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Sputtering Parameters Influence on the as Deposited PZT Thin Films: Surface Roughness and Morphology

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Abstract: A homemade target of Pb ($Zr_{0.48}$, $Ti_{0.52}$) O_3 is sputtered out under different conditions of power and Argon pressure on platinized silicon substrates, however, the distance inter-electrodes and time of growth were fixed. The deposition is running at room temperature. The surface morphology and roughness formed depend strongly on the input power and working pressure. When the argon (Ar) pressure is set to 2 pa, at 100 W of input power we observe bubbles due to low energy nucleation on the substrate, at 150 W we obtained more dens surface, at 200 W the sputtered particles from the target has more energy, therefore, we observe the impact of a collision on the surface. When the working pressure of Ar is set to 3 pa the surface contains large grains, at 150 W we observe the presence of holes, and at 200 W high roughness surface is obtained. The optimum conditions are 2 pa with 150 W of input power. New samples are prepared in the optimum conditions of 2 pa and 150 W during 2 h of sputtering the thickness measured by the tilting image of a scanning electronic microscope (SEM) is 300 nm. Then, conventional annealing at 650 °C, for 1 h. The XRD pattern shows polycrystalline microstructure with preferred orientation on (300) and (110) the measured average roughness by atomic force microscopy (AFM) is 38 to 45 nm. The ferroelectric measurement shows a hysteresis loop.
Keywords: PZT thin films, RF sputtering, surface and morphology, ferroelectricity

1 Introduction

The sputtering deposition is one of the physical thin films deposition technics currently used in the process fabrication of integrated circuit technology [1], therefore most new material development are involved compatible technics to the IC technology [2]. However, the conditions of elaboration must be mastered to guarantee the reproducibility of some important requirements needed in IC technology as surface quality and morphology [3].

Ferroelectric materials as PZT is intensively studied for numerous applications in memories, sensors, actuators or more recently for energy harvesting, using chemical and physical elaboration technics [4,5,6,7]. However, the CMOS-MEMS technology is the most compatible with IC integration. Among this process, the RF magnetron sputtering is low cost and the simplest technique for such a purpose. In this paper, we treat one of two important problems when we want to integrate PZT in IC technology. The first one is the surface quality and morphology [8,9]. The second is the temperature compatibility, which is beyond the scope of this article [10].

In this technical paper, we present a simple experimental plan for thin films growth inspection. the conditions of pulverization were set to pressure of working at 2 pa, then the input power is set to 100 W,150 W, and 200 W. after that we set the second value of pressure at 3 pa, with input power at 100 W and 150 W then 200 W. The sputtering time is 60 min and the inter electrodes are 5 cm for all experiences. the SEM and AFM tools for surface quality, morphology, and roughness measurements analyzed the as-deposited thin films. Then ferroelectric properties are verified showing a hysteresis loop.

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2 Experimental Sections

The PZT target is homemade in the Morphotropic phase boundaries (MPB), Pb ($Zr_{0.48}$, $Ti_{0.52}$) O_3 [11]. Then we used RF magnetron sputtering system, the operating chamber is pumped to the secondary vacuum of 1.6 10^{-5} Torr the substrates are not heated when the sputtering is running, however, we measured heat rising because of the energy of sputtered particles when hits the substrate [14]. The conditions of growth are shown in table 1. The morphology is measured using SEM Philips XL30-FEG microscope and JEOLE JSM 6360LV equipment. However, JEOL SPM 5200 measures roughness. The PZT thin films under the optimum deposition conditions are annealed at 650 °C during 1 h in air then polarize under 320 °C, voltage of 15 V during 30 min this operation makes all the dipolar moments in the same direction as the electric field applied. Then the microstructure was investigated using X-ray Diffraction tool of Bruker D8 Advance diffractometer using a CuKα radiation source (λCuKα=1.5406 Å) with a Copper anode, and operating at 40 kV and 40 mA in grazing incidence setup with a fixed source angle of 0.5°. The peak indexing and phase identification were performed by using the program EVA 5.0 associated with the XRD equipment. Finally, the ferroelectric property of the films is measured Sawer-Tower circuit.

