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Growth of Sol-Gel Derived Lead Titanate Thin Film for Non-Volatile Memory Device Application

Khin Moe Thant[1] and Yin Maung Maung[1]

Abstract

Sol-gel derived lead titanate transparent sol is firstly prepared from lead acetate trihydrate and titanium isopropoxide as starting materials. It is refluxed with Oil-bath at 110°C for 6 h and lead titanate sol-gel is formed. Dynamic viscosity measurement is carried out by viscometer. The precursor sol is deposited on defect-free and highly polished p-Si substrate by local-made spin –coating machine. Scanning Electron Microscopy (SEM) analysis is carried out to examine the microstructural properties of fabricated film. Before the contact process, front and counter metallizations are done. 100 kHz C-V characteristics is observed by LCR meter (Digital Impedance Analyzar: Quch Tech: 1730) and gives the memory function. 100 kHz P-E hysteresis loop is also measured by Sawyer – Tower circuit. Hysteresis parameters such as spontaneous polarization density, remanent polarization density and coercive field are evaluated.

Key words: C-V characteristics, P-E hysteresis loop, SEM analysis.

Introduction

In recent years, ferroelectric materials have become very interesting materials for use in nonvolatile random access memory (NVRAM) (Hirotaka, 2005). Ferroelectric materials have attracted much attention for applications to ferroelectric random-access memories and microelectromechanical systems because of their excellent pyro-, piezo- and ferroelectric properties (Mamoru, 2005).

Ferroelectricity describes a collection of phenomena which contribute to the generation of a spontaneous electric polarization in materials called ferroelectrics. Spontaneous polarization is a result of the materials having a unique polar axis. The important point is that the direction of the spontaneous polarization can be switched by an external electric field (Matthew, 2001). The control of bistable polarization states in ferroelectric perovskite oxides by applying an electric field is the underlying basis of their applications to nonvolatile memories, piezoelectric devices and uncooled infrared detectors (Yuji, 2005).

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Experimental Procedure

The first step of this work was the mixing and stirring of lead acetate trihydrate (4.93 g) and ethylene glycol solvent (1.55 ml) by magnetic stirrer with constant speed (500 rpm) for 4 h. Next, the titanium isopropoxide (2.5 ml) and triethanolamine (TEA) (4 ml) were mixed and stirred by magnetic stirrer for 4 h in N_2-atm of glove-box. It was reluxed at 110 °C for 2 h in Oil-bath. After cooling, yellow liquid was significantly formed. After that resulting two sols were mixed to form the complete mixture. The above product sols were poured into 3-neck flask and refluxed at 110 °C for 6 h. Finally, lead titanate sol was formed. Dynamic viscosity was measured to be 65cP. The sol-gel was deposited onto cleaned p-Si (100) substrate by local-made spin-coating machine. The rotational speed was set 800 rpm and spinning time was 120 s. The substrate temperature was 400 °C. Managements of process temperatures were 500 °C, 550 °C, 600 °C, 650 °C and 700 °C maintained 1 h. Thus lead titanate film was formed. The film thickness was calculated to be 4.68 μm and it was found to be within the range of accepted value for thin film.

Results and Discussion

The $PbTiO_3$ (PT) thin films were thus obtained on p-Si substrates at different process temperatures. As the material evaluation, PT film was examined by SEM. Fig.1 (a~e) represented the SEM photo-graphs of the films at 500 °C, 550 °C, 600°C, 650 °C and 700 °C. Moreover, the images became rough after it was treated at 650 °C and 700 °C in contrast to the smooth the film treated at 500 °C, 550 °C, and 600 °C. The orientation of each film was almost the same and related to the substrate orientation. All films looked little dense except the film at 500 °C. Memory effect of C-V characteristic was measured to study whether the ferroelectric films could control the p-Si substrate. 100 kHz C-V characteristics of PT films were measured by LCR meter (Digital Impedance Analyzer) and described at Fig. 2 (a~e). From the figure, it was clearly observed that three distinct regions were formed. Moreover, it was found that the capacitance value went steadily in inversion state, gradually increased through the depletion region and almost reached its saturated value in accumulation region. Process temperature dependence of the memory window for PT film was given as Fig. 3 (a). As it was seen in figure, it was significant that memory window was gradually increased with increasing process temperature. The change in threshold voltage with respect to process temperature was shown in Fig. 3 (b). It was found that all threshold voltages were found positive and threshold voltage (swept up) was the largest at 600 °C and the film (500 °C) exhibited the smallest degree of threshold voltage. Moreover, the threshold voltage (swept down) was gradually increased with increasing process temperature. From Fig. 3 (c), all flat band voltage was examined to be negative values. To examine the ferroelectricity of PT film, hysteresis loop at the applied frequency of 100 kHz was tested by Sawyer-Tower circuit. Fig. 4 (a~e) showed the process temperature dependence of the hysteresis loop. These loops were symmetric and slim in shape. Saturation properties (2Pr–V) at different process temperatures were plotted in Fig. 5 (a). From the figure, it was clear that the remanent polarization density was linearly increased with increasing process temperature at low voltage region and reached the constant value after 6V. The spontaneous polarization densities were also derived from ferroelectric hysteresis loops. Dispersion of spontaneous polarization density at process temperature was illustrated at Fig. 5 (b). The coercive field of the fabricated film could be evaluated from hysteresis loops. Variation in coercive field and process temperature of PT films was described in Fig. 5 (c).

