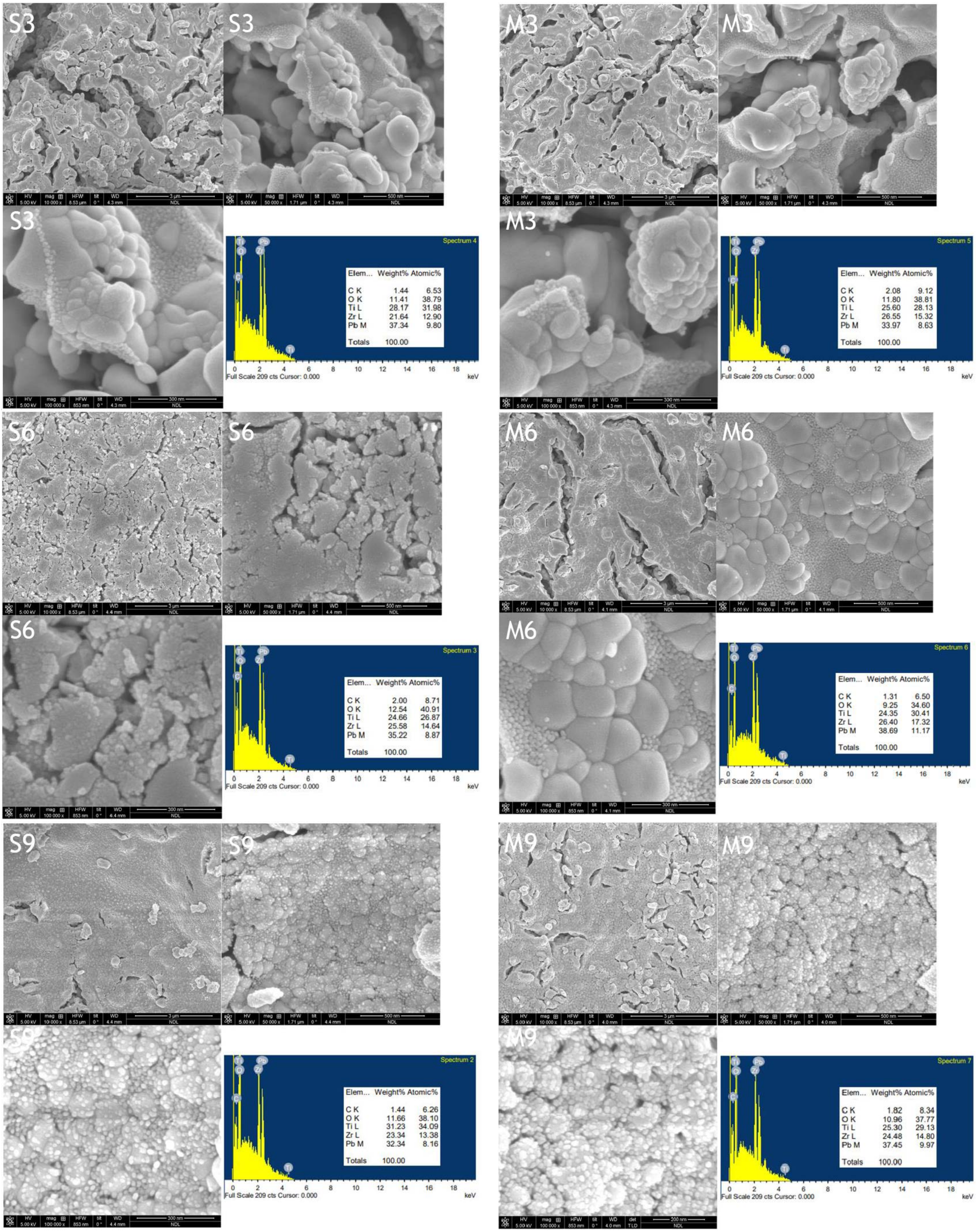
