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$PbTiO_3$ Thin Film Obtained At Low Temperature By Metallo-Organic Decomposition Processing

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ABSTRACT

This work had the objective of studying the lead titanate thin film on quartz, n-type silicon and platinum substrates by the metallo-organic decomposition processing. Thin film were heat treated between 450-750°C and characterized by thermal analysis, X-ray diffraction, scanning electron microscope and FT-IR spectroscopy. A low temperature synthesis was possible with a crystallites material heat treatment at 450°C.

KEYWORDS

$PbTiO_3$;
Ferroelectric thin film;
MOD;
Thermal analysis.

INTRODUCTION

Ferroelectrics materials have been applied to many electronic and optical devices utilizing their excellent dielectric, ferroelectrics, and piezoelectric and optical properties. $PbTiO_3$ is well-known ferroelectrics materials are used for the applications such as nonvolatile ferroelectrics memories, pyroelctric detectors, optical wave-guides, spatial light modulators and devices[1]. Ferroelectrics thin films have been prepared by a variety methods such as Rf sputtering, metal organic chemical vapor deposition, laser ablation, sol-gel and metallo-organic decomposition processing[2]. The MOD solution deposition has been used by almost every electro ceramic research and development organization through the world to evaluate thin films, ferrites, high temperature superconductors etc. had a significant impact relatively on the processing and appears promising for meeting the stringent quality requirement for devices. In the present work a systematic research was performed to investigate the optimum growth condition for the ease. Metallo-organic decomposition (MOD) processing using spin coating technique has been applied to the formation of crack free and polycrystalline $PbTiO_3$ thin films deposited on quartz, n-type silicon and platinum substrates.

EXPERIMENTAL

$PbTiO_3$ thin films were synthesized by metallo-organic decomposition processing as summarized in figure 1. Lead nitrate solution was added into the 2-ethyl hexanoic acid followed by the addition of 5% KOH solution to maintain pH 7 with continuous magnetic stirrer at 60^{0}C. Pale yellow gummy lead hexaonate soap was settled down at the bottom and extracted with distilled water and xylene. Tetra butyl orthotitante was dissolved in the xylene. In order to determine stiochiometric and effect of molar ratio of above solutions (1:1) to the formation of $PbTiO_3$. Polyethylene glycol as a binder was poured into the precursor stock solution. Filtered solution was dispensed using a 0.2 μm syringe filter and refluxed directly to adjust the homogeneity of the solution and spin coated at a speed of 4000 rpm for 60sec on substrates. After each coating, the films were heated at 300^{0}C in a furnace for 5min. After every two coatings, the films were kept in a furnace already maintained at 300^{0}C for 20min for densifica-

