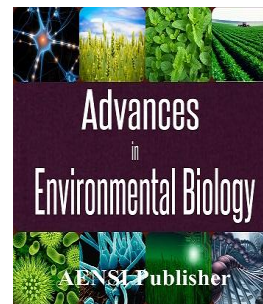




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## A Study on PZT Solutions and Determining the Optimal Solution for Fewer Cracks on the Chip Surface

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### ABSTRACT

Thermal energy in the environment is regarded as a potential source of energy for low-power electronic devices. Thermoelectric Modules work on the thermal gradient provided by body heat and batteries can be charged by using thermal gradients. Pyroelectric materials produce power from fluctuations of time temperature instead of harvesting energy from gradient of space temperature. With regard to the importance of ferroelectric thin films in Piezoelectric, electro-optic, and pyroelectric applications and compatibility of these films with the integrated circuit technology for achieving the highest function and miniaturizing new devices, this issue has been one of the important subjects of the world research. The recent work has been provided for use of PZT pyroelectric materials for harvesting and saving energy. To obtain thin film, sol-gel method was used that is an optimal and cost effective method with good control on the level and thickness for producing thin films. The new work done in this paper was to study a variety of solutions with different molar ratios. As observed, PZT with lead nitrate solution is a proper solution because curing temperature and time are reduced by reduction of carbon volume in the solution. So, cracks are reduced. The solvent 2-Methoxyethanol has higher chelating due to having smaller colloidal molecules and provides lower viscosity of soluble organic compounds tangibly. It helps removal of organic materials and so reduction of the required temperature for PZT crystallization. With regard to the test results, fewer cracks were observed by increase in the solution concentration.

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### INTRODUCTION

With regard to the increasing use of piezoelectric and pyroelectric ceramics, these materials are widely used in manufacturing electronic, optical, and electro-optical parts including infrared sensors, memory modules, pyroelectric infrared detectors of piezoelectric and pyroelectric transducer, and ultrasound transducers [8]. PZT ceramics have a perovskite structure which has the ability to save electrical load, weak coercive field, and thermal stability near the morphotropic border. The common method to prepare PZT powder is mixed oxide method in which a combination of TiO<sub>2</sub>, ZrO<sub>2</sub>, and PbO are used. This method is faced with such problems as high sintering temperature, high phase changes when mixing oxides, and weakness of micro-structure. To remove these problems, heating temperature is reduced as far as possible which leads to high homogeneity and better control on ceramic compounds. Chemical methods including gel-combustion and co-precipitation lead to formation of small particles with high homogeneity and facilitation in making ceramics [1,5]. One of the most important applications of PZT thin films is to use them as a pyroelectric element in infrared sensors. Many different types of pyroelectric sensors with different structures have been designed and are aimed at improving function of the developed part. By using advanced MEMS technology, IR sensors are studied for reducing thermal mass and are used to integrated IR sensor thin film with their control circuits on the silicon chip as an integrated part. In such a IR micro-machined sensor, sensing element with a thickness of several micrometers is placed and so heat waste from PZT pyroelectric layer to substrate is reduced.

At present most of the studied ferroelectrics are a series of oxide materials and are not chemically compatible with traditional semi-conductors such as Si and GaAs. That's why frequently an oxide layer with

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undesirable properties (low dielectric constant, non-spontaneous polarization) between them before sediment of ferroelectric oxide on a semi-conductor is observed which is resulted from semi-conductor oxidation. This layer separates semi-conductor and ferroelectric from each other. Even if this layer is not created, when hetero-structure is built, high temperature processes that are required for PZT formation may lead to gradual destruction of the shared surface and it is required to use buffer layers [6].

In general, there are two types of solution to prepare PZT solution by sol-gel method.

- 1- Sol-gel method based on acetic acid
- 2- Sol-gel method based on 2-Methoxyethanol

Between these two methods, sol-gel method based on 2-Methoxyethanol was selected. So, it is first introduced. Sol-gel method is based on 2-Methoxyethanol solvent. 2-Me reacts with alkoxide metals that acts as a chelating factor effectively and prevents from complete hydrolysis of alkoxide metals in the added water. Hydrolysis reactions are controlled by the amount of 2-Me/ added water [9].