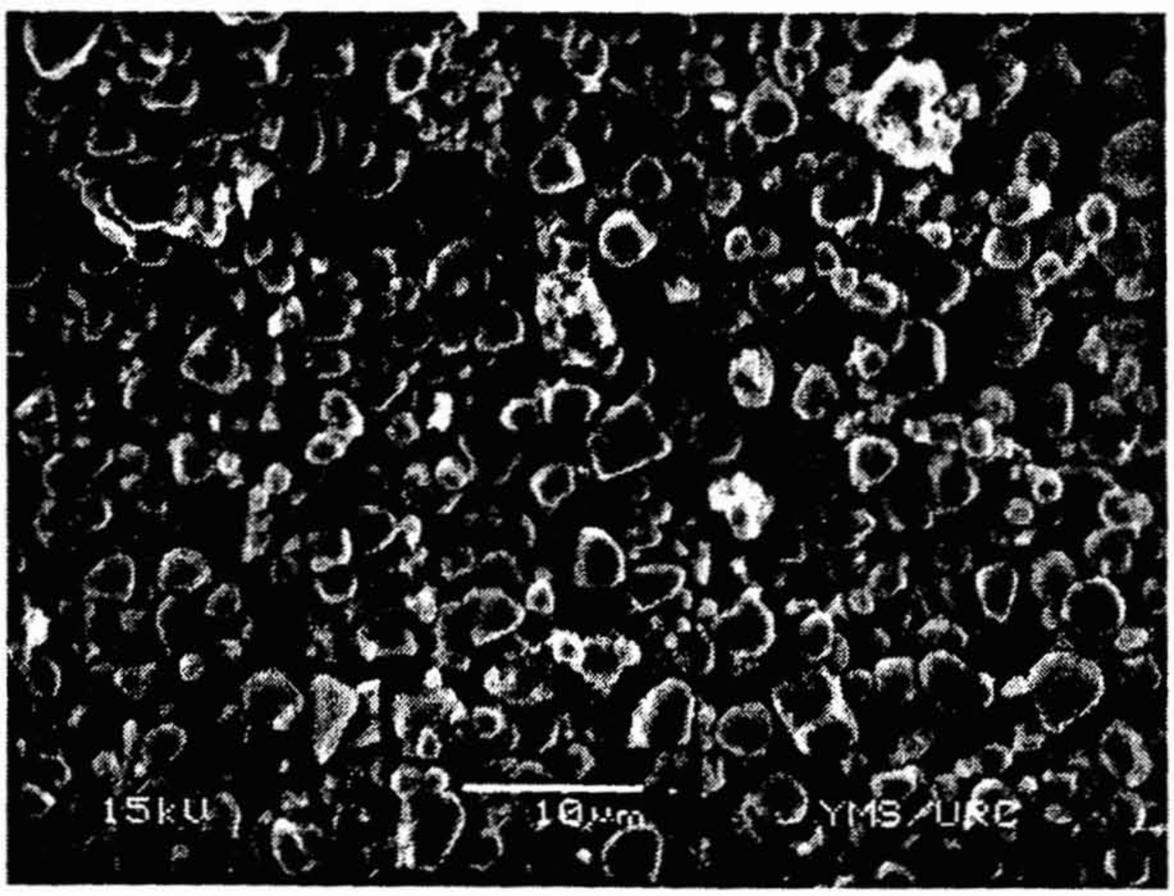

Figure 1. (a) SEM image of $PbTiO_3$ film (process temperature at 500 °C)

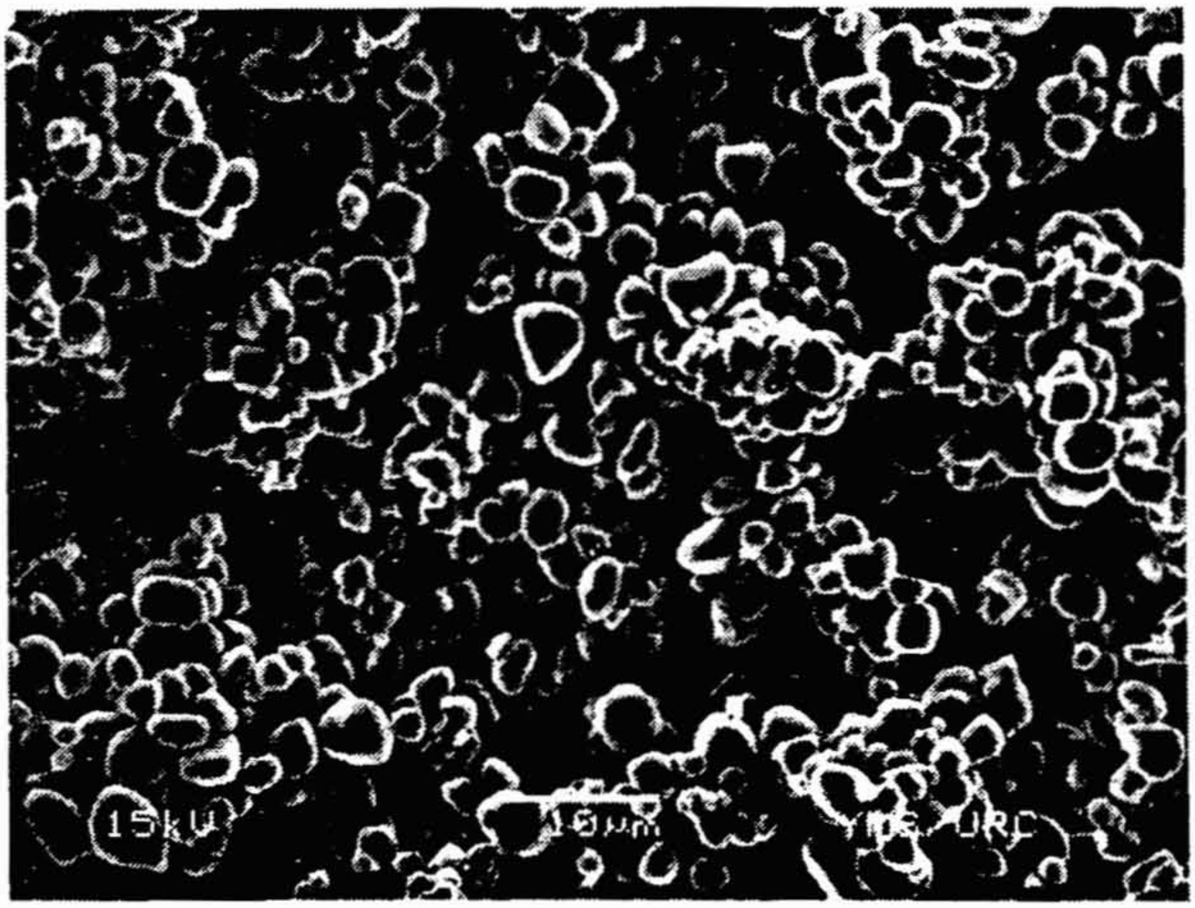

Figure 1. (b) SEM image of $PbTiO_3$ film (process temperature at 550 °C)

