ESRF	**Experiment title:** **In-situ GIXD Studies of nucleation and growth of chemically deposited PbS and PbSe epitaxial films on GaAs**	**Experiment number**: MA335
Beamline: ID1	**Date of experiment**: from: 03.10.07 to: 09.10.07	**Date of report**: 25.04.08
Shifts: 15	**Local contact(s):** T. Metzger and A. Malachias	*Received at ESRF:*
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The morphological and structural evolution in the very early stages of nucleation and growth of ultrathin PbS films grown from solution were studied *in-situ* using GIXD as a function of experimental conditions. We were able, for the first time, to monitor the development of texture and in-plane orientation *in-situ* for epitaxial monocrystalline films chemically deposited from solution. Our previous studies revealed that PbS has a tendency to develop with a [110] orientation on the GaAs(100) substrates. Figure 1 shows the GIXD rocking curve of the $(220)_{PbS}$ Bragg reflection obtained from thin epitaxial PbS film deposited on GaAs(100) from an alkaline solution containing $Pb(NO_3)_2$, $CS(NH_2)_2$ and NaOH at 30°C for various periods of time.

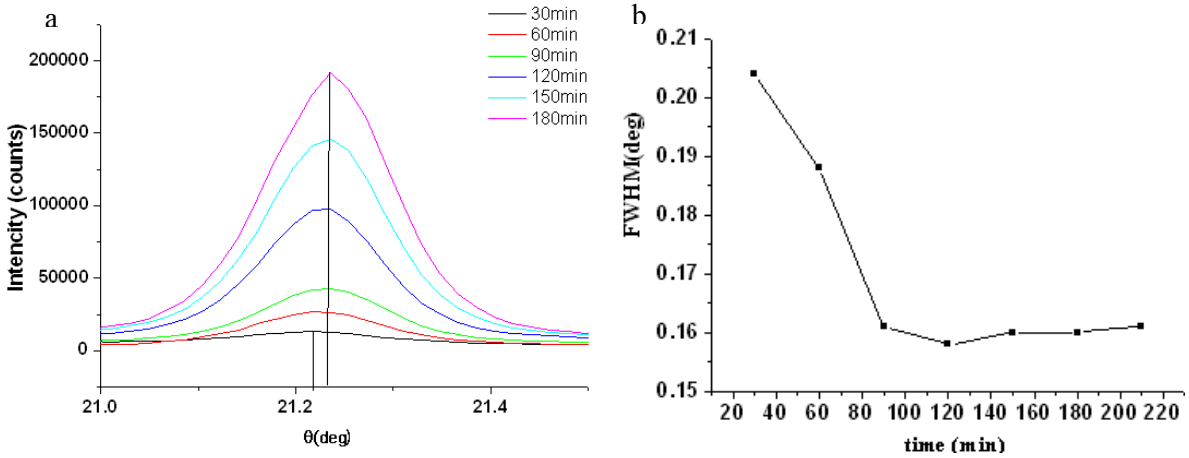

Figure 1: (a) GIXD rocking curve of $(220)_{PbS}$ on GaAs(100) deposited at 30°C for 30-180 min. (b) FWHM of $(220)_{PbS}$ peak at various deposition times.

The graphs indicate decrease in FWHM of the $(220)_{PbS}$ (Fig. 1b) with increasing deposition time that was accompanied by increasing film thickness. This indicates that the film quality is improving with thickness (i.e., with deposition time), strongly confirming our previously obtained in-house observations. In addition, slight shift of the $(220)_{PbS}$ peak position towards $(220)_{GaAs}$ were observed. However, in films deposited for longer than 180 min, a sharp decrease in the $(220)_{PbS}$ intensity was obtained (not shown). This could be either due to dissolution of the film as the solution reaches equilibrium or (more likely) may be due to residual stresses above a critical thickness which result in peeling of the films from the substrate surface. In addition, GIXD radial θ/2θ scan at the vicinity of $(200)_{PbS}$ and $(111)_{PbS}$ for samples deposited on GaAs(100) at 30°C for 30-180 min showed small amounts of [200] and [111] oriented material within the film, indicating that the [110] orientation of the films is by no means perfect and there is a small number of grains oriented in other crystallographic directions. This could be explained by the SEM observations of the beam-sample interaction region as shown below.