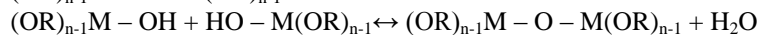
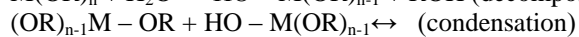
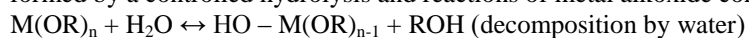
Comparison of this method with acetic acid method shows some differences in the path of crystallization. PZT films based on crystallization of sol-gel are better than those with acetic acid sol-gel. This is attributed to 2-Me colloidal sol smaller molecules. Lower viscosity of organic compounds of solution has tangibly lower "relationship" with the structural part of sol-gel. Removal of organic materials and so reduction of the required temperature helps PZT crystallization. However, recently 2-Me has completely been removed from sol-gel commercial method in the semi-conductor industry of USA due to its carcinogenic nature. Steps of reaching films by acetic acid sol-gel method with electrical properties comparable with 2-Me sol-gel films are important.

Thermal process method is very important. Films have the highest relative permeability by processing on hot-plate and consecutive annealing cycles in the furnace. Di-electric constant values for PZT films by this method have been reported over 900 in the papers. Kumar and colleagues introduced rapid thermal process (RTP) in which increase in the temperature stems from light absorption of quartz lamp with silicon substrate. Processing cycles are reduced considerably over time and they are highly renewable that has made this method very attractive for the industry. Basically rapid thermal process for PZT is used to made crystallization in sputtering films. This method is also used in processing 2-Me based on sol-gel films and has been shows to improve crystallization of films by using shorter maintenance times than furnace annealing. Di-electric constants were obtained in 600-700 range. No result has been published for sol-gel method based on PZT acetic acid method by this technique. With regard to the advantages of using the system with more than one common furnace, RTP method was selected but the recent reports have shown that RTP creates crack on PZT thin film [4].

This project is aimed at obtaining a PZT ferroelectric thin film with crack and with the desirable thickness and obtaining PZT pyroelectric properties by sol-gel method. In this paper, MEMS technology has not been used to reach this goal. Since selecting a proper substrate is very important for successful growth of films with crystallized quality and lowest stress and defect, a proper substrate according to the latest technology, i.e. silicon, is used for PZT growth. In the conducted studies, no difference is observed in the type of used silicon (n or p), but type p had been used in most papers.

### 1.1 sol-gel method:

Sol-gel is an organic metal colloidal solution  $[M(OR)_n]$  or organic metal- oxygen – organic metal molecules  $[(OR)_{n-1}M-O-M(OR)_{n-1}]$  that have been connected in the form of polymer network O-M-O. The solvent is formed by a controlled hydrolysis and reactions of metal alkoxide concentration [11].



In the above equations, R shows alkyl radicals (like methyl  $(CH_3)$ , ethyl  $(CH_2CH_3)$ , etc.). Hydrolysis composed of replacement of one or a group of alkoxide with hydroxyl ion (OH) releases a molecule of alcohol in the process. Reaction of concentration between two hydrolyzed molecules  $[(OR)_{n-1}M-OH]$  and one alkoxide  $[M(OR)_n]$  is in the form of molecule M-O-M with one  $(OR)_{n-1}$  connected to each atom of metal. According to this definition, density depending upon the primary compounds is able to release one molecule of alcohol or water [11].

If hydrolysis and reactions of alkoxide metal condensation have not been checked, large molecules containing M-O-M will be formed and sediments are formed depending upon reaction speed and will finally be converted into strong viscosity or solid gel. These reactions may occur anywhere and anytime (below one second to several weeks). Reactions of hydrolysis and concentration are reversible. Speed of reaction depends upon density of the primary compounds and may vary with other reactors that prevents from hardness. If hydrolysis / concentration reaction reaches balance with its reverse reaction, the solution will never become gel in the sealed container. Such a solvent is called sustainable sol-gel. By changing water/ alcohol ratio, controlling speed of each reaction is possible. If alcohol is removed from sustainable sol-gel, it will reduce speed of reverse

