IN-SITU PHASE AND TEXTURE CHARACTERIZATION OF SOLUTION DEPOSITED PZT THIN FILMS DURING CRYSTALLIZATION

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Ferroelectric lead zirconate titanate (PZT) thin films are used for integrated capacitors, ferroelectric memory, and piezoelectric actuators. Solution deposition is routinely used to fabricate these thin films. During the solution deposition process, the precursor solutions are spin coated onto the substrate and then pyrolyzed to form an amorphous film. The amorphous film is then heated at a higher temperature (650° - 700°C) to crystallize the film into the desired perovskite phase. Phase purity is critical in achieving high ferroelectric properties. Moreover, due to the anisotropy in the structure and properties of PZT, it is desirable to control the texture obtained in these thin films. The heating rate during crystallization process is known to affect the sequence of phase evolution and texture obtained in these thin films. However, to date, a comprehensive understanding of how phase and texture evolution takes place is still lacking.

To understand the effects of heating rate on phase and texture evolution, in-situ diffraction experiments during the crystallization of solution deposited PZT thin films were carried out at beamline 6-ID-B, Advanced Photon Source (APS). The high X-ray flux coupled with the sophisticated detectors available at the APS synchrotron source allow for in-situ characterization of phase and texture evolution at the high ramp rates that are commonly used during processing of PZT thin films. A PZT solution of nominal composition 52/48 (Zr/Ti) was spin coated onto a platinum-coated Si substrate (Pt//TiO_x//SiO_2//Si). The films were crystallized using an infrared lamp, similar to a rapid thermal annealing furnace. The ramp rate was adjusted by controlling the voltage applied to the infrared lamp and increasing the voltage by a constant step with every acquisition. Four different ramp rates, ranging from ~1000°C/s to ~1°C/s, were investigated. The sample was aligned in grazing incidence to maximize the signal from the thin films. Successive diffraction patterns were acquired with a 1s acquisition time using a MAR SX-165 CCD detector during crystallization. The sample to detector distance and the tilt rotations of the detector were determined in Fit2D© by using Al_2O_3 as the calibrant. These corrections were applied to the patterns when binning the data into radial (2θ) and azimuthal bins. The texture observed in the thin film was qualitatively analyzed by fitting the intensity peaks along the azimuthal direction with a gaussian profile function to obtain the integrated intensity of the peaks. Data analysis and peak fitting was done using the curve fitting toolbox in MATLAB©.

A fluorite-type phase was observed to form before the perovskite phase for all ramp rates. Pt_xPb is a transient intermetallic formed due to the interaction of the thin film and the bottom electrode during crystallization. Ramp rate was observed to significantly affect the amount of Pt_xPb observed in the thin films during crystallization. Ramp rate was also observed to affect the final texture obtained in the thin films. These results will be discussed in the poster in view of the current understanding of these materials.