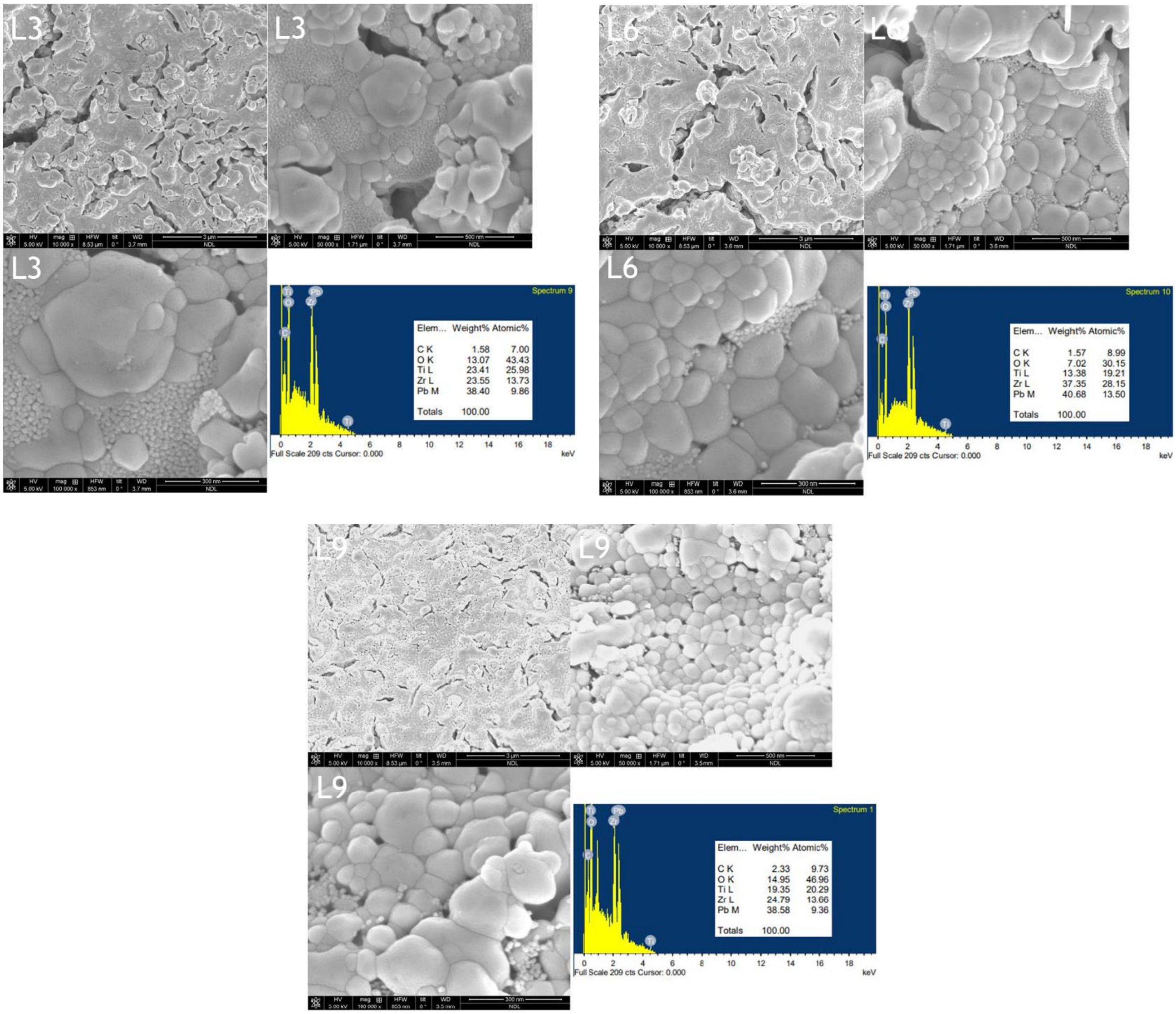