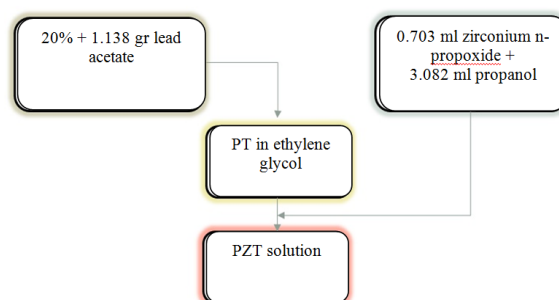
reactions and the balance between hydrolysis and concentration will be changed. If alcohol is removed from the solution sufficiently, finally gel will be formed [10]. In fact, this is favorable for making thin films. When the solvent is spread on the surface (with a deep coverage), evaporation of solvent and formation of gel make a thin film that is enough hard for replacement. Also chelating compounds may change concretion. These compounds include  $\beta$ -diketones Phosphorus like Acetylacetone (ACAC) and carboxide hydroxide acids like lactic acid. Compounds with high boiling point like Ethylene glycol may control the temperature in which decomposition occurs.

## 2. Research method:

In this project, Electron beam evaporation method (e-beam) was used for deposition. Titanium with a thickness of 40 Nm was deposited and removed from the case so that a little oxide was formed on it and then platinum with a thickness of 150 Nm was deposited. To form  $TiO_x$  with the existing facilities, it must be annealed for 30 minutes after depositing Ti in the presence of oxygen at a temperature of 650 °C.

### 2.1 Preparing PZT solution No. 1:

The first step in PZT deposition is to prepare its solution; but a brief knowledge about general chemistry of sol-gel is necessary to prepare it and improvement in the properties of sol-gel films occurs through changing its chemistry. To prepare PZT solution, usually lead acetate tri-hydrate is dehydrated and this is done by different methods. One of the methods is to place lead acetate tri-hydrate powder in the vacuum oven at a temperature of 80 °C for 24 hours or more. In so doing, 40 gr lead acetate tri-hydrate was weighted and it was expected that it loses 2 gr of its weight according to the calculations. By placing it in the oven for 24 hours, its weight was reduced to 36 gr. The first precursor includes mixture of titanium isopropoxide and ethylene glycol that dehydrated lead acetate is added to it and constant rotation of 60 magnets was used to mix it. The second precursor includes Zirconium isopropoxide with 2-propanol placed in the constant rotation. Both precursors were mixed separately for 2 hours. Then to achieve PZT solution, the second precursor was added to the first one and placed in the constant rotation 60 for 24 hours. Figure 1 shows the steps of preparing PZT solution briefly.



**Fig. 1:** steps of preparing PZT solution No. 1.

To make PZT thin film by sol-gel method, usually spin coat is used. The main advantage of using spin coat is to control thickness of layers. Sol liquid was spun directly on the substrates with the following speeds and times by a spin coat system existing in the laboratory. Calculations of the ratio of used materials are presented in the following [3].

The amount of lead acetate tri-hydrate:

$$10ml \times \frac{0/3 (mmol)_{PZT}}{1 (ml)} \times \frac{1 (mmol)_{Pb}}{1 (mmol)_{PZT}} \times \frac{1 (mmol)_{lead acetate}}{1 (mmol)_{Pb}} \times \frac{379/34 (mgr)}{1 (mmol)_{lead acetate}} = 1/138 (gr)$$

The amount of n- zirconium propoxide:

$$10ml \times \frac{0/3 (mmol)_{PZT}}{1 (ml)} \times \frac{0/53 (mmol)_{Zr}}{1 (mmol)_{PZT}} \times \frac{1 (mmol)_{n-zirconium propoxide}}{1 (mmol)_{Zr}} \times \frac{327/57 (mgr)}{1 (mmol)_{n-zirconium propoxide}} \times \frac{100}{70} \times \frac{1 (ml)}{1/058} = 0/703 (ml)$$

The amount of titanium isopropoxide:

$$10\text{ml} \times \frac{0}{3} \frac{(\text{mmol})_{\text{PZT}}}{1(\text{ml})} \times \frac{0}{47(\text{mmol})_{\text{Ti}}} \times \frac{1(\text{mmol})_{\text{titanium isopropoxide}}}{1(\text{mmol})_{\text{Ti}}} \times \frac{284}{26(\text{mgr})} \times \frac{100}{99} \times \frac{1(\text{ml})}{0/9711} = 0/417(\text{ml})$$

## 2.2 Heating:

After spinning PZT sol on the substrate, it is required to heat it to make gel better. This step is broken into three sub-steps [7].