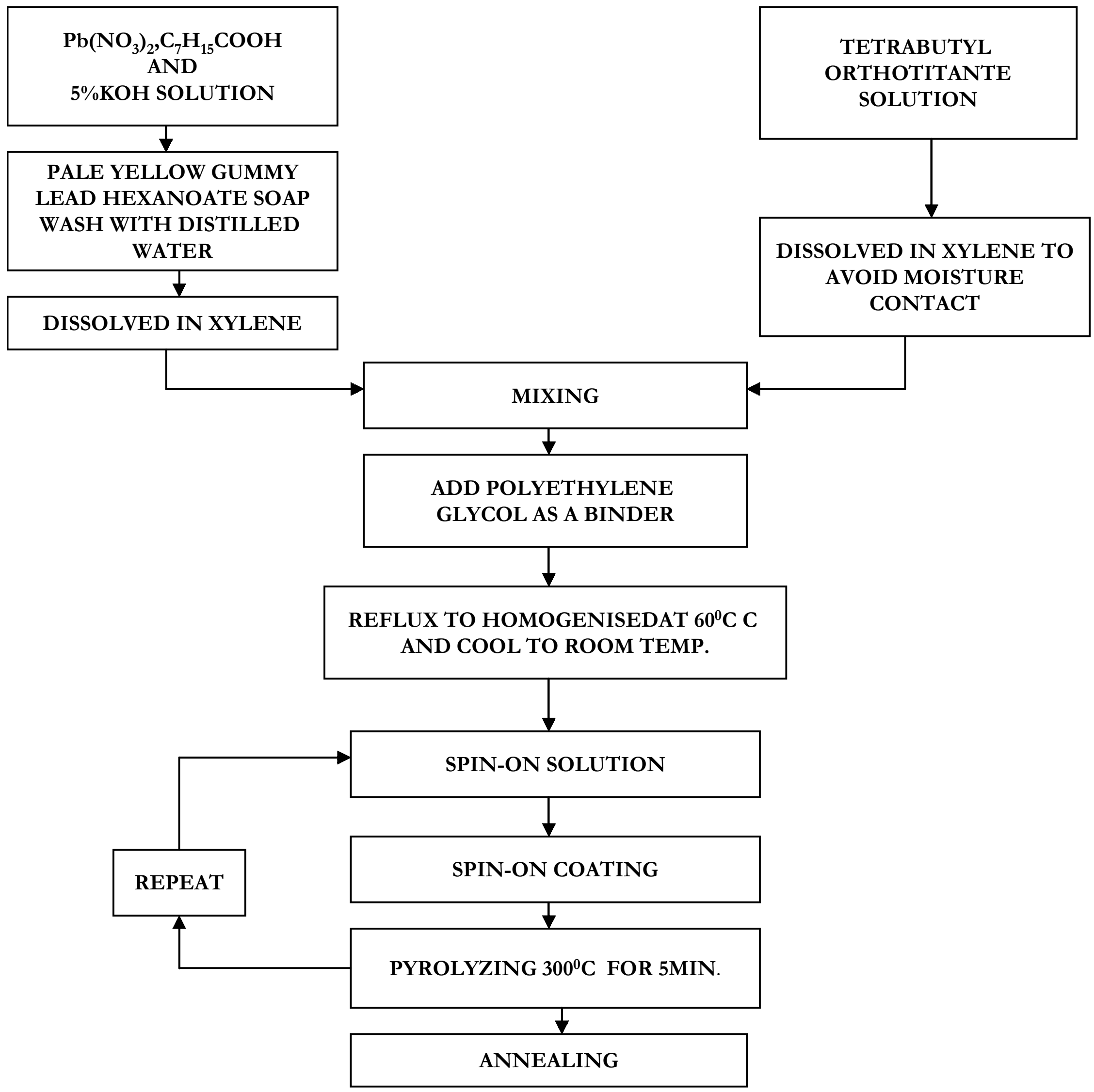

Figure 1: Flow diagram for preparatory of precursor and $PbTiO_3$ thin films by MOD method.