FIGURE 4. Top-view SEM images of all samples.



Spectrum 9

Elem...	Weight%	Atomic%
C K	1.58	7.00
O K	13.07	43.43
Ti L	23.41	25.98
Zr L	23.55	13.73
Pb M	38.40	9.86
Totals	100.00	

Spectrum 10

Elem...	Weight%	Atomic%
C K	1.57	8.99
O K	7.02	30.15
Ti L	13.38	19.21
Zr L	37.35	28.15
Pb M	40.68	13.50
Totals	100.00	

Spectrum 1

Elem...	Weight%	Atomic%
C K	2.33	9.73
O K	14.95	46.96
Ti L	19.35	20.29
Zr L	24.79	13.66
Pb M	38.58	9.36
Totals	100.00	

FIGURE 4. ***(Continued.)*** **Top-view SEM images of all samples.**

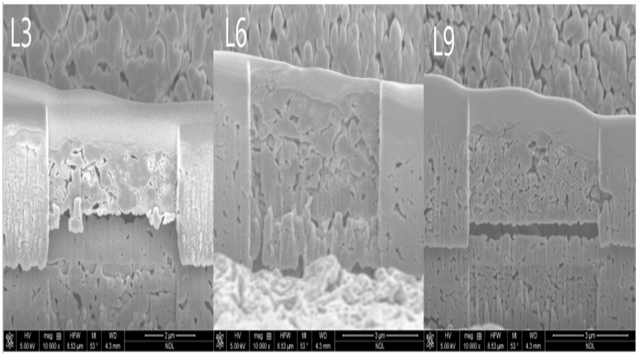


FIGURE 5. **Cross-sectional SEM images of L3, L6 and L9.**

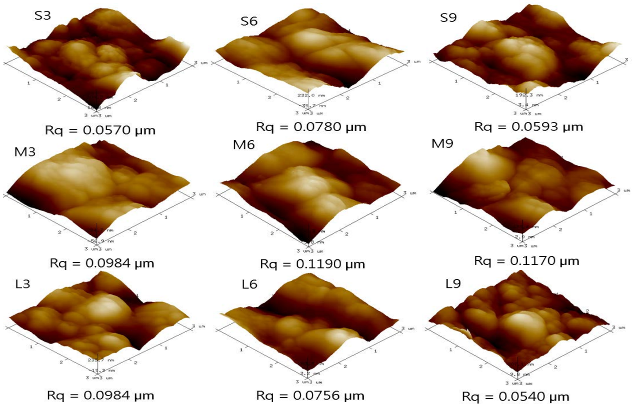


FIGURE 6. **AFM images and measurements of all samples.**

the strongest (110) peak compared with the other samples, L9 exhibited a slightly weaker (110) peak than all the other samples. Based on the PZT (110) peak intensity shown in Table 2, it can be observed that the intensity of the characteristic peak of PZT at (110) increased with the number of coating layers. The maximum peak intensity occurred at a

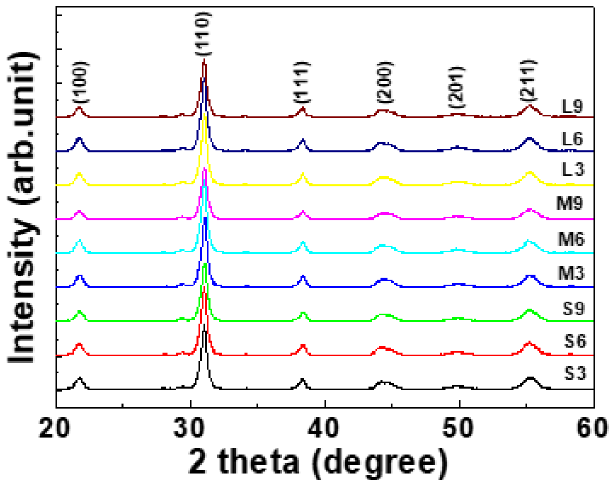


FIGURE 7. XRD patterns of all the samples.

TABLE 2. Maximum PZT (110) XRD intensity of all the samples.

Sample No.	31.06° (110) Intensity
S3	3843
S6	3989
S9	3433
M3	4180
M6	4350
M9	3084
L3	4176
L6	4300
L9	3414

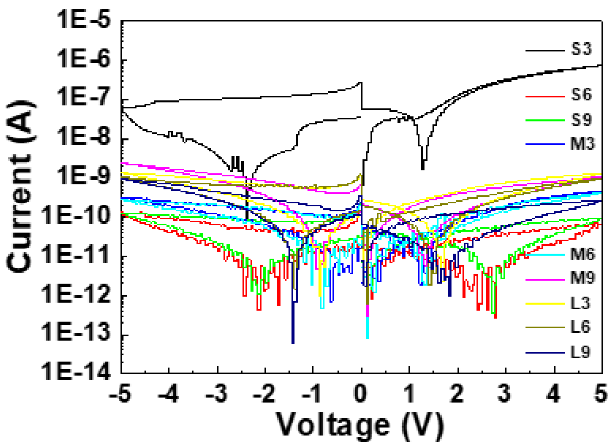


FIGURE 8. I-V curves of all the samples with applied voltage swept from 5V to −5V.

coating of 6 times for all S, M, and L samples, and the peak value greatly dropped for all the samples when the number of layers increased from 6 to 9. Finally, the I-V curves of all the samples with applied voltage swept from -5 V to 5 V were taken, as shown in Fig. 8. Apparently, S3, with the smallest iron plate thickness and 3 coating layers, had much stronger leakage current; around the order of 10^{-7} A. Both L6 and L9 had leakage current smaller than 10^{-9} A. Therefore, thicker iron plate thickness and small electrical leakage current are required to detect ultrasonic signals.

IV. CONCLUSION

In this study, ultrasonic PZT-based sensing devices with various fabrication conditions were successfully demonstrated. Multiple material analyses indicated that uniformly grown PZT grain and a grain size of around 150 nm is required for PZT devices to be capable of detecting ultrasonic signals. Moreover, appropriate thickness, lower Ti concentration, and low leakage current of the PZT chip are also necessary for ultrasound detection. Owing to their rapid response, compact size, and low cost, PZT-based ultrasonic sensing devices are promising for industrial ultrasound-related applications.

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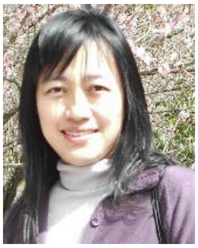

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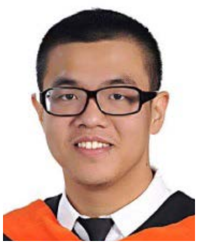

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