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## ORGANIC CHEMISTRY | RESEARCH ARTICLE

# Sol-gel synthesis and characterization of lead titanate films

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**Abstract:** Lead titanate thin films have been deposited by spin coating on stainless steel substrate using the precursor sol, which was synthesized from acetylacetonate chelating with titanium isopropoxide and ethylene glycol as a solvent, in the sol-gel process. The influence of film thickness, pyrolysis temperature, and heating rate on (the formation of thin films) microstructure and morphology of thin films have been systematically investigated. The phase purity, particle size, and morphology are studied using X-ray diffraction and scanning electron microscopy (SEM) measurements. It is observed that the preferred oriented thin films could be obtained by pyrolysis of wet films at 150°C and annealed at 600°C at a slow heating rate of 4°C/min. It is also confirmed from X-ray patterns that the tetragonal perovskite structure of the titanate exists without any impurity peaks in the films formed. The TGA/DSC curves of the PT precursor show weight loss of ~7% and two exotherms at 492 and 522°C. They are attributed to the crystallization of tetragonal PbTiO<sub>3</sub>. SEM shows that the PbTiO<sub>3</sub> thin films annealed at 600°C for 1 h crystallized well. The FTIR analysis showed the characteristic absorption bands of Pb-O groups at 663 cm<sup>-1</sup> and Ti-oxo groups at 600 cm<sup>-1</sup>.



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Vijendra A Chaudhari has completed his PhD in Physics at Dr. BAMU, Aurangabad. He has published 28 research articles in international journals as well as conferences. He is a reviewer for many journals. He has expertise in synthesizing and investigating nanomaterials using variety of chemical methods.

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Recently, the authors have pursued research on perovskite-structured ferroelectric material which is widely used in many technological applications. They synthesized and characterized ferroelectric materials using various chemical routes to obtain nanostructural materials and also worked on electrical and dielectric properties in the MPB region.

### PUBLIC INTEREST STATEMENT

Development of nanoparticles and thin films is an emerging field for the researchers working in the area of nanomaterials. The paper deals with the sol-gel synthesis and characterization of lead titanate films. The influence of film thickness, pyrolysis temperature, and heating rate on (the formation of thin films) microstructure and morphology of thin films have been systematically investigated. The films have been characterized using X-ray diffraction, scanning electron microscopy, differential scanning calorimetry (DSC), and IR spectroscopic measurements. The sol-gel technique, for preparation of ferroelectric thin films, has many advantages and this method has attracted considerable interest due to its low cost and ease to integrate with semiconductor silicon technology.

**Subjects:** Condensed Matter Physics; Crystallography; Matter & Solid State Physics

**Keywords:** thin films; chemical synthesis; sol-gel growth; X-ray diffraction (XRD); electron microscopy (SEM); differential scanning calorimetry (DSC); FTIR spectroscopy; crystal structure; microstructure; thermal properties

## 1. Introduction

In recent years, there has been increased interest in lead-based ferroelectric thin films, such as  $\text{PbTiO}_3$  [PT],  $\text{Pb}(\text{Zr,Ti})\text{O}_3$  [PZT],  $(\text{PbLa})\text{TiO}_3$  [PLT], and  $\text{Pb}(\text{LaZr})\text{TiO}_3$  [PLZT], for their applications in a variety of piezoelectric device application (White & Turner, 1997), which include high-frequency ferroelectric sonar transducers (Bernstein et al., 1997), elastic surface wave devices (Cicco, Morten, & Prudenziati, 1996), new ultrasonic composite transducers (Cicco, Morten, & Prudenziati, 1997), pyroelectric infrared sensors (Łoziński, Wang, Uusimäki, & Leppävuori, 1998), microelectromechanical system devices (Polla & Schiller, 1995), torque sensors (Morten, De Cicco, & Prudenziati, 1994), and hydrophones (Chan et al., 1999). Pb-based ferroelectric films possess the merits of both bulk materials and thin films. Devices made from Pb-based ferroelectric thin films not only work at low voltage and high frequency, as they are compatible with semiconductor-integrated circuit, but also possess superior electric properties approaching near bulk values. Lead titanate ( $\text{PbTiO}_3$ ) ceramics are ferroelectric, with a transition (Curie) temperature of  $490^\circ\text{C}$  (Kim, Jun, & Hwang, 1999). Compared with other piezoelectric materials,  $\text{PbTiO}_3$  has many useful properties, including a high transition temperature, a low ratio for the planar-to-thickness coupling factor, a low aging rate of the dielectric constant, and a low dielectric constant (Kim et al., 1999). Therefore,  $\text{PbTiO}_3$  shows great promise as a stable pyroelectric (Yuhuan, Zhungyung, & Xiujuan, 1981) and piezoelectric material for high-temperature or high-frequency applications (Ikegami, Ueda, & Nagata, 1971).