Drying film: it is usually done on the hot plate with a temperature between 100°C and 200°C depending upon film combination. Within this step, the redundant solvent is removed and film is condensed and compressed. If PZT film thickness does not reach the desirable amount, the trend of spin coat and drying must be repeated to reach the desirable thickness. Then we go through the second part of heating.

1- Heating to remove organic compounds: this step is known as decomposition. Temperature for PZT film annealing can be between 400°C and 500°C for 60 seconds with the speed 4°C/s. along this step, the liquid remained from drying step is removed. Some of the organic materials are evaporated and the remaining organic compounds are decomposed in the form of such gases as H<sub>2</sub> and CO<sub>2</sub>. After this step, film is formed from amorph non-organic oxides.

2- Higher temperature for PZT crystallization: this step is mainly related to film "annealing" and can be done in the furnace or rapid thermal process. After decomposition and removal of organic elements, mineral elements are placed close to each other. This has the advantage of removing atoms distance which requires spreading in the film volume.

In the following, we placed some samples with coating liquid at a temperature of 120°C for 20 minutes on the hot plate to remove and dry its solvent. By using electron microscope, the surface of samples was observed. All samples were obtained full of cracks. So, we sought other various annealing types and tests of speed and time. One can say that one of the major reasons of these bad results is to place samples at a temperature of 120°C. However, some papers have mentioned that cracks on the first layer are natural and they are removed by increasing layers; but by repeating this trend, samples did not become better and cracks remained.

We used AFM system to obtain thickness of PZT layer. We placed photo-resist AZ1813 on a half side of sample surface and placed PZT in its corrosive solution to remove other half side of PZT. So, PZT corrosive solution was prepared with H<sub>2</sub>O/HCl/HF with the ratio 50:50:1 and the single layer sample was submerged in it and was removed within 20 seconds. Its thickness was obtained 33 Nm. That is, etching speed of PZT by the above mentioned solution is near 1/5 nm/s.

## 2.3 PZT etching:

### 1- PZT dry etching:

Different dry etching processes including ion reaction and ion beam etching have been reported for determining models of ferroelectric materials. However, it is difficult to obtain satisfactory PZT models due to different speeds of deposition of lead, zirconium, titanium, weak selection of PZT on photo-resist and lower platinum electrode. Moreover, chemical pollutions during RIE process lead very likely to roughness of the surface and decline of etched PZT films properties. So we follow humid etching.

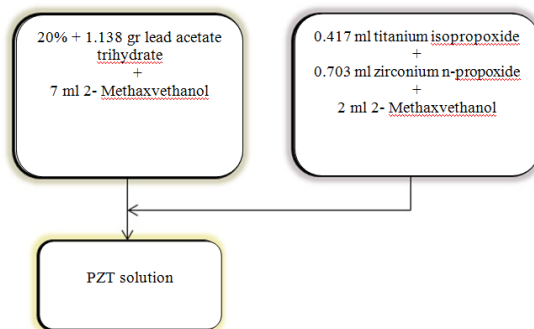
### 2- Humid etching:

Compared to dry etching, humid etching is an effective method in MEMS with regard to its high etching rate, low cost, and high selection. In the recent years, many researchers have studied PZT/Pt film production by using humid etching method. However, considerable undercutting is observed in the model. Selection of electrode structure, type of etches, and its density plays a major role in obtaining a film without defect.

HF is a required material for etching because only HF may etch ZrO<sub>2</sub> effectively. HCl and HNO<sub>3</sub> were used as other components; because their etching speed on PbO and TiO<sub>2</sub> matches with HF on ZrO<sub>2</sub> and TiO<sub>2</sub>. HF is the common etching solution used for oxide humid etching. But it needs controlling high activities of F<sup>-</sup> ions and controlling too much etching. F<sup>-</sup> ions etching Zr<sub>x</sub>O<sub>y</sub> and Ti<sub>x</sub>O<sub>y</sub> easily and very rapidly. Also it may etch Pb<sub>x</sub>O<sub>y</sub> too, but it has lower etching speed. Lead compounds are formed easily on the surface of electrode and these compounds may be easily removed by etching solutions with regard to replacement reaction. HNO<sub>3</sub> and HCl solutions soften strong lead compounds and reduce its power. So, it may lead to removal of strong lead compounds from the platinum electrode surface after etching by the above solutions. Formation of rich lead compounds on the platinum electrode surface can be reduced by decreasing density of etching solution. This is possible through adding H<sub>2</sub>O to the solution.

