A polycapillary based method of monochromatic time-resolved x-ray reflectivity

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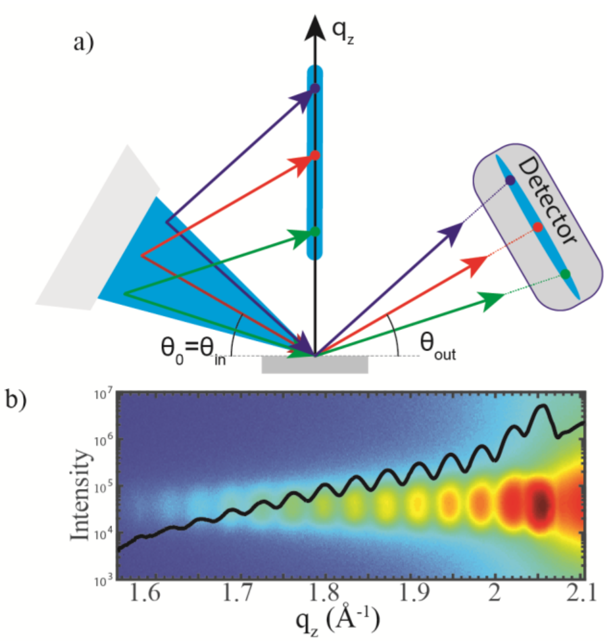
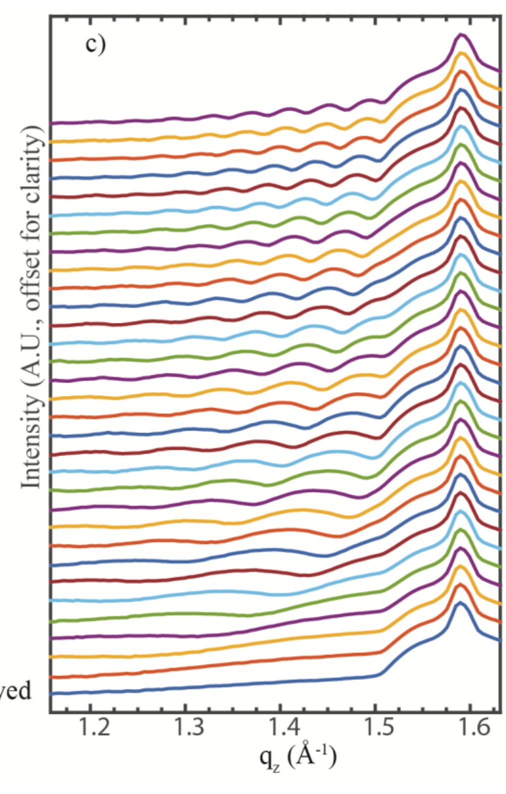
Here we present on the development of a new technique for simultaneous collection of x-ray reflectivity (XRR) curves using monochromatic radiation. Our method utilizes a polycapillary x-ray optic to create a nominally-planar converging fan of radiation incident on a sample surface, providing a range of incident angles. An area detector is used to simultaneously record diffracted intensity over a range of exit angles equal to that of the incident fan. For a sufficiently smooth sample the specular reflection dominates the diffracted signal for each exit angle. In this manner we can collect XRR spectra without the need for moving the sample or detector, limiting time resolution to the available flux and detector speed.

At the Cornell High Energy Synchrotron Source (CHESS), using this technique, we have demonstrated the ability to record XRR curves covering ~5° in 2θ at with a time resolution of 100 ms. We have recorded changes in Kiessig fringe spacing during the growth of epitaxial $La_{.7}Sr_{.3}MnO_3$ by pulsed laser deposition (PLD) as well as monitored the integrated area, width, and peak position of an oxygen vacancy superstructure peak as the film transformed from the perovskite to the Brownmillerite phase. The technique makes efficient use of the high intensity, ribbon-shaped monochromatic beam produced by an undulator through the use of a half-focusing optic. Additionally, this technique should be well suited to performing XRR using a lab source through the use of a full-focusing optic. The long working distances that are achievable, 22 cm in our case, allow for easy integration with a variety of in-situ process chambers; larger angular ranges can be achieved if sample geometry allows for a shorter working distance.

We discuss the factors determining the range and instrumental resolution of the technique in reciprocal space, in addition to the signal-to-background ratio. The particular setup we utilize covers a $\vec{q}_z$-range of 0.45 Å^{-1} at 11.4 keV with a capillary transmission efficiency of 42%. Our measurements have an instrumental resolution of 6×10^{-3} Å^{-1} in the $\vec{q}_z$-range of interest, and we show Kiessig fringes from samples with layer thicknesses ranging from 3 to 57 nm.





a) The geometery of our technique showing an angular dispersive geometry b) a map of the diffracted intensity on the detector overlayed with the measured reflectivity c) XRR paterns recorded during the growth of La.7Sr.3MnO3 by PLD, captured in 100 ms each