To date, various deposition techniques, including pulsed laser deposition (Brazier, McElfresh, & Mansour, 1998), magnetron sputtering (Kim, Kim, & Lee, 1998), metal organic chemical vapor deposition (Shimizu & Shiosaki, 1995), sol-gel processing (Yang & Haile, 2006) etc., have been used to deposit films of Pb-based ferroelectric materials. Among these techniques, the sol-gel method has attracted considerable interest due to its low cost and ease to integrate with semiconductor silicon technology. The sol-gel technique, for preparation of ferroelectric thin films, has many advantages such as easier composition control, better homogeneity, nonvacuum process, lower processing temperature, and easier fabrication of large area films (Bao, Yao, & Zhang, 2000; Bao, Zhang, & Yao, 2000). In sol-gel process, for the fabrication of PT thin films, the most commonly used starting chemicals are lead acetate trihydrate, titanium tetra isopropoxide or tetra butoxide, and methoxyethanol; these have been widely selected as solvents due to their chelating properties and low viscosity. Generally, reflux and distillation are needed for synthesizing homogeneous precursor solution when lead acetate and titanium alkoxide are selected as raw materials and methoxyethanol as a solvent.

Sol-gel processing of  $\text{ABO}_3$ -type ceramics generally involves the following steps:

(i) preparation of a clear solution containing the A and B constituents in the required stoichiometry, (ii) hydrolysis of the solution leading to the formation of an inorganic polymeric  $(-\text{A}-\text{B}-\text{O}-)$  network (gel) through condensation reactions, and (iii) conversion of the gel to the ceramic by appropriate heat treatment. The sol-gel process is based on the chemistry of alkoxide.

Lead titanate has been reported to be prepared by a variety of sol-gel processes (Calzada, Sirera, Carmona, & Jimenez, 1995; Wright & Francis, 1993). In an all alkoxide sol-gel process, alkoxides of the constituent elements show different reactivities toward water so that the preparation of multi-component homogeneous systems is difficult. The high cost of the alkoxide reagents and the need to work under inert atmospheres are major disadvantages of using this system. Inorganic and organic salts have also been used for sol-gel processing of multicomponent systems when the use of constituent alkoxides becomes difficult or unnecessary. Calzada and Del Olmo (1990) have reported

a sol-gel process using inorganic precursors,  $\text{TiCl}_4$  and  $\text{Pb}(\text{NO}_3)_2$ . Alternate synthetic methods (Calzada et al., 1995; Kezuka, Hayashi, & Yamaguchi, 1989) developed for the sol-gel processing of lead titanate use lead acetate trihydrate,  $\text{Pb}(\text{OOCCH}_3)_2 \cdot 3\text{H}_2\text{O}$  as the lead precursor. In such systems, the lead salt has to be first dehydrated *in situ* by refluxing in 2-methoxy ethanol solvent except for a special system stabilized by diethanolamine (Takahashi, Matsuoka, & Yamaguchi, 1990; Takahashi & Yamaguchi, 1990). The variations in precursor solution chemistry as a result of the generation of free acid, ester, and water significantly affect the homogeneity of lead titanate (Ramamurthi & Payne, 1990). Another major disadvantage of this process is that the precursor solution obtained is extremely moisture sensitive (Lakeman & Payne, 1992), and has to be stored and processed under controlled dry atmosphere. Although the system, alkoxide-diethanolamine, has been reported to be stable to moisture, the presence of diethanolamine lowers the concentration of metallic components in the gel. In this work, basic lead acetate is used as the lead precursor. The easy solubility of basic lead acetate in ethylene glycol helps in the formation of a highly stable and stoichiometric precursor solution with titanium isopropoxide in acetylacetone. Solvents like acetylacetone and ethylene glycol act as donor ligands (Nabavi, Doeuff, Sanchez, & Livage, 1990) to titanium isopropoxide and control their hydrolysis and polycondensation reactions. The procedure used can be summarized by the following equation.