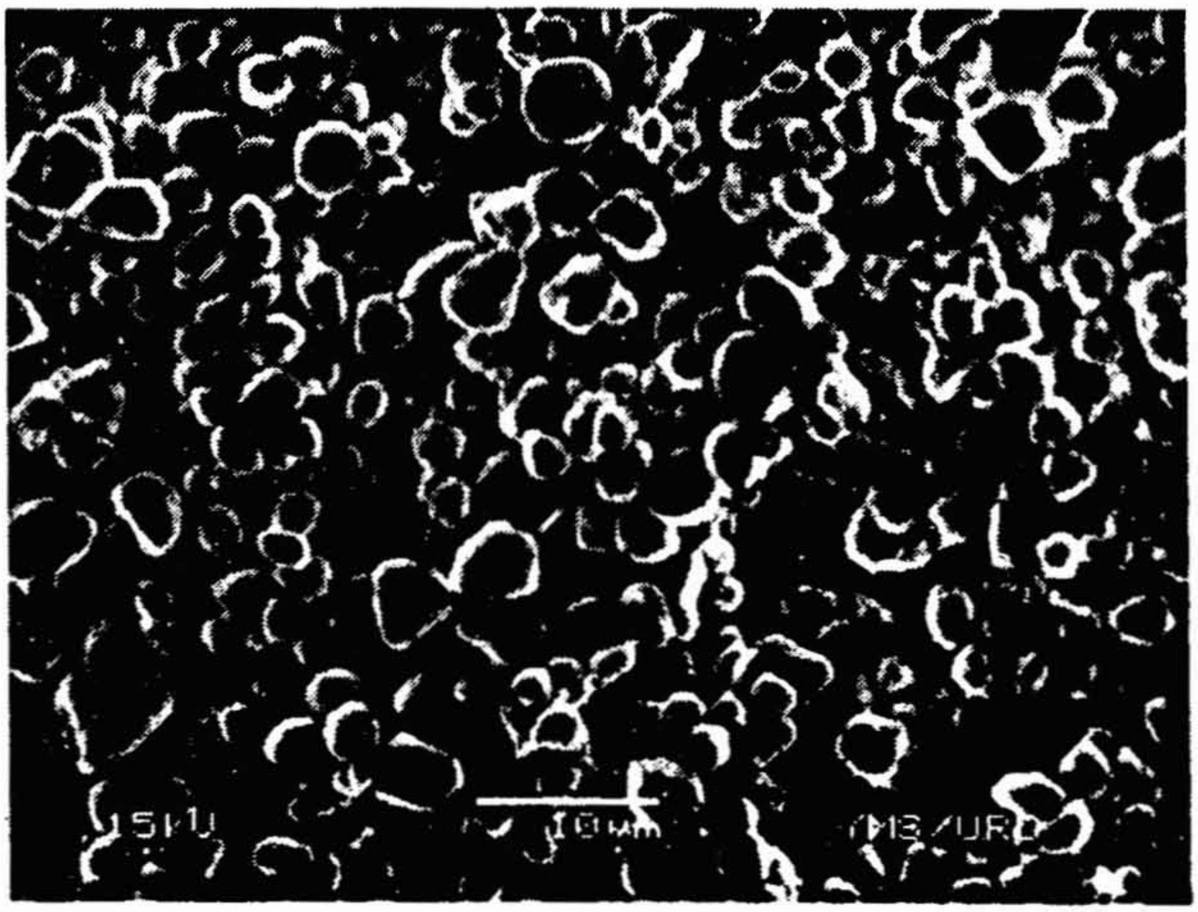

Figure 1. (c) SEM image of $PbTiO_3$ film (process temperature at 600 °C)

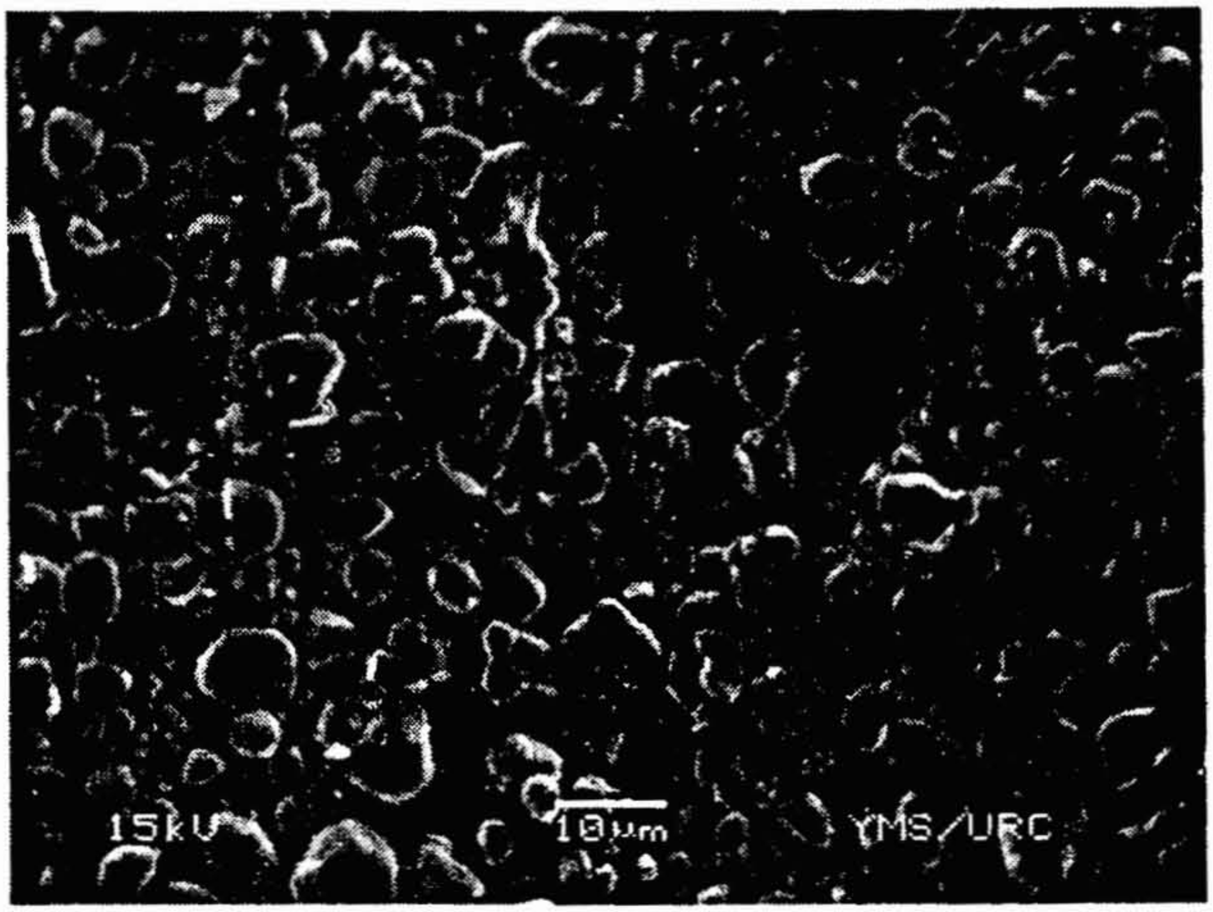

Figure 1. (d) SEM image of $PbTiO_3$ film (process temperature at 650 °C)