#### 2.4 Preparing the second solution:

To obtain the required solution, first the precursor lead tri-hydrate and 2-methaxvethanol were mixed for 3 hours to gain a homogeneous solution. For more homogeneity, we placed it in reflux system for half an hour. The second precursor including zirconium propoxide, titanium isopropoxide, and 2-methaxvethanol was prepared separately and placed it on stirrer to become homogeneous. We placed the first precursor in the distiller with the speed 4000 rpm and a temperature of 100°C to remove the water existing in the lead tri-hydrate. Finally white foam remains. Gradually temperature is cut to reach the room temperature and vacuum was removed. Again some 2-methaxvethanol was added to the remaining material and was placed at a temperature of 105°C on stirrer for 1 hour. Then the second precursor was added gradually and was mixed for 7 hours at a temperature of 80°C. Figure 2 shows steps of preparing solution briefly. This time, molar solution 0.4 was prepared.



**Fig. 2:** steps of preparing PZT solution No. 2.

#### 2.5 Preparing third solution:

Since lead nitrate [Pb(NO<sub>3</sub>)<sub>2</sub>] has more alk compounds compared to lead acetate and they must be removed in the steps of organic materials annealing, use of lead nitrate instead of lead acetate as a method for film with very low carbon is suggested. The carbon reduction is one of the reasons for low crystallization temperature of sol-gel based on 2-Me and since temperature has a direct effect on the cracks, this time molar solution 0.5 with lead nitrate was prepared.

Lead nitrate is mixed with 2- methaxvethanol until lead nitrate is fully solved in it. Then zirconium alkoxide is added to the solution and then after 10 minutes titanium alkoxide is added. The reason that why titanium alkoxide is not added first is that large chains of titanium alkoxide are formed due to higher reaction speed of titanium iso-propoxide that cause non-homogeneity or in some cases sediment in the solution. To prevent it, zirconium propoxide is usually added first. After 10 minutes, Acetylacetone is added to the solution. Finally to reach a homogeneous solution, it is mixed for 4 hours.

The amount of the used lead nitrate:

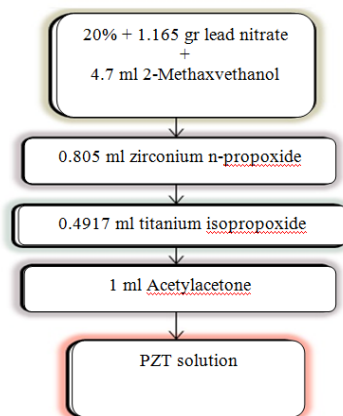
$$7 \times 0/5 \times 331/21 \times \frac{100}{99/5} = 1/165 \text{ (gr)}$$

The amount of zirconium n-propoxide:

$$7 \text{ ml} \times \frac{0/5 \text{ (mmol)}_{\text{PZT}}}{1 \text{ (ml)}} \times \frac{0/52 \text{ (mmol)}_{\text{Zr}}}{1 \text{ (mmol)}_{\text{PZT}}} \times \frac{1 \text{ (mmol)}_{\text{n-zirconium propoxide}}}{1 \text{ (mmol)}_{\text{Zr}}} \times \frac{327/57 \text{ (mgr)}}{1 \text{ (mmol)}_{\text{n-zirconium propoxide}}} \times \frac{100}{70} \times \frac{1 \text{ (ml)}}{1/058} = 0/805 \text{ (ml)}$$

The amount of titanium iso-propoxide:

$$7 \text{ ml} \times \frac{0/5 \text{ (mmol)}_{\text{PZT}}}{1 \text{ (ml)}} \times \frac{0/48 \text{ (mmol)}_{\text{Ti}}}{1 \text{ (mmol)}_{\text{PZT}}} \times \frac{1 \text{ (mmol)}_{\text{titanium isopropoxide}}}{1 \text{ (mmol)}_{\text{Ti}}} \times \frac{284/26 \text{ (mgr)}}{1 \text{ (mmol)}_{\text{titanium isopropoxide}}} \times \frac{100}{99} \times \frac{1 \text{ (ml)}}{0/9711} = 0/4917 \text{ (ml)}$$



**Fig. 3:** shows the steps of preparing solution briefly.

### 3. Research Results:

#### 3.1 First solution:

In the following, samples deposition was conducted with different speeds and times for spin coat and different times of annealing presented in the below table.