In this study, the PT thin films on stainless steel substrate were prepared by the sol synthesized from the acetylacetone/ethylene glycol route by a sol-gel method. The growth of thin films has been studied as a function of thin film conditions, such as film thickness, pyrolysis temperature, and heating rate. The thin film was deposited by spin coating technique. The film thickness, pyrolysis temperature, and heating rate influence the microstructure of the deposited thin films. The films having thickness more than the critical film thickness help to control the nucleation sites. The films' annealing temperature influences the formation of structural phase.

## 2. Experimental

Lead titanate thin films were prepared by the sol synthesis from acetylacetone/ethylene glycol route by a sol-gel method. For sol preparation, lead acetate (99.9% purity; Aldrich Chemicals, 3.123 g) was dissolved in ethylene glycol on reflux at 120°C for 1 h to decrease the residual water. Acetylacetone was used as the chelating agent to mix with titanium isopropoxide (99.9% purity, 5 ml), the molar ratio of acetylacetone/titanium isopropoxide being 4. The above solution was also refluxed at 120°C for 1 h and then about 15 ml of ethylene glycol was added to the acetylacetone/titanium isopropoxide solution. The above acetate solution was added to the titanium solution and refluxed at 120°C for 4 h, then 5 ml of deionized water was added for further hydrolysis and polycondensation until the solution becomes a sol and easy for spin coating.

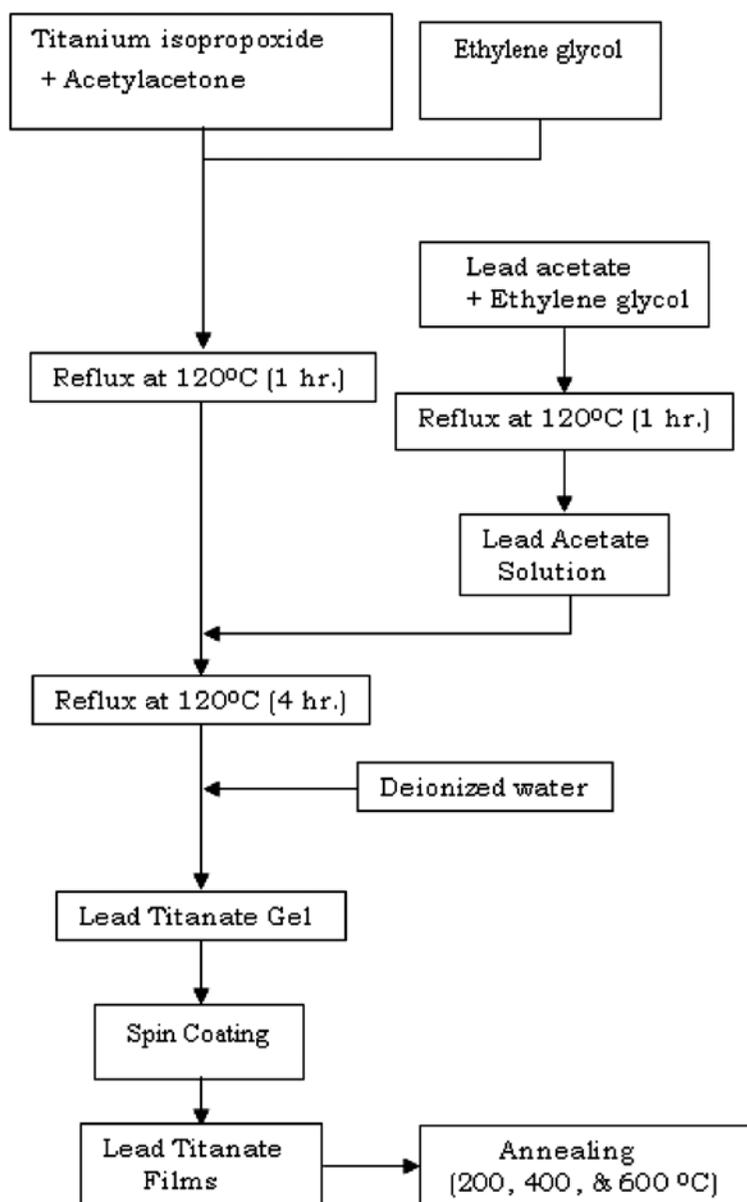
Prior to the deposition, the metallic substrates (stainless steel (10 × 10 × 5 mm)) were thoroughly polished using zero fine grade (3/0) polish paper (supplied by Kohinor Products, India) and then washed with liquid detergent followed by ultrasonic cleaning with double-distilled water. In order to remove oily substances from the surface, cleaned substrates were etched in 10%  $\text{H}_2\text{SO}_4$  for 2 min and finally ultrasonically cleaned with double-distilled water.