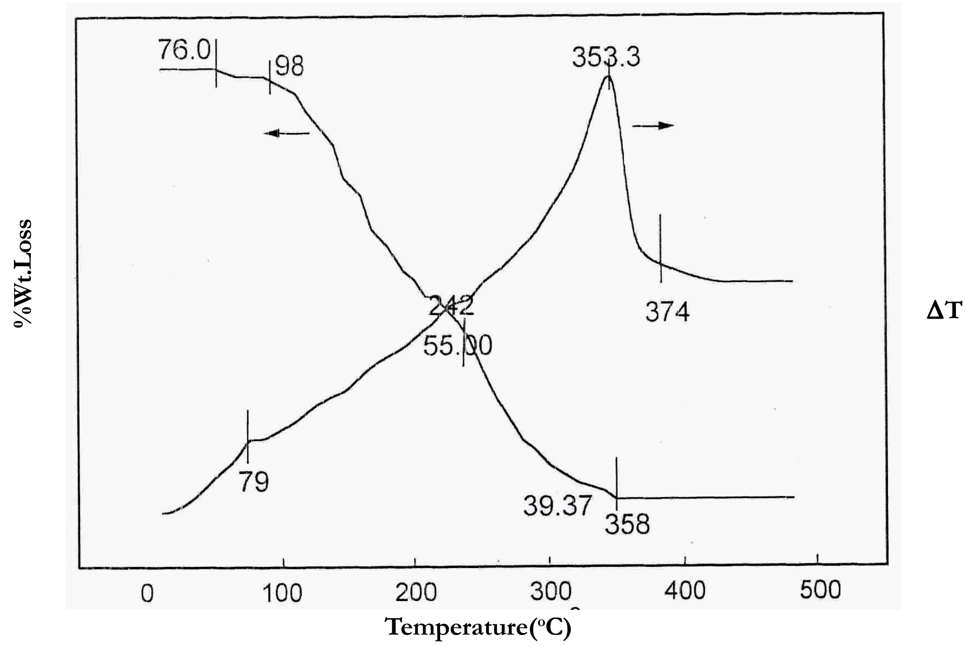

Figure 2: TGA/DTA curves of the $PbTiO_3$ thin film

tion and removal of volatile organics. The films were finally annealed at 450°C-750°C for 2h. in an oxygen atmosphere to obtain crystallization of film. Simultaneously thermo gravimetric and differentials thermal analysis were used to monitor the decomposition and pyrolysis of the film at a heating rate of 20°C min^{-1}. The film structure was identified by X-ray diffraction analysis using PW3710 diffractometer with CuK radiation (λ =1.54056Å). Scanning electron microscopy (JSM6100) was used to observe the grain size and the morphology of the $PbTiO_3$ thin film. The FTIR (Perkins Elmer spectrum Rx) spectrometer was used to carry out the infrared spectroscopic studies.

RESULTS AND DISCUSSION

Figure 2 illustrate the thermo gravimetric curve of the lead titanate thin film formation in xylene. It indicates two decomposition steps. The first one was attributed to the loss of water and xylene. The second step was ascribed to the decomposition of organic matter. A major weight loss below 200°C that is due to the evaporation of xylene solvents and polyethylene glycol. Further weight loss in the range 240°C -300°C corresponds to the removal of hexaonate group. However, when lead 2-ethylhexanoate is mixed with tetra butyl orthotitante the reactivity during pyrolysis is so high that crystalline $PbTiO_3$ is formed below 360°C[3]. The DTA curve shows a highly exothermic peak around 353°C associated to considerable mass loss. This peak is related to combustion of organic material.

Figure 3a and 3b shows XRD spectra of the $PbTiO_3$ thin film annealed at 450°C and 750°C for 2h.The films having Pb_2TiO_3 polychore phases are seen at 450°C, however at 450°C $PbTi_3O_7$ coexisted with $Pb_2Ti_2O_6$. High intensity peaks at 450°C confirms the c-axis orientation ratio. The calculated ratio ~ 0.32 which is slightly equal to the value of 0.37 by[3-5]. The perovskite structure of $PbTiO_3$ thin film at 750°C obtained (Figure 3b). Figure 4 shows the surface structure of $PbTiO_3$ thin film deposited on silicon substrate at 750°C. Thin film annealed at 550°C exhibited no resolution of micro structural features. As increasing the temperature up to 650°C, the grains range from 200nm-600nm. Further at 750°C temperature, the homogeneous microstructure consisting of uniform grains size of about ~500nm has been observed. This is because grain growth oc-