Table 1: Deposition conditions for RF magnetron sputtering.

	2 Pa	3 Pa
100 W	T= 60 min. P= 1.7 10^{-5} torr P_{Ar}=2 Pa	T= 60 min. P= 1.6 10^{-5} torr P_{Ar}=3 Pa
150 W	T= 60 min. P= 1.7 10^{-5} torr P_{Ar}=2 Pa	T= 60 min. P= 1.7 10^{-5} torr P_{Ar}=3 Pa
200 W	T= 60 min. P= 1.7 10^{-5} torr P_{Ar}=2 Pa	T= 60 min. P= 1.6 10^{-5} torr P_{Ar}=3 Pa

3 Results and Discussion

The morphology of the samples under 2 pa for the input power of 100 W, 150 W, and 200 W is shown in figure1. At 100 W Figure 1 A), we observe bubbles on the surface due to the low adhesion to the substrate, this is because of the low energy of the sputtered PZT particle when hit the substrate therefore the nucleation energy is not enough leading to form bubbles. At 150 W Figure 1 B1, 2) the surface is free from bubbles, we observe a condensed layer with cracks. On C) at 200 W the high energy of sputtered PZT particles left an impact footprint on the surface of the thin films.Paper

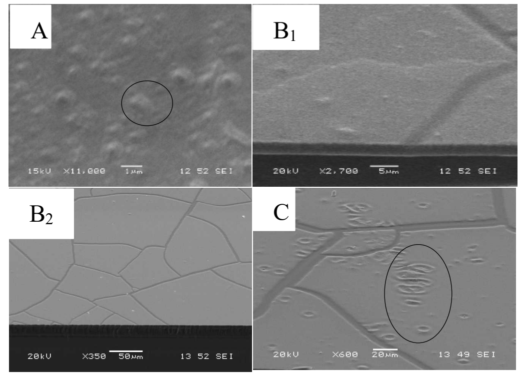

Fig. 1: SEM images of PZT as deposited thin films at Ar presser of 2 Pa, A) 100 W, B) 150 W and B) 200 W.

When we set the working presser of Ar at 3 pa we observe an important change in the texture which becomes rougher. At 150 W on B) even holes density becomes important, on C) at 200 W the surface, we observe high roughness and the texture become granular.

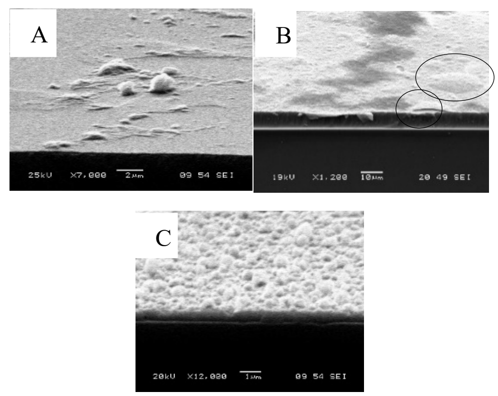

Fig. 2: SEM images of PZT as deposited thin films at Ar pressure of 3pa A) at 100 W, B) 150 W and C) 200W.

In this section, we observe a big change in the surface and texture of the as-deposited thin films of PZT when the conditions of sputtering are varied. We could say that the optimum conditions would be 2 Pa in terms of gas pressure of Ar and 150 W of input power. The second observation is that the tilted images do not show an important change in deposition rate, when the input power is increasing. In our previous work we observe low deposition rate when we used a simple RF sputtering system without magnetron [12].Therefore, we prepared a new sample under the optimum conditions see table 2 the deposition is running for 2 hours in order to increase the deposition rate, therefore the tilted image show 300 nm of thickness Figure 4 A). Then the as-deposited thin film of PZT is annealed at 650 °C for 1 hour to induce the crystallization. The surface investigation at low and high magnification Figure 4 B, C) shows free holes surface with a smooth surface. The XRD patterns recorded are shown in Figure 3, the result show formation of polycrystalline ceramic with relative preferred orientation to

(300) and (110) due to the homo-nucleation during the conventional annealing. The hetero-nucleation is advantaged when rapid thermal annealing (RTA) is applied, therefore the preferred crystal orientation becomes following the substrate orientation [13]

Table 2: The optimum sputtering condition for PZT thin films.