The thin films were deposited by a spin coating technique by utilizing the as-prepared PT-sol (pre-coating 1,000 rpm for 10 s. followed by 4,000 rpm for 30 s.). The as-deposited film was then pyrolyzed on

a hot plate for 5 min to evaporate residual organic species, respectively, for each layer deposition. The deposition and pyrolysis procedures were repeated and then the films were finally annealed at various temperatures (200, 400, and 600°C) for 2 h, and a slow cooling rate of 2°C/min was used to avoid cracking of the films. In the case of thin films, film thickness (repeated by coating layers) and pyrolysis temperatures were studied systematically to investigate their influence on the formation of PT films. However, the coating layers and annealing temperature were 1–6 and 200–600°C, respectively. Figure 1 shows the schematic diagram of lead titanate using the sol-gel technique.

The crystal structure and orientation of the PT films synthesized on stainless steel substrates were examined using an X-ray diffractometer (Xpert Pro-PAN Philips) with Cu-K $\alpha$  radiation source. The surface morphology was studied using scanning electron microscopy (SEM, JEOL JSM-6360 A). For determining the average particle size from full width at half maximum (FWHM), the intensity of Bragg peak

Figure 1. Schematic diagram of lead titanate using the sol-gel technique.



(1 0 1) was used. The grain morphology and average grain size were determined using the mean linear intercept method (Brandon & Kaplan, 1999). Differential scanning calorimetry (DSC) measurements were carried out at 10°C/min with DSC-60 Shimadzu for measuring the phase transition temperature. The FTIR data of  $\text{PbTiO}_3$  were recorded using computer-controlled FTIR-8400 Shimadzu spectrophotometer in the wave number range 4,000–500  $\text{cm}^{-1}$  in a KBr medium at room temperature (25°C). The experiment was carried out in an inert atmosphere. The optical resolution for data collection was 4  $\text{cm}^{-1}$ .

### 3. Results and discussion

Figure 2 shows the X-ray diffraction (XRD) spectra of  $\text{PbTiO}_3$  thin films prepared by various layer coatings, pyrolyzing at 150°C, and then annealing at 600°C for 1 h by a heating rate of 10°C/min. Figure 3 shows the XRD pattern of  $\text{PbTiO}_3$  thin films prepared from six-layer coating and annealed at (a) 200°C, (b) 400°C, and (c) 600°C temperature for 1 h by a heating rate of 10°C/min. The appearance of the sharp nature of all the Bragg peaks observed in the diffractogram suggests the crystalline nature of the films. The  $d$ -values and intensities of the peaks agree very well with those given in ASTM data card (78-0298) for  $\text{PbTiO}_3$ , suggesting that the films were polycrystalline and possessed tetragonal structure. Table 1 lists the  $d$ -observed and  $d$ -calculated values for the film deposited with six-layer coating, annealed at 600°C. It is observed from the XRD patterns (Figure 2) that the films with four and six layers show a single perovskite phase, without any other noticeable phase. During the thin film preparation, it was observed that the annealing temperature was higher, and the heterogeneous nucleation and growth of perovskite grain at the interface between the film and the substrate occurred much easier because of a higher thermal energy applied during the nucleation process. From the observation of XRD patterns, the full width at half maxima (FWHM) of (0 0 1)/(1 0 0) peaks were found to be broader with the increase in film thickness. In addition, the (1 1 0) and (1 1 1) peaks increased gradually with the increase in film thickness. When the coating layers of PT thin films were more than four, the films thicker than the critical film thickness are too bulky to control the nucleation sites; therefore, the nucleation sites of the other planes, such as (1 1 0) and (1 1 1) planes, as well as the main peak of (0 0 1)/(1 0 0) plane are observed in the films of four- and six-layer coatings. The XRD patterns of the six-layer coating films, prepared from the sol pyrolyzed at 150°C and annealed at different temperatures (200, 400, and 600°C), show that the intensity of Bragg peaks increases with increase in annealing temperature. Specially, the films annealed at 400 and 600°C exhibit peaks of strong intensity.