**Table 1:** spin times and speeds of PZT deposition.

Sample number	Spin time (s)	Spin speed (rpm)
1	1 hour	500
2	20- 30- 40- 50- 60	1000
3	20- 30- 40- 50- 60	1500
4	20- 30- 40- 50- 60	2000
5	20- 30- 40- 50- 60	2500
6	20- 30- 40- 50- 60	3000
7	20- 30- 40- 50- 60	3500
8	20- 30- 40- 50- 60	4000
9	-	Without spin

It must be noted that in these tests, samples that were annealed got warm gradually (almost 10°C/min). The samples that were dried in the air without annealing showed more favorable results compared to those that were annealed but again the rate of cracks was badly unacceptable. Figure 4 shows the sample dried at a temperature of 110°C and in the air. In the second test, we sought to change the solution and use 2-methaxvethanol to observe better results.



**Fig. 4:** sample dried by hot plate (right side) – sample dried in the air (left side).

#### 3.2 Second solution:

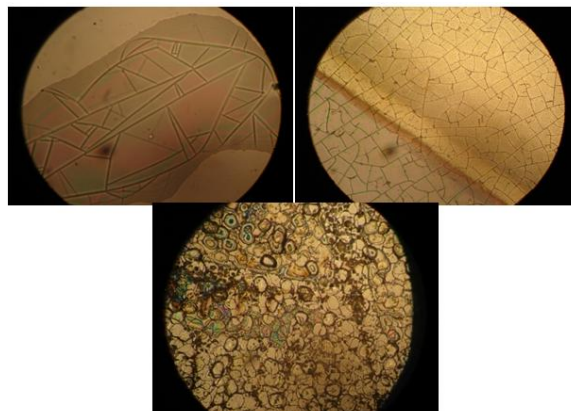
Samples deposition was conducted with different speeds and times for spin coat and different times of annealing presented in the below table.

This time, better results were achieved compared to the last time. It must be noted that the speed 3000 rpm and time 20-30 seconds were obtained as the favorable speed and time.

This solution created some cracks on the surface too and its major problem was the long process of solution preparation and more importantly, sedimentation after several minutes. So we continued to find a better solution.

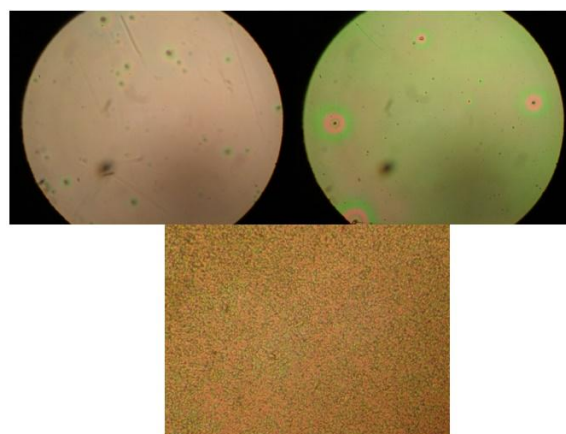
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1	1 hour	500
2	20- 30- 40- 50- 60	1000
3	20- 30- 40- 50- 60	1500
4	20- 30- 40- 50- 60	2000
5	20- 30- 40- 50- 60	2500
6	20- 30- 40- 50- 60	3000
7	20- 30- 40- 50- 60	3500
8	20- 30- 40- 50- 60	4000
9	-	Without spin

**Fig. 5:** samples deposited by PZT.

### 3.3 Third solution:

deposition was conducted with the ideal speed 3000 rpm and time 30 seconds achieved in the last experiment and samples were annealed for 10 minutes in the air and then on the hot plate at a temperature of 120°C for 15 minutes. We dare say that none of the prior studies has achieved such a level of production without crack.