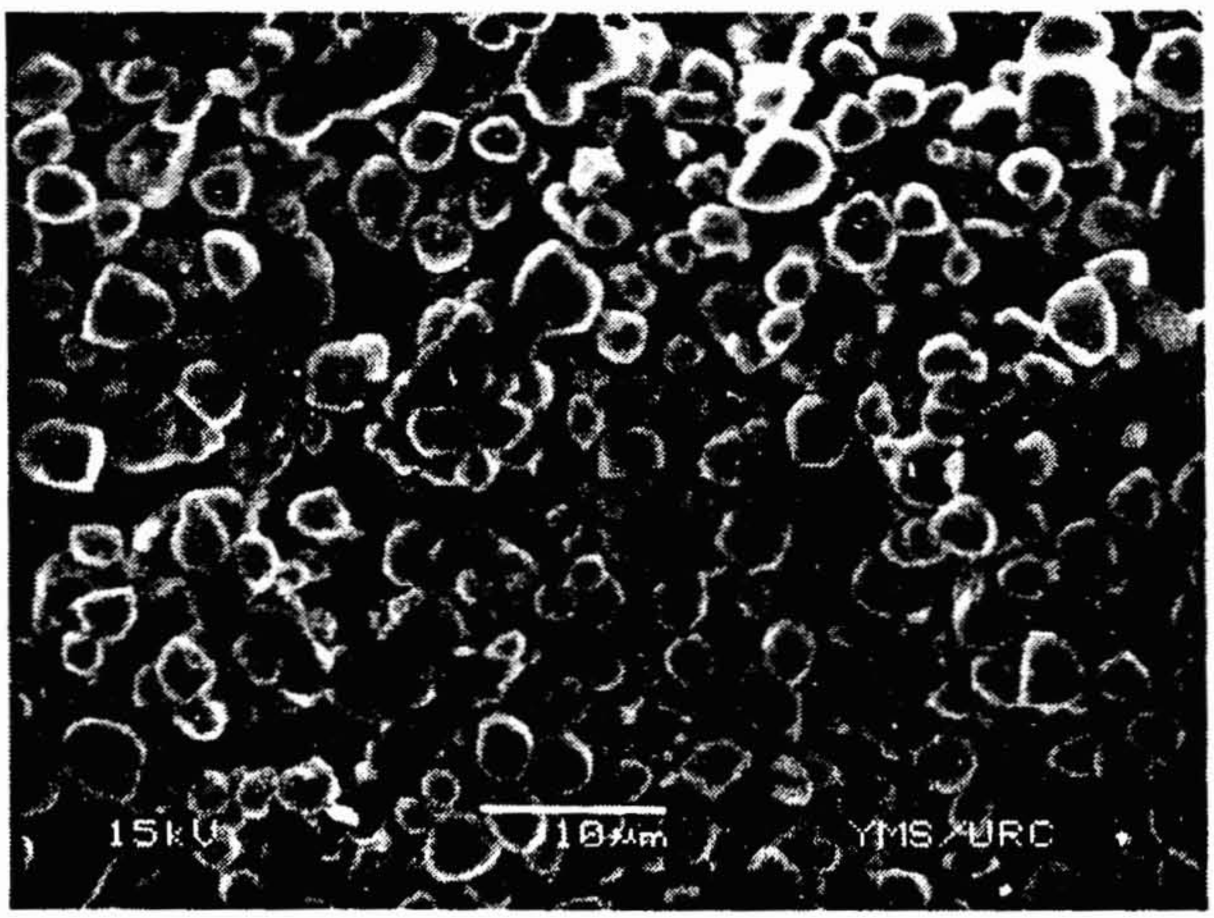

Figure 1. (e) SEM image of $PbTiO_3$ film (process temperature at 700 °C)

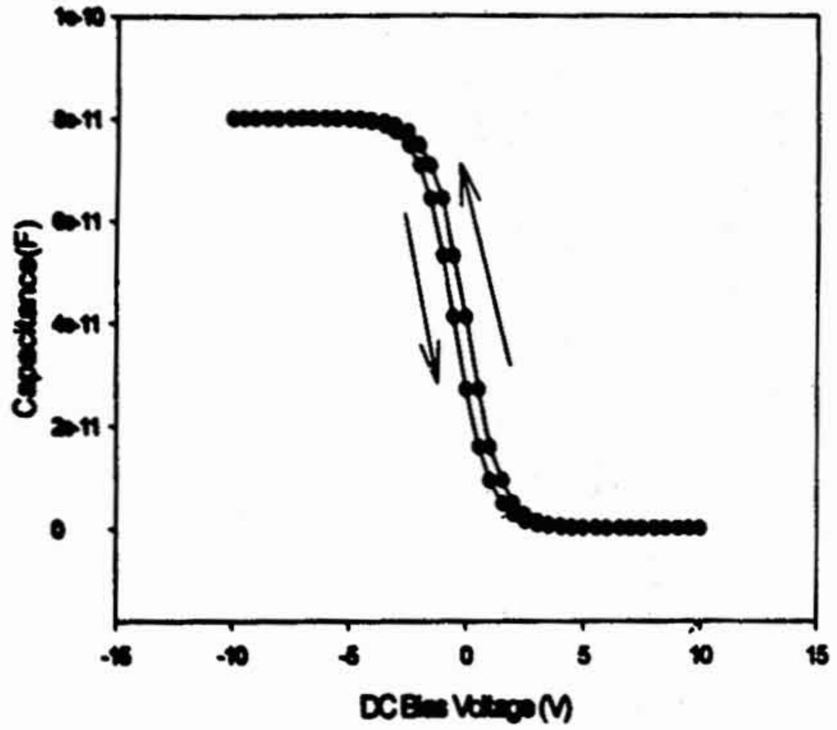

Figure 2 (a) Capacitance-Voltage characteristics of $PbTiO_3$ thin film (process temperature at 500 °C)

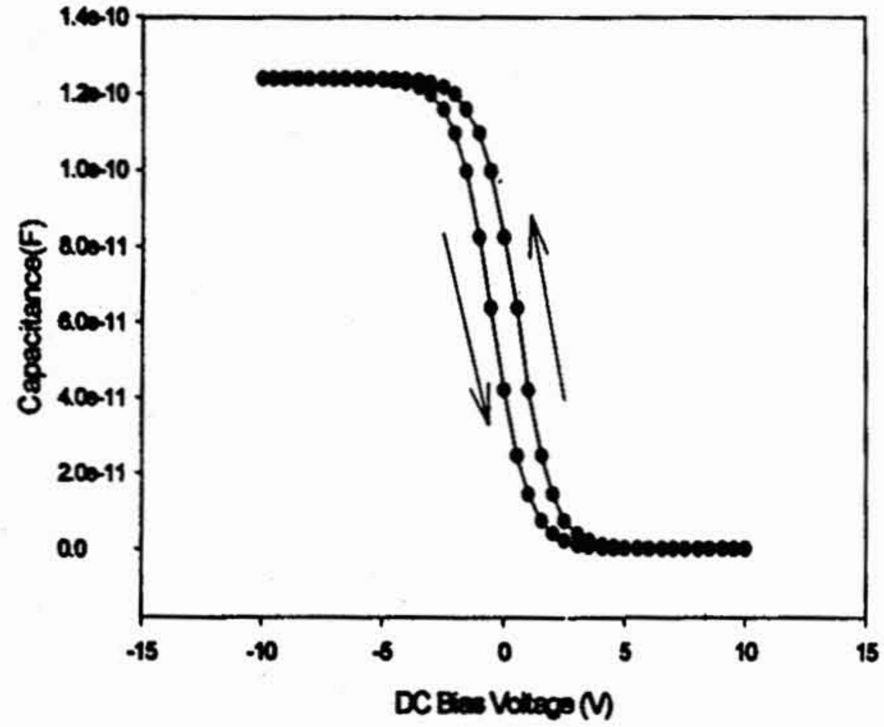

Figure 2 (b) Capacitance-Voltage characteristics of $PbTiO_3$ thin film (processtemperature at 550 °C)

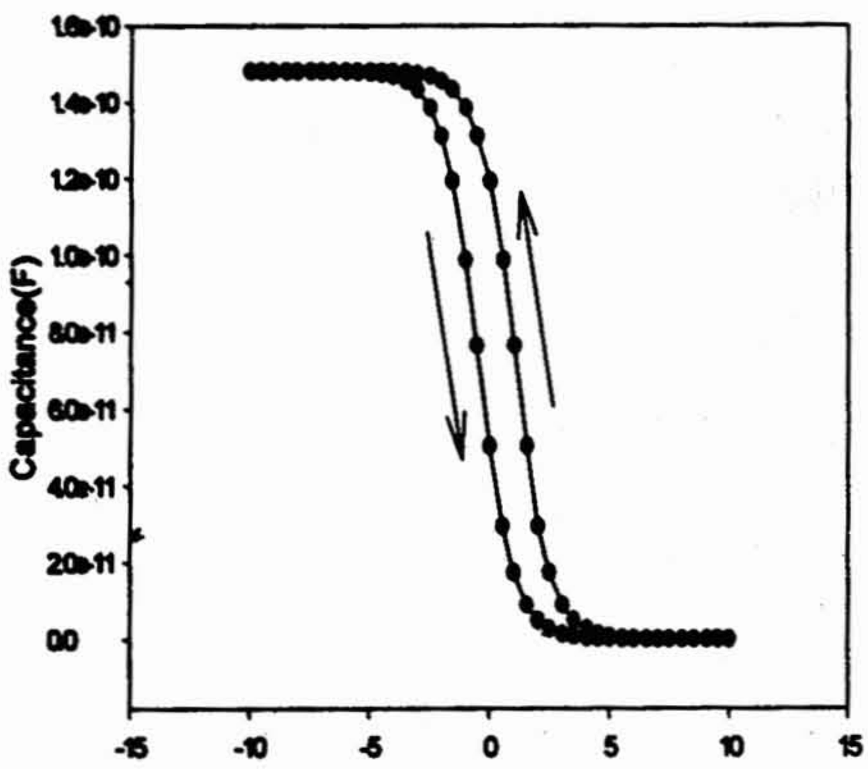

Figure 2. (c) Capacitance-Voltage characteristics of $PbTiO_3$ thin film (process temperature at 600 °C)