Figure 2. XRD patterns of  $\text{PbTiO}_3$  thin films prepared from various layer coatings.

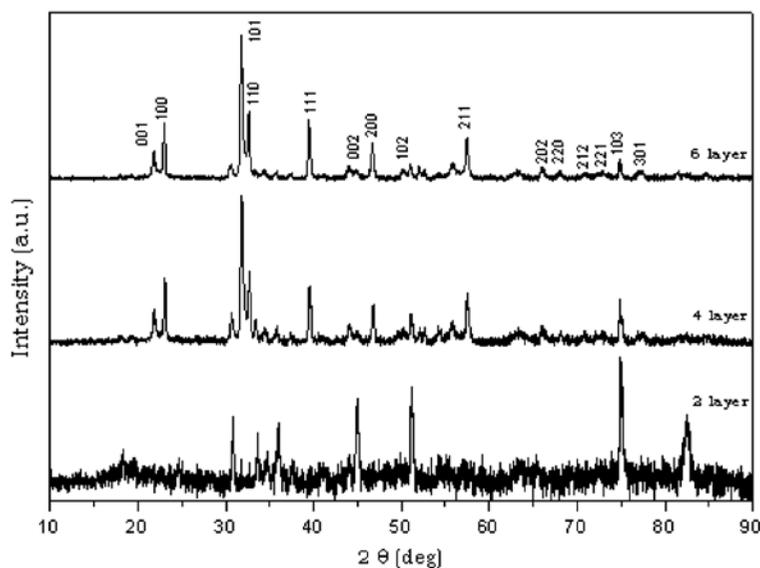


Figure 3. XRD patterns of  $\text{PbTiO}_3$  thin films prepared from six-layer coating and annealed at (a) 200°C, (b) 400°C, and (c) 600°C temperatures for 1 h.

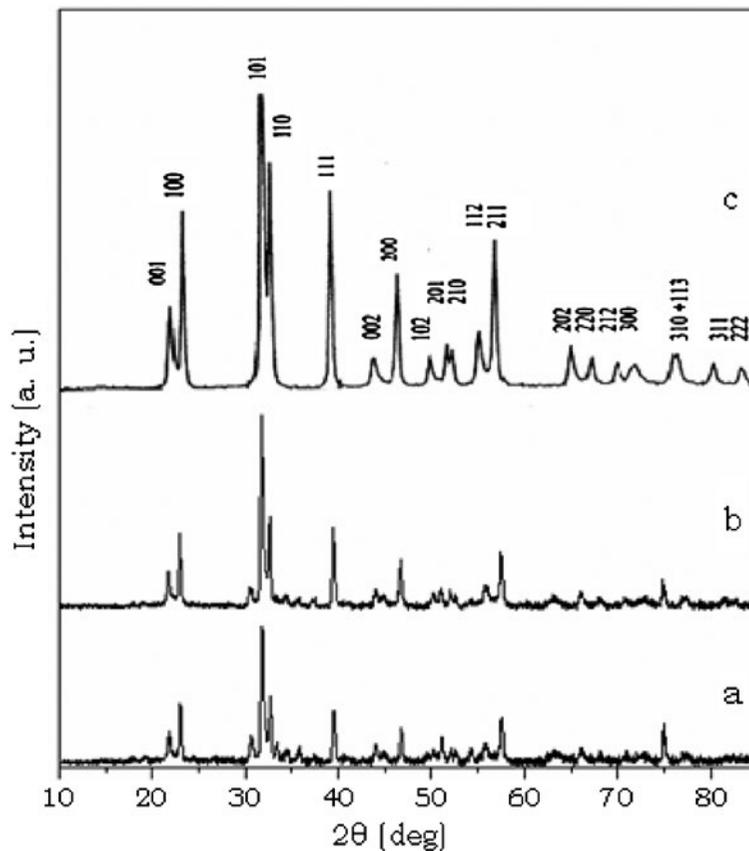


Table 1. List of the  $d$ -observed and  $d$ -calculated values for the film deposited with six layers, annealed at 600°C

