6. ELECTRICAL CHARACTERIZATIONS OF PZT THIN FILM PREPARED BY PULSED LASER DEPOSITION (PLD) METHOD

6.1 INTRODUCTION

A variety of thin film deposition techniques such as sputtering [77], sol-gel [78], metalorganic chemical vapor deposition (MOCVD) [79] and pulsed laser deposition [80, 81] have been extensively used to fabricate nanostructured PZT thin film. Each deposition technique has its own advantage and disadvantages and efforts are still necessary to optimize the deposition parameters and conditions for most of them. Among them the PLD technique is the most popular and powerful one in terms of stoichiometric transfer from the multi component oxide target to the growing film and its easy applications of PZT material. Even though several studies [52, 53] have been dedicated to the investigation of the structural, ferroelectric and photoelectric properties of PZT thin film, the studies on the grain size dependent ferroelectric characterization are scarce.

The present study reports on the structural and ferroelectric characterizations of nanostructured PZT thin film coated on Pt/TiO$_2$/SiO$_2$/Si (100) substrate by pulsed laser deposition method with the Zr/Ti ratio of 52/48. The variation of ferroelectric properties with grain size of the thin film have been studied and compared with other experimental results. Also, the results of the structural, ferroelectric and electrical properties have been discussed.

6.2 RESULTS AND DISCUSSION

6.2.1 Structural and morphological studies

Figure. 6.1 shows the XRD pattern of PZT pellet sintered at 1200 °C for 12 hours. Figure. 6.2 shows the XRD pattern of the PZT thin film coated on platinized Si substrate with the substrate temperature of 600 °C. The XRD pattern of the thin film shows the formation of a single phase perovskite structure.
Figure 6.1: XRD pattern of the PZT (52/48) pellet sintered at 1200 °C for 12 hrs.

Figure 6.2: XRD pattern of the PZT (52/48) thin film prepared by pulsed laser deposition method on Pt/TiO\textsubscript{2}/SiO\textsubscript{2}/Si (100) substrate.
The XRD pattern reveals a main peak with high orientation of PZT thin films along (111) planes with two low intense peaks appeared along planes of (101) and (002) and it is shown in Figure. 6.2. Formation of the unwanted pyrochlore phase was eliminated in the film through a careful selection of deposition parameters.

Figure. 6.3: SEM images of the PZT (52/48) thin film coated on Pt/TiO$_2$/SiO$_2$/Si(100) substrate (a) magnified at 10 kV and (b) magnified at 30 kV.

The surface morphology of the PZT thin film taken by SEM is shown in Figure 6.3.
(a) and (b). The SEM images show the well-developed grain structure with dense and uniform distribution. The average grain size of the PZT thin film is about 50-100 nm.

6.2.2 Ferroelectric properties

The ferroelectric hysteresis behavior was studied for PZT bulk as well as thin film deposited by pulsed laser deposition method. Figure 6.4 shows the polarization vs electric field (P–E) hysteresis loop for the PZT pellet with thickness of 0.73 mm. The comparison of remnant polarization and coercive electric field for various applied voltages are shown in Table 6.2.

![P–E hysteresis loop for PZT pellet](image)

Figure. 6.4: Polarization vs Electric field studies of PZT (52/48) pellet for various applied voltages.

From Table 6.2, it is observed that when the applied voltage is increased from 2 kV to 3 kV, the values of $P_s$, $P_r$ and $E_c$ increase by 6.23 µC/cm$^2$, 2.18 µC/cm$^2$ and 3.35 kV/cm respectively. When the applied voltage increased from 3 kV to 3.5 kV, the values of $P_s$, $P_r$ and $E_c$ increase by 10.7 µC/cm$^2$, 3.03 µC/cm$^2$ and 5.12 kV/cm respectively.
Table 6.1: Remnant ($P_r$) and saturation ($P_s$) polarization and coercive field ($E_c$) determined from the measured hysteresis loop for PZT (52/48) ceramics.

<table>
<thead>
<tr>
<th>Applied Voltage (kV)</th>
<th>Saturated Polarization ($\mu$C/cm$^2$)</th>
<th>Remnant Polarization ($\mu$C/cm$^2$)</th>
<th>Coercive field (kV/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>9.56</td>
<td>3.05</td>
<td>5.06</td>
</tr>
<tr>
<td>3</td>
<td>15.79</td>
<td>5.23</td>
<td>8.41</td>
</tr>
<tr>
<td>3.5</td>
<td>26.49</td>
<td>8.46</td>
<td>12.60</td>
</tr>
</tbody>
</table>

Figure 6.5: Polarization vs Electric field loop of the PZT (52/48) thin film.

Figure 6.5 shows the P-E loop for the PZT (52/48) thin film coated on Pt/TiO$_2$/SiO$_2$/Si<100> substrate. It is lower than the values reported by Pandey et al. [53] and Tyunina et al. [82]. Pandey et al. [53] reported values of $P_r$ and $E_c$ as 25
µC/cm² and 44 kV/cm respectively for grain size of about 500 nm. Also, Tyunina et al. [82] reported values of P_r and E_c as 17 µC/cm² and 50 kV/cm respectively for the grain size of about 100 nm. In the present studies, the values of P_r and E_c are 2.09 µC/cm² and 11.98 kV/cm respectively for the grain size of about 50 nm. The present results confirm that the spontaneous polarization, remnant polarization and coercive electric field decrease with decrease in the grain size of the thin film.

6.2.3 I-V characteristic studies

![I-V characteristic curve](image)

Figure. 6.6: I-V characterization of PZT (52/48) thin film.

The Current –Voltage characteristics curve for the PZT thin films is shown in Figure. 6.6. The I-V characteristics was recorded at room temperature after pre-poling the PZT thin film with a suitable dc voltage in order to minimize the influence of the polarization reversal current over the I-V characteristics. The apparent decrease/change in I–V is mainly due to change in the conductivity nature of the electrode. From the Figure 6.6 it is seen that the leakage current of the PZT thin film is minimum (4.31 A/cm² at -1.44 V) with the Pt electrode as negative bias and maximum (2.89 A/cm² at 1.02 V) with the Pt electrode as positive bias. Similar results were obtained by other studies [83,
6.3. CONCLUSION

The nanostructured PZT thin films were prepared using PLD technique on Pt/TiO$_2$/SiO$_2$/Si(100) substrate at the substrate temperature of 600 °C. X-ray diffraction patterns of PZT thin film shows the formation of perovskite crystalline with highly (211) orientation. The SEM image reveals that the film is in good surface microstructure with dense and grain size of 50 -100 nm. Typical P-E hysteresis loop has been observed at room temperature with low applied voltage. The measured values of $P_r$ and $E_c$ are 2.07 $\mu$C/cm$^2$ and 11.98 kV/cm respectively. The present results confirm that the spontaneous polarization, remnant polarization and coercive electric field decrease with decrease in the grain size of the thin film. The results of Current – Voltage relation agree with other similar studies.