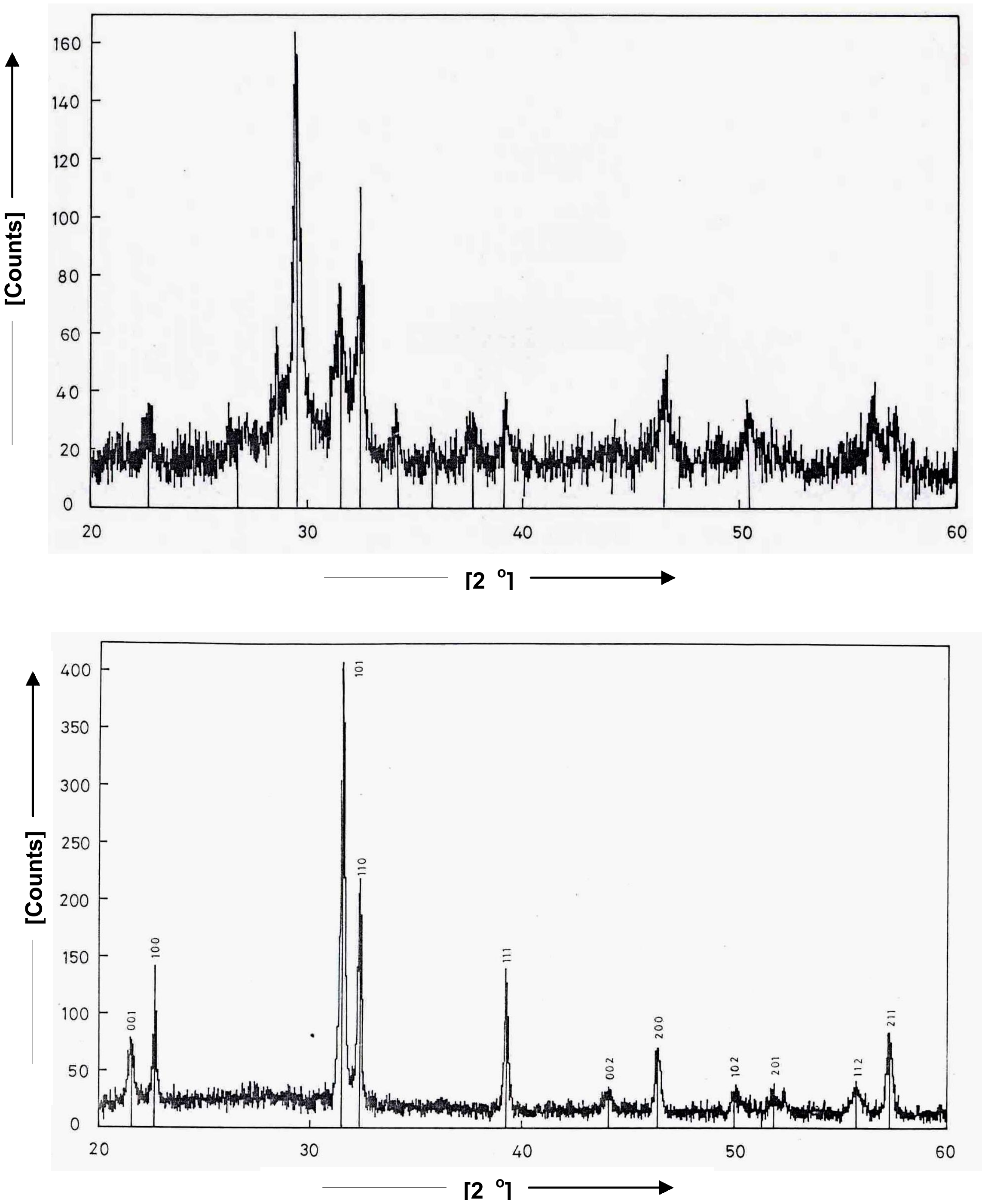

Figure 3: XRD pattern of $PbTiO_3$ thin film (a) at 450^{0}C (b) at 750^{0}C.

curs with temperature thereby, increasing the grain size. This is due in surface mobility with annealing, thus allowing the film to lower its total energy by growth of grains and decreasing the grain boundary area[6]. The FT-IR absorption spectra of $PbTiO_3$ thin film annealed at 750^{0}C as shown in figure 5. Four absorption bands were observed corresponding to 3851.7cm^{-1}, 2360.6cm^{-1}, 873.7cm^{-1} and 515.4cm^{-1} confirms the presence of Pb-O, Ti-O and Pb-Ti stretching.