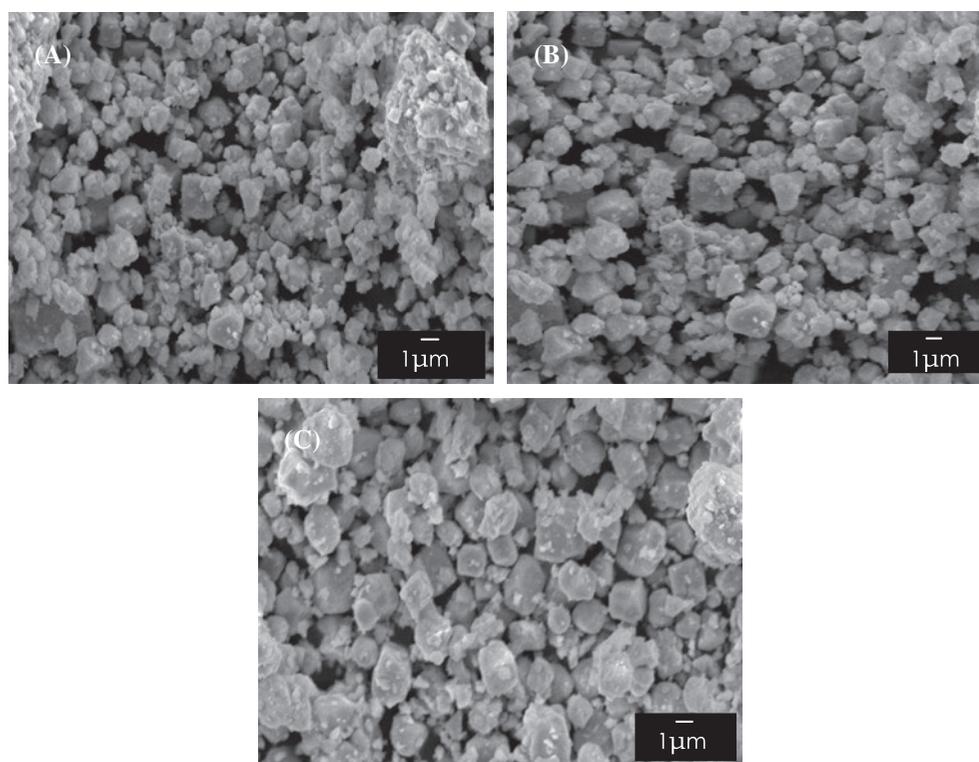
$h$	$k$	$l$	$d$ (obs.)	$d$ (calc.)
0	0	1	4.1491	4.1403
1	0	0	3.9285	3.9156
1	0	1	2.8442	2.8449
1	1	0	2.7723	2.7687
1	1	1	2.3000	2.3015
0	0	2	2.0727	2.0701
2	0	0	1.9580	1.9578
1	0	2	1.8296	1.8301
2	0	1	1.7686	1.7699
2	1	0	1.7493	1.7511
1	1	2	1.6563	1.6579
2	1	1	1.6113	1.6128
2	0	2	1.4205	1.4224
2	2	0	1.3833	1.3801
2	1	2	1.3357	1.3369
3	0	0	1.3017	1.3016
3	1	0	1.2430	1.2382
3	1	1	1.1849	1.1863
2	2	2	1.1484	1.1507

Figure 4(A–C) shows the scanning electron micrograph of the surface of the sol-gel-grown  $\text{PbTiO}_3$  films on stainless steel substrates. The films were annealed at 200, 400, and 600°C for 15 min and allowed to cool to room temperature for 1 h. The films did not show any cracks and the film surface shows a crystalline phase. The films were prepared under the same deposition conditions and underwent various post-deposition annealing treatments. The films showed fine-grained microstructure. As the annealing temperature increased from 200 to 600°C (Figure 4), the grain size gradually increased and this may be due to the improvement in the perovskite crystallization at high annealing temperature. The average grain size was determined using the linear intercept method and the values of the average grain size are listed in Table 2. The particle size was deduced from the half-width of the XRD peak at  $2\theta = 39.40$  using Scherrer's equation. The average particle size determined from the peak broadening in XRD was 34 nm. Table 2 also includes the value of average particle size determined from (FWHM) of (1 0 1) peak. The TGA/DSC curve (Figure 5) shows a weight loss of ~7% accompanied by four exothermic peaks at 300, 345, 495, and 523°C, which are attributed to the loss of water from the hydroxyl group of the precursor. Weight loss occurring below 100°C was due to the loss of adsorbed water. The small exotherm at 523°C can be attributed to the heat of crystallization of lead titanate (Kumar, Marimuthu, Patil, Ohya, & Takahashi, 1996). Figure 6 shows the FTIR spectra heated at different temperatures. Two bands, at ~3,400 and 1,625  $\text{cm}^{-1}$ , wave number is assigned to the stretching vibrations of OH groups, Pb–O groups from the peak at 663  $\text{cm}^{-1}$ , and Ti–oxo groups from the peak at 600  $\text{cm}^{-1}$ . The weight loss accompanied by crystallization around 500°C can be attributed to the loss of water as show by the FTIR analysis (Nakamoto, 1978). The broad peak around 3,500  $\text{cm}^{-1}$  due to the hydroxyl groups became weaker on heat treatment but still remained small even after the crystallization at 525°C, and then disappeared.