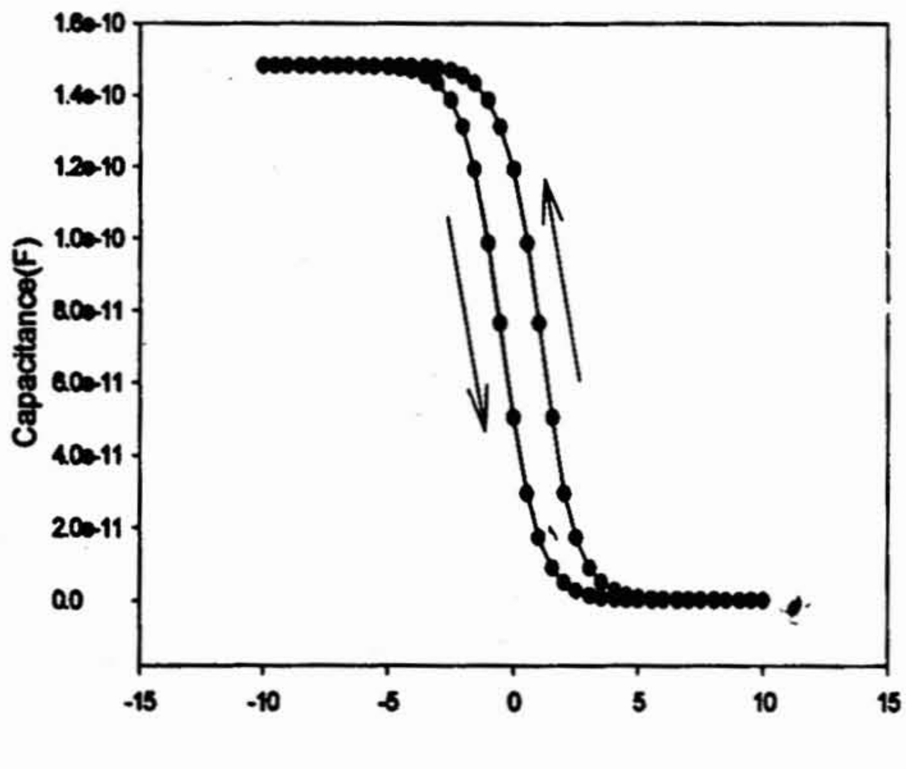

Figure 2. (d) Capacitance-Voltage characteristics of $PbTiO_3$ thin film (process temperature at 650 °C)

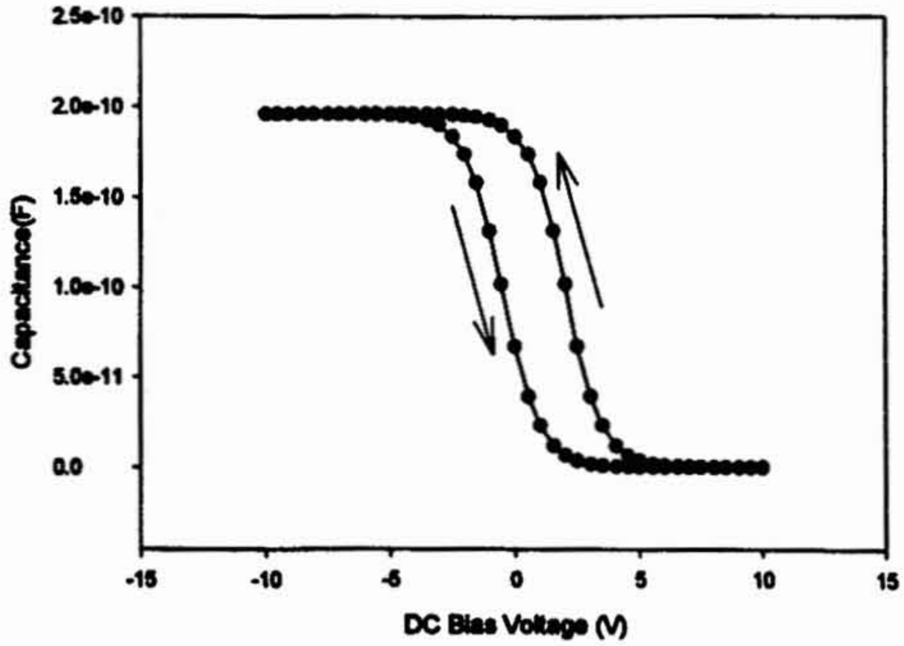

Figure 2. (e) Capacitance-Voltage characteristics of $PbTiO_3$ thin film (process temperature at 700 °C)

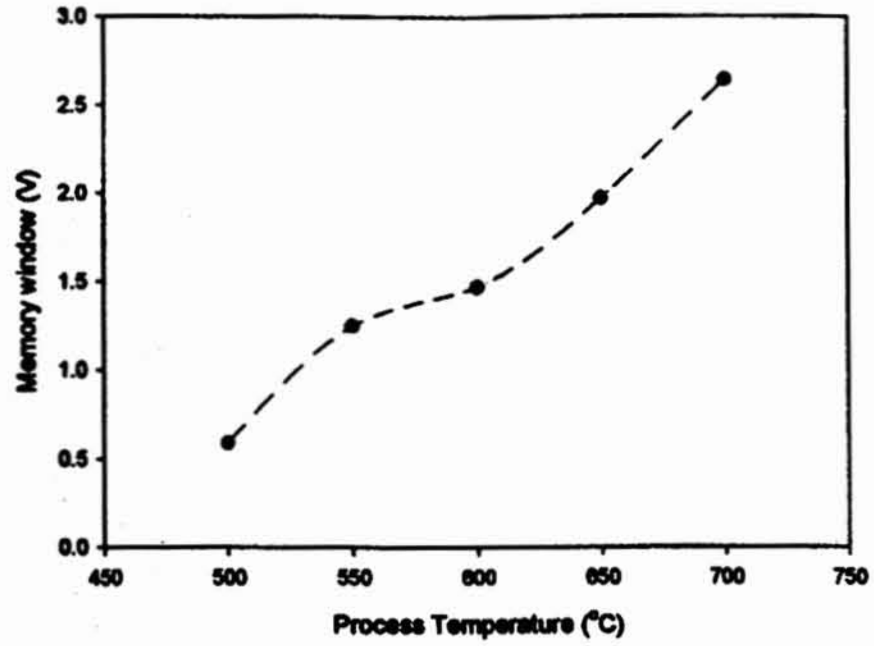

Figure 3. (a) Process temperature dependence of threshold voltage of $PbTiO_3$ thin film