**Fig. 6:** samples deposited with the thickness of 250 Nm (right side) and 800 Nm (left side).

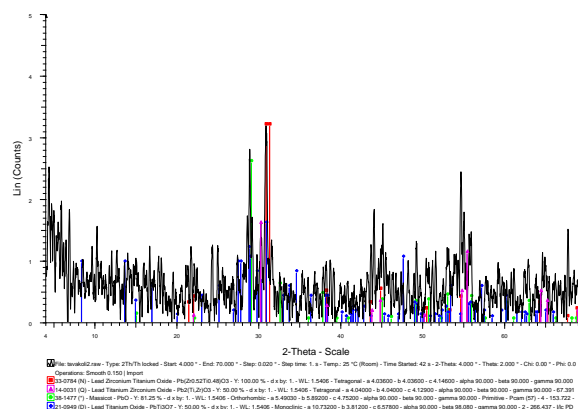
Then they were placed in the furnace for the final annealing with the speed 5°C/min at 550°C. Up to 400°C, no gas is entered into the furnace so that organic materials are removed from the surface due to heat and then oxygen is entered with the pressure 100sccm and remains for 30 minutes at a temperature of 550°C. Then the furnace cooled with the same speed. The following figures show samples after final annealing.

### 3.4 Results of XRD test:

To get the initial XRD, it was required to prepare a PZT film. Since PZT film is not dependent upon substrate, we prepared it on a piece of glass with a thickness of 1 cm without spin coating but with the same annealing conditions. Figure 8 shows the obtained results.



**Fig. 7:** samples after final annealing.

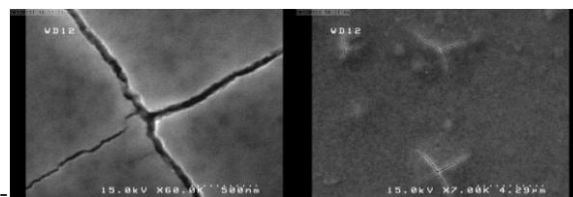


**Fig. 8:** XRD of PZT film.

The obtained XRD property shows formation of the single phase perovskite PZT that rectangular sign shows its trigonal phase. As seen, the biggest peak relates to tetrahedron PZT with the orientation (111). This indicates that PZT has been crystallized by placing in lower temperature. It must be noted that if PZT was placed at higher temperature, the orientations (100) and (011) of Tetrahedron phase would show themselves more. While one can only obtain PZT (111) on Pt (111), we succeeded to obtain PZT (111) by sol-gel method in low temperature that has considerable ferroelectric properties compared to other orientations.

### 3.5 SEM test results:

Non-conductor materials are usually coated by a thin layer of carbon, gold, or golden alloys. So, before imaging PZT, it is coated by a conductor layer (chromium) with a thickness of 13 Nm. SEM results are shown in the following.



**Fig. 9:** SEM pictures of the prepared PZT layer.

### 4. Conclusions:

As mentioned earlier, PZT deposition by sol-gel method is highly dependent upon speed and time of spin coat. As the speed gets more, the layer will become more homogeneous with fewer cracks. But simultaneously its thickness becomes less; so to reach the required thickness, the number of deposition times is increased. This problem can also be solved by the spinning time – to a very low extent. Spin speed 3000 rpm and spin time between 20 and 30 seconds are proper. A new work done in this paper was to study different types of solutions with different molar ratio. As it was seen, PZT solution with lead nitrate is a proper solution because by reduction of carbon rate, annealing temperature and time are reduced and so the rate of cracks is decreased too.

The solution 2-methoxyethanol has higher chelating due to having smaller colloidal molecules and provides tangibly lower viscosity of organic compounds. It helps removal of organic materials and so reduction of the



temperature required for PZT crystallization. According to the tests results, fewer cracks were observed by increase in the solution concentration.

5. *Suggestions according to the research findings:*

- 1- Increasing PZT capacitance by making a multi layer PZT
- 2- Obtaining Hysteresis loop by AFM method
- 3- Simulating the part with different parameters and optimizing the part by Ansys and Coventor software and comparing these two applications
- 4- Connecting PZT capacitor to one of the MOSFETs and building a non-volatile random memory (FeRAM)
- 5- Building PZT ferroelectric Nano switches for increasing speed and making switches smaller

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