#### 4. Conclusion

$\text{PbTiO}_3$  thin films with submicrometer particle size have been deposited by the sol-gel method using spin coating technique by employing lead acetate as the lead precursor. When the coating layers of

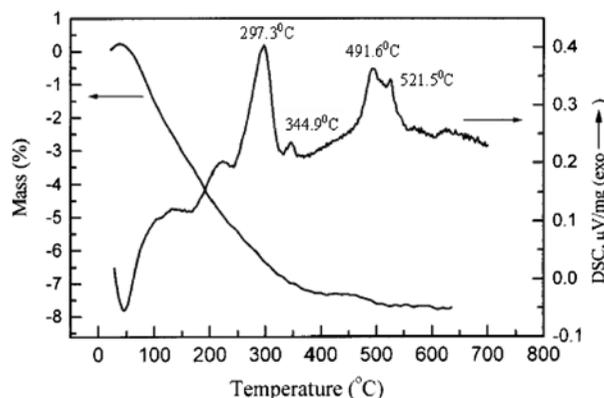
Figure 4. Scanning electron micrograph of the surface of the sol-gel-grown  $\text{PbTiO}_3$  films (A–C).



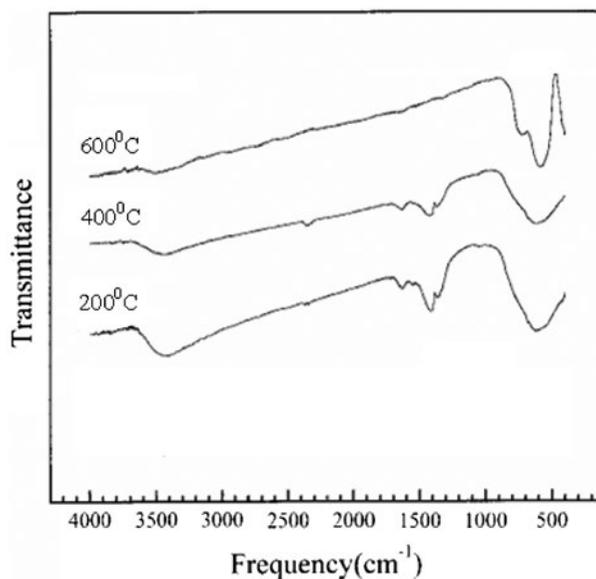
**Table 2. Particle size (XRD) and grain size (SEM) of PbTiO<sub>3</sub> thin films annealed at different temperatures**

Sintering temperature (°C)	Particle size (nm)	SEM (nm)
200	43	215
400	75	432
600	115	643

**Figure 5. TGA/DSC curve of PbTiO<sub>3</sub> precursor heated at a heating rate of 10°C/min.**



**Figure 6. FTIR spectra of PbTiO<sub>3</sub> precursor heated at different temperatures.**



PT thin films were more than two, all the diffraction peaks were obtained in the pattern. The influences of film thickness, pyrolysis temperature, and heating rate on the characterization and microstructure of PT thin films were investigated systematically. When the coating layers of PT thin films were more than four, the films thicker than the critical film thickness were too bulky to control the nucleation site; therefore, the nucleation sites of other planes, such as (1 1 0) and (1 1 1) planes, as well as the main peak of (0 0 1)/(1 0 0) plane were obtained in the films of four- and six-layer coatings. However, it is also found that the film annealing temperature can effectively change the crystallization temperature, and alter the driving force governing the formation of perovskite phase. From the SEM studies, it is found that as the annealing temperature increases from 200 to 600°C, the grain size gradually increases.

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