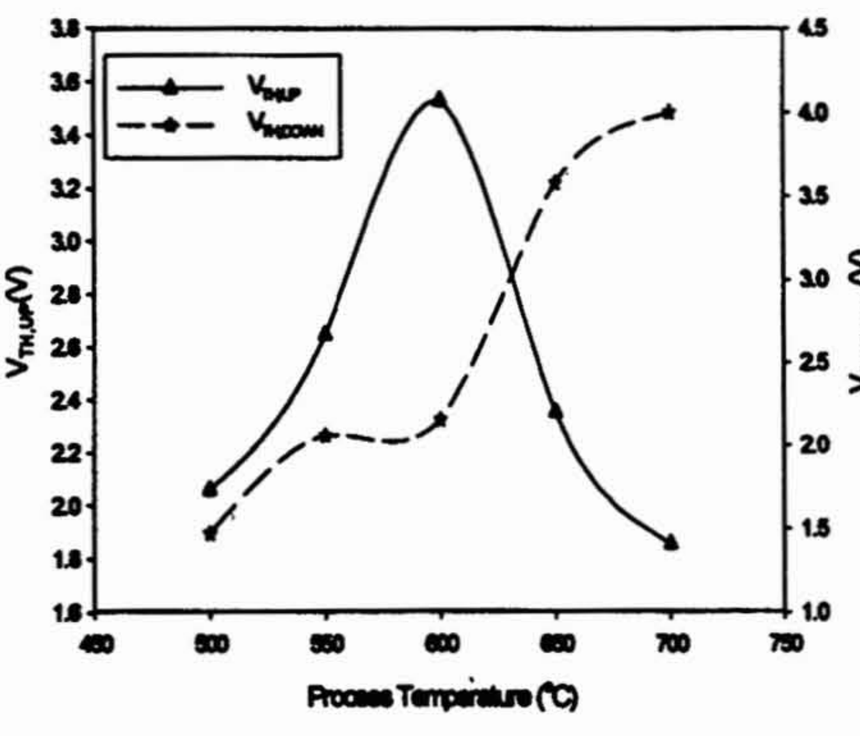

Figure 3. (b) Process temperature dependence of threshold voltage of $PbTiO_3$ thin film

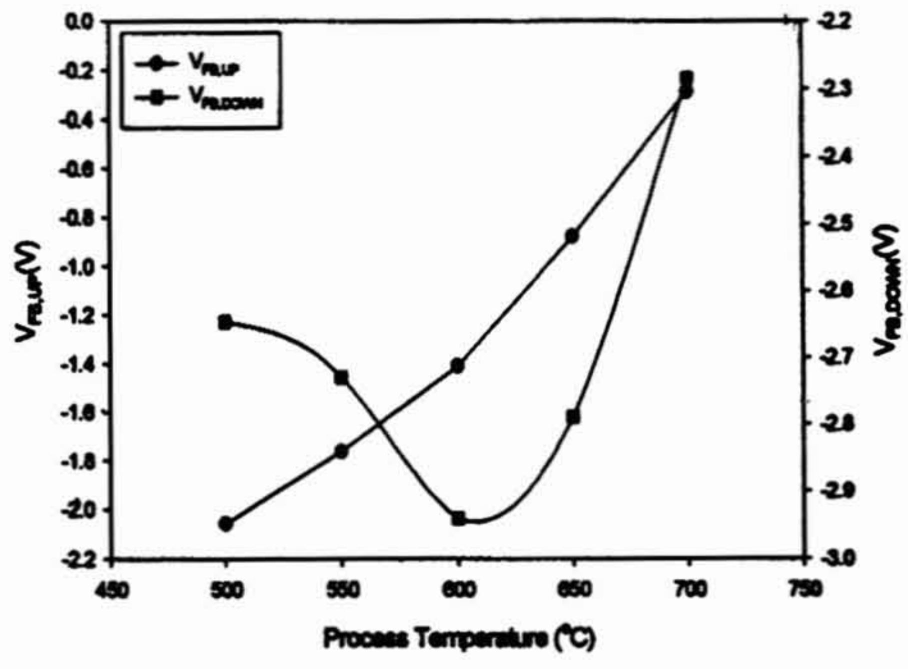

Figure 3. (c) Process temperature dependence of the flatband voltage of $PbTiO_3$ thin film

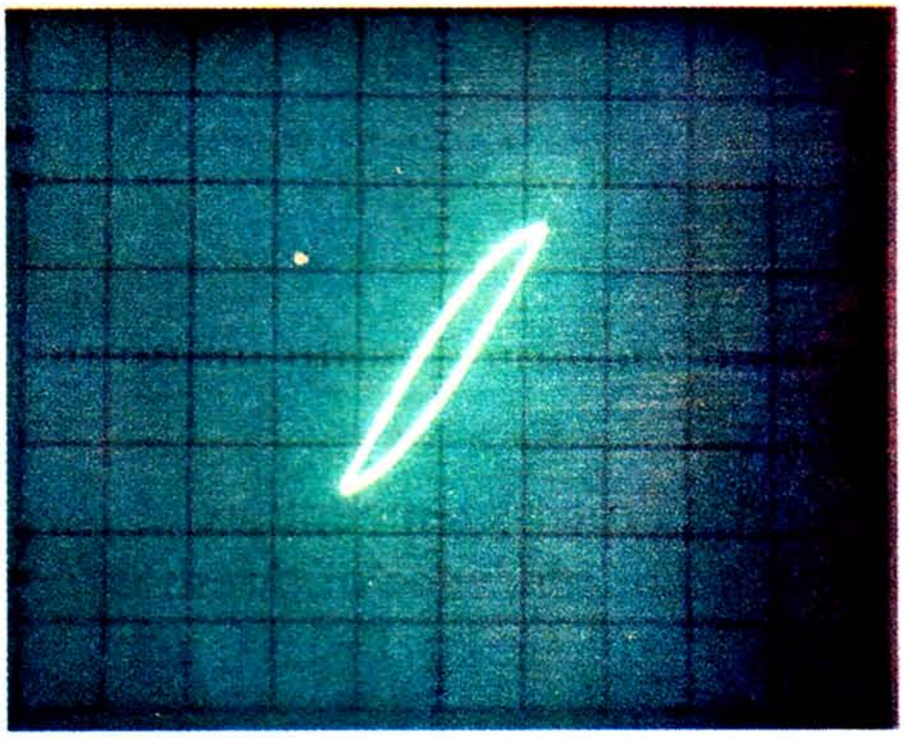

Figure 4. (a) P-E hysteresis loop of $PbTiO_3$ film (process temperature at 500 °C)