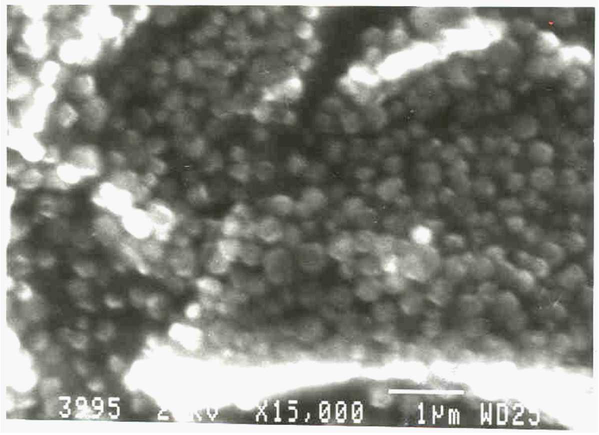

Figure 4: SEM of $PbTiO_3$ thin film on silicon substrate at 750^{0}C.

c-axis oriented $PbTiO_3$ thin film in which MOD Processing has observed 32% of c-axis orientated successfully. Therefore this process is simple, low cost and time saving.

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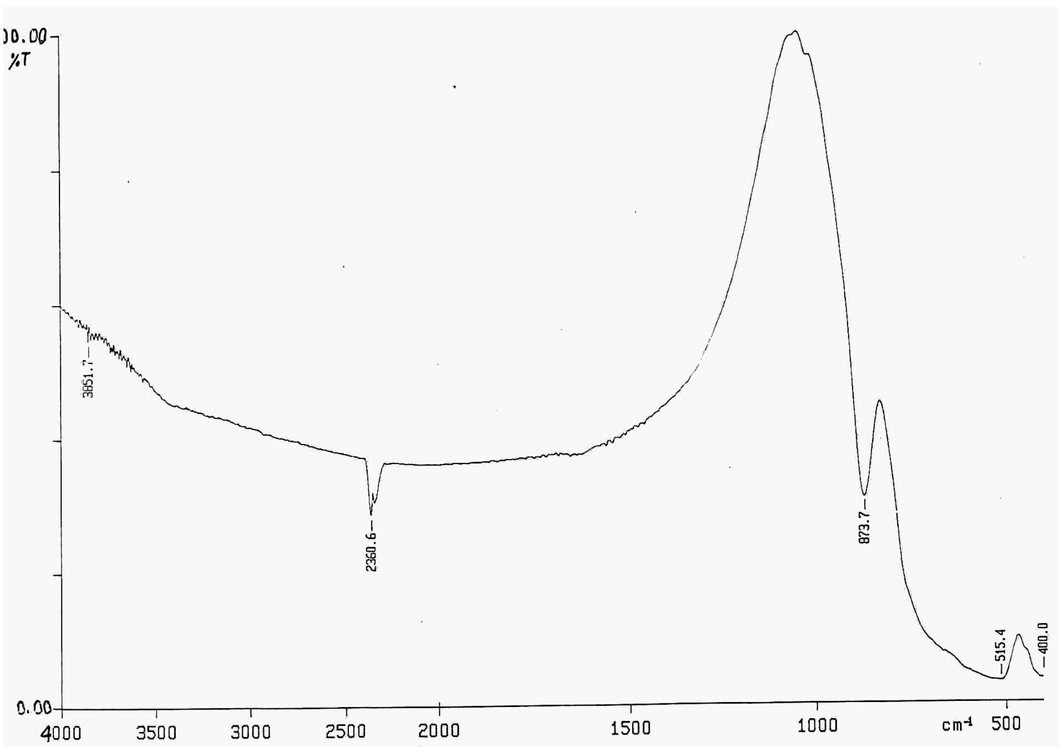

Figure 5: FT-IR spectra of $PbTiO_3$ thin film on silicon substrate at 750^{0}C.

CONCLUSIONS

The route of fabrication of PT thin films through metallo-organic decomposition (MOD) processing at low temperatures has been investigated and optimum conditions for obtaining homogeneous and crack free thin films have been determined. Slightly

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