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Author: K.K. Maurya S.K. Halder Suchitra Sen Ankita Bose
Sandip Bysakh



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High resolution X-ray and electron microscopy characterization of PZT thin films prepared by RF magnetron sputtering

K.K. Maurya and S.K. Halder

CSIR-National Physical Laboratory, Centre for Nanoscale Science, New Delhi – 110012, India

Suchitra Sen*, Ankita Bose and Sandip Bysakh[#]

CSIR-Central Glass and Ceramic Research Institute, Kolkata- 700032, India,

Abstract

Pb(Zr_{0.52}Ti_{0.48})O₃ (PZT) thin films grown on Pt/TiO₂/SiO₂/Si(100) substrates in the thickness range of ~ (30-550) nm by Radio Frequency (RF) Magnetron sputtering method under different sputtering pressure were studied in the present work. High resolution Grazing Incidence X-ray diffraction method for residual stress and Electron Microscopy, particularly Cross Sectional Transmission Electron Microscopy (XTEM), were used to identify the crystalline phases, and structure of the thin films at the nano-scale. Microstructure of the ultrathin films in <50 nm thickness range, consists predominantly of para-electric, oxygen deficient pyrochlore phase of Pb₂Ti₂O₆ structure co-existing with ferroelectric perovskite PZT phase in tetragonal form. In films with thickness around 500 nm, sputtering pressure shows a strong influence on the purity of PZT phase grown. High resolution X-ray diffraction method for wafer curvature measurement and Stoney's equation were used to evaluate the biaxial stress in the films. It is found that the ultrathin films are compressively stressed with high magnitude of the order of 2 GPa which gets reduced with increasing film thickness. In the thickness range of ~500 nm, at an optimum sputtering pressure of 4.5 Pa, the stress becomes tensile in nature with a small magnitude of 2.3 MPa. At this pressure, the film consists of almost pure perovskite phase and comparatively better electrical characteristics. X-Ray Reflectometry study indicates very low density of PZT films with an interlayer formed at the interface with Pt. XTEM study throws valuable insight into the nano-scale structure and reveals presence of nano-porosity along the interface as well as within the film microstructure. This has been attributed to the observed interface roughness, reduced density, tensile residual stress as well as the poor ferroelectric properties encountered.

Keywords:

PZT, R F Sputtering, Perovskite phase, Residual stress, X-ray Reflectometry, Cross Sectional Transmission Electron Microscopy

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