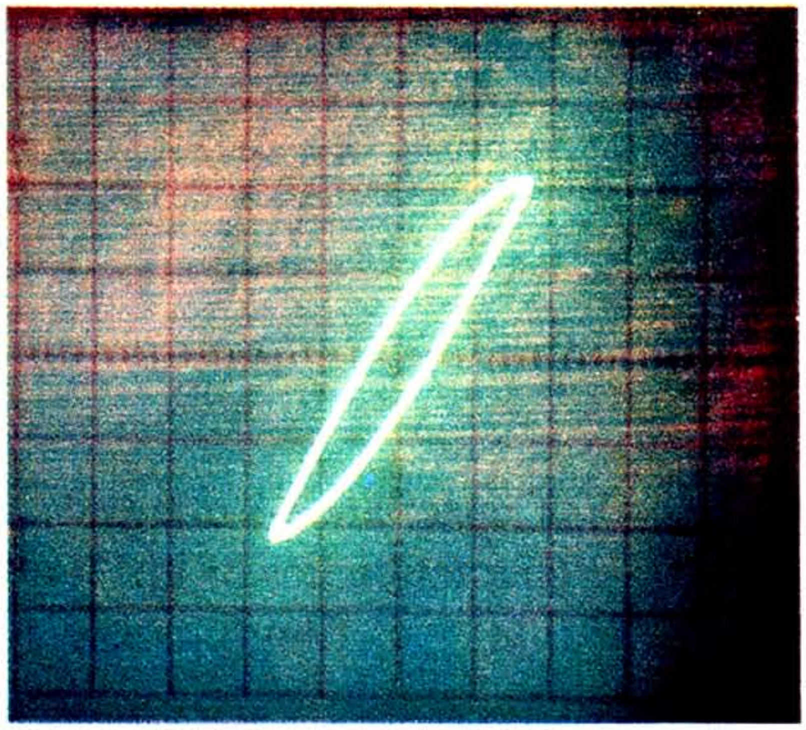

Figure 4. (b) P-E hysteresis loop of $PbTiO_3$ film (process temperature at 550 °C)

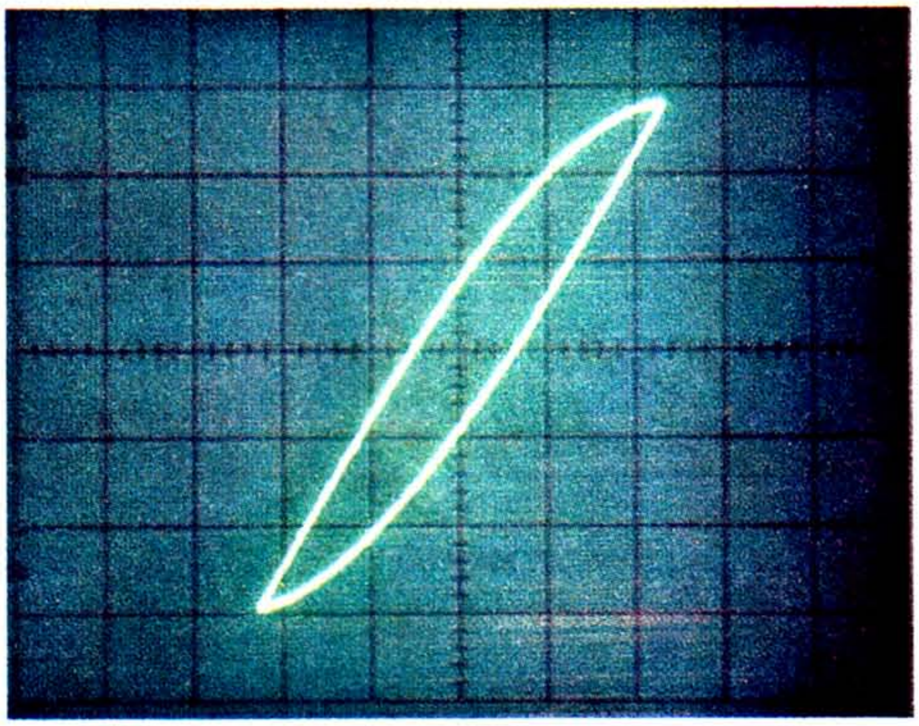

Figure 4. (c) P-E hysteresis loop of $PbTiO_3$ film (process temperature at 600 °C)

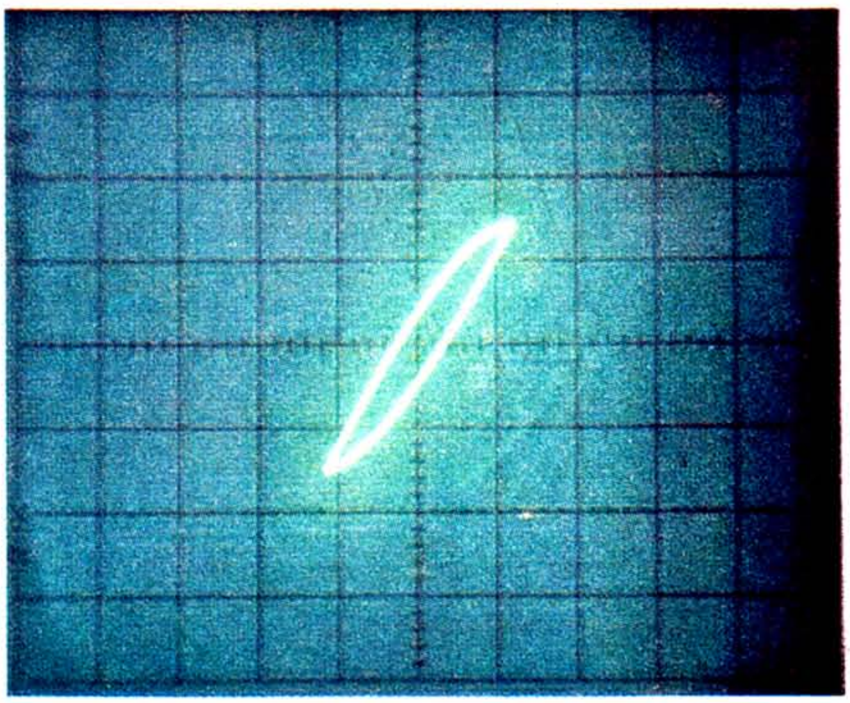

Figure 4. (d) P-E hysteresis loop of $PbTiO_3$ film (process temperature at 650 °C)

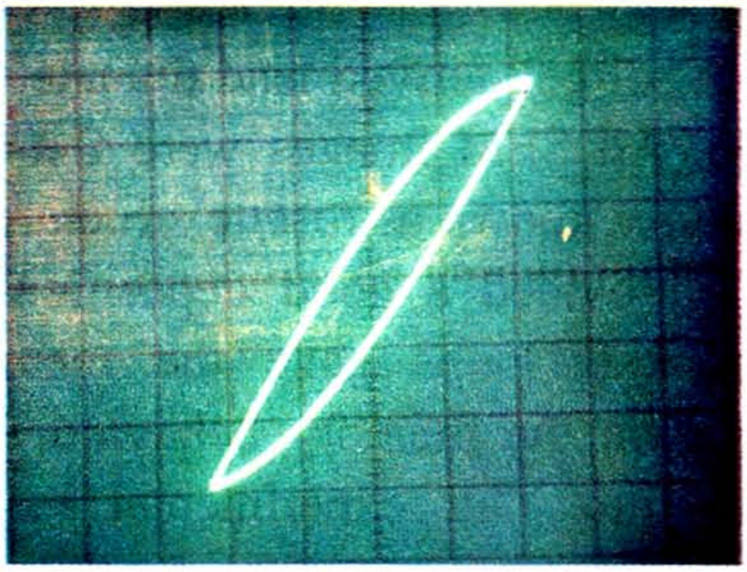

Figure 4. (e) P-E hysteresis loop of $PbTiO_3$ film (process temperature at 700 °C)