Parameters	Values
Target	$Pb(Zr_{0.48},Ti_{0.52})O_3$
Target diameter	7.6 [cm]
Target-substrate distance	5 [cm]
RF power	150 [W]
Sputtering gas	Argon (100%)
Sputtering pressure	2 [Pa]
Sputtering time	120 [min]
Postdeposition annealing	650 [°C] in air

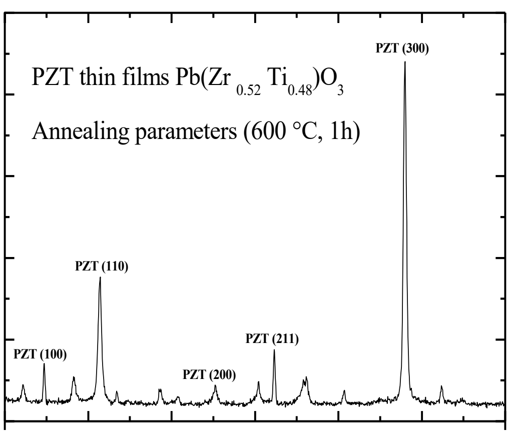

Fig. 3: XRD pattern for PZT thin films deposited under the optimum conditions and annealed at 650 °C.

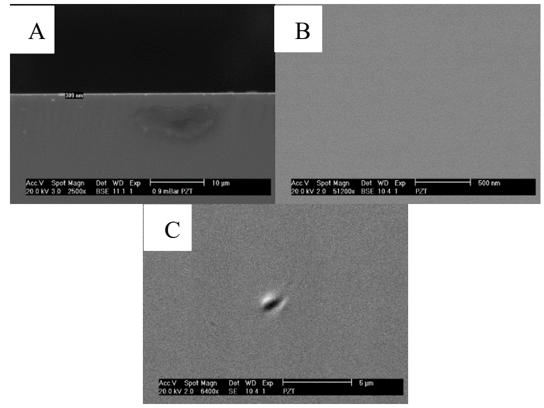

Fig. 4: SEM images for annealed PZT thin films A) tilted image showing 300 nm, B) high magnification image, C) low magnification image.

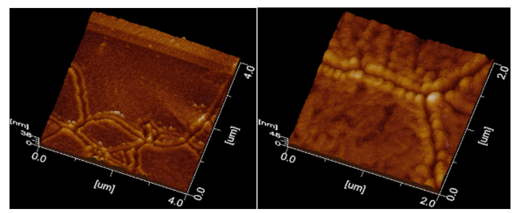

Fig. 5: AFM 3D images measuring the roughness of 38 to 45 nm for the annealed PZT thin films.

The roughness measured (Figure 5) is about 15% of the total thickness the morphology obtained has a smooth surface and regular texture that pretty much matches with microelectronic and microsystem requirements.

The ferroelectric property of the PZT thin film is characterized by showing a hysteresis loop with obvious saturation of the top of the loop Figure 6.

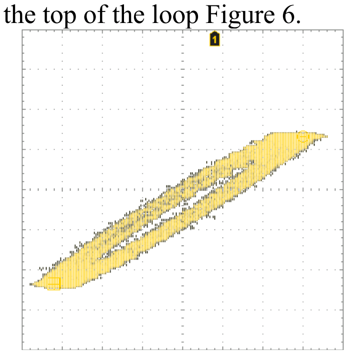

Fig. 6: The hysteresis loop of the annealed PZT thin film.

4 Conclusions

We demonstrate simple experimental plan for RF sputtering thin films deposition investigation and deposition rate. We show the optimum conditions of growth for the PZT in term of morphology and surface roughness, the same strategy is very useful for other material. The pressure of Argon gas in rage of 2 pa and 150 W of input power gives the overall requirements for micro-electric technology.

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Conflict of Interest

All authors declare that there is no conflict of interest regarding the publication of this paper.

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