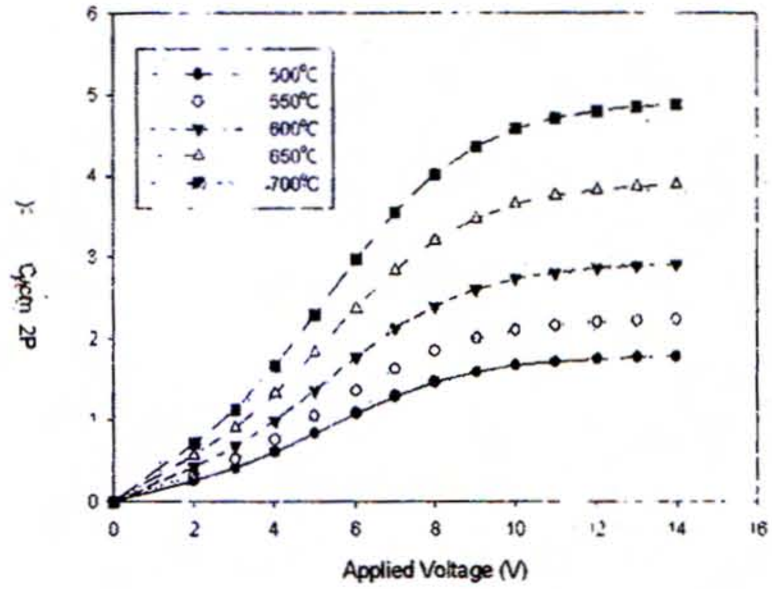

Figure 5. (a) Saturation properties of lead titanate thin film

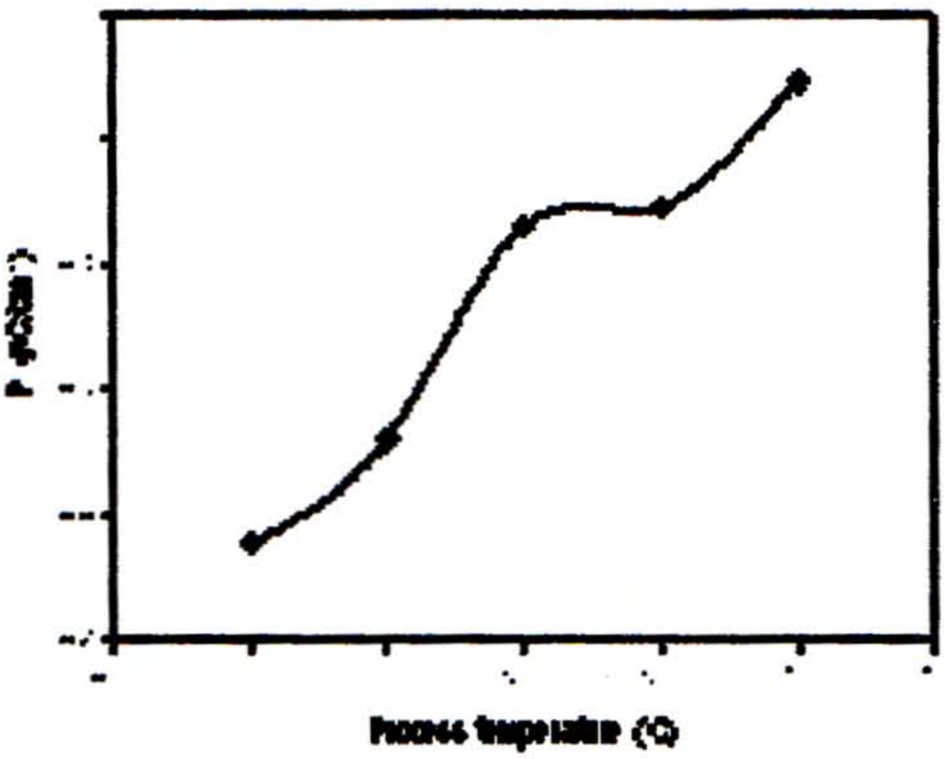

Figure 5. (b) Process temperature dependence of the spontaneous polarization density of $PbTiO_3$ thin film

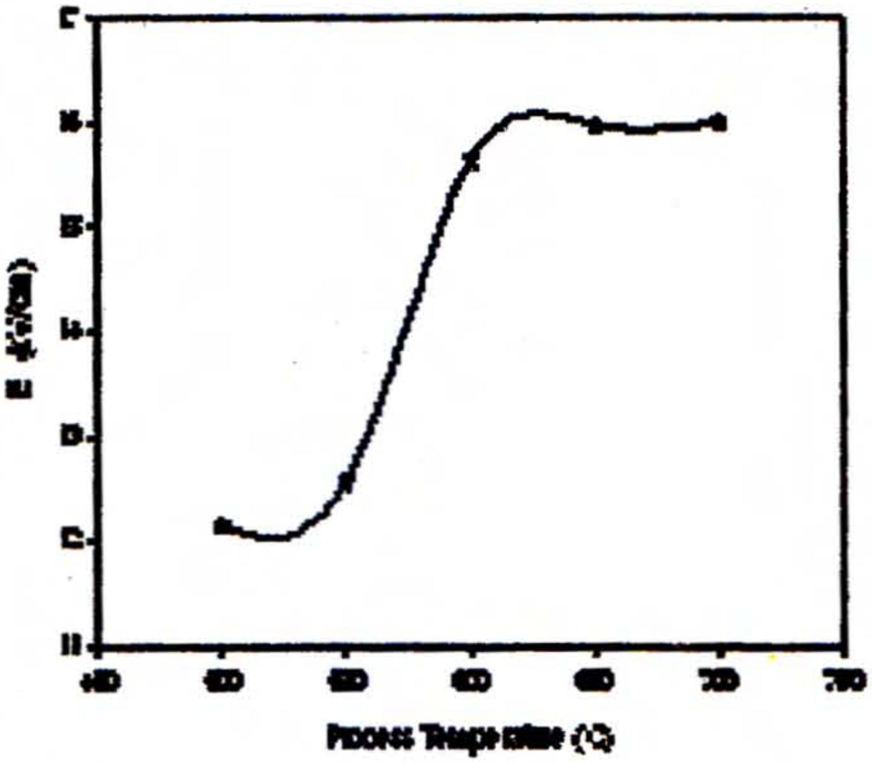

Figure 5. (c) Process temperature dependence of the coercive field of $PbTiO_3$ thin film

Conclusion

Fabrication and characterization of sol-gel derived lead titanate thin film have been successfully investigated. All SEM images were flat and creak-free. They consisted of circular features known as rosette structure. It was well-known that lead titanate film provided a good deposition on substrate. Counter-clockwise C-V hysteresis loops gave the memory function. All P-E hysteresis loops also gave the memory behaviour and ferroelectric properties of lead titanate film. Our experimental results indicated strongly that the sol-gel derived PT film was quite feasible for non-volatile memory device application.

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