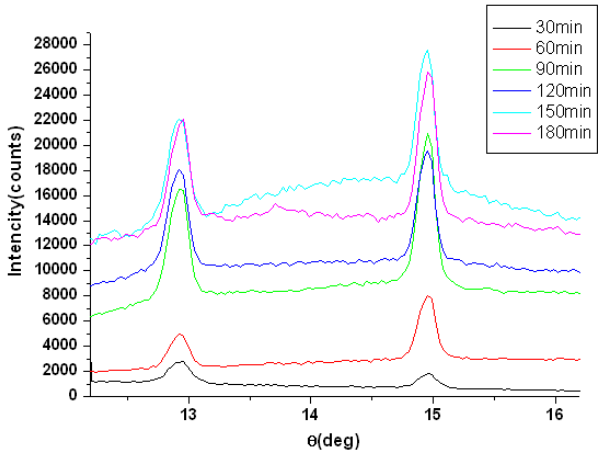

Figure 2: GIXD radial θ/2θ scan at the vicinity of $(200)_{PbS}$ and $(111)_{PbS}$ for sample deposited on GaAs(100) at 30°C for 30-180min.

Interestingly, in-house SEM examination of the samples surfaces after the synchrotron experiments revealed that the high brilliance synchrotron beam might have had some influence on the morphology evolution. The width of the region sampled by the beam was estimated to be 150 μm. The morphology of the film within the area of the beam interaction with the sample differed markedly from the morphology far from the beam interaction area as could be seen in Fig. 3a.

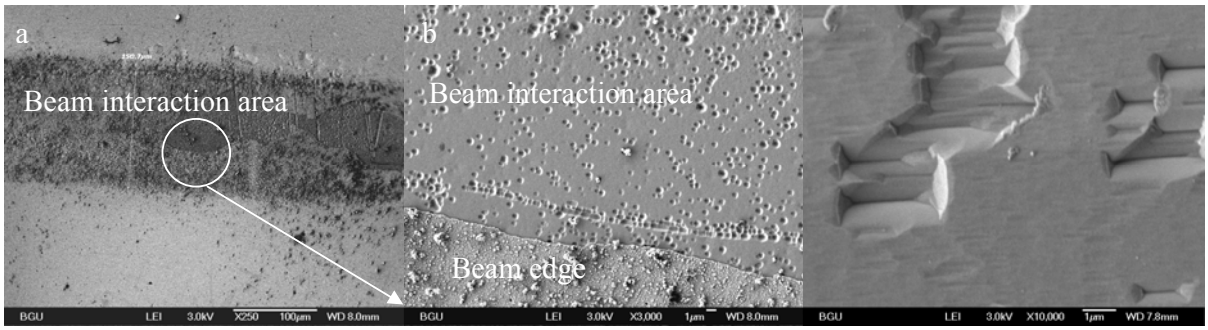

Figure 3: SEM images of PbS film deposited on GaAs(111) at 30°C for 6 hr (a) indicating the beam-sample interaction area, (b) Magnified view showing the edge of the beam-sample interaction zone (c) The film surface far from the beam-sample interaction zone.

The topography of the film surface within the beam interaction area seems to be much rougher than that far from the beam interaction area, and that there is big amount of disoriented aggregates at the outskirts of the beam-sample interaction region (marked as "beam edge" in Fig. 3b). In addition some scrapings were observed (Fig 3a). A magnified view of the "beam edge" region is shown in Fig. 3b. SEM micrograph of the film surface far from the beam interaction area revealed a typical <110> oriented monocrystalline topography as shown in Fig 3c. This is in fact not surprising since the deposition conditions were optimized in-house (in the absence of a high brilliance x-ray beam), while the in-situ experiments were strongly perturbed by the energy provided by the high brilliance x-ray beam at ID-01. This suggests that lower deposition temperatures should be used in future *in-situ* synchrotron experiments in order to work closer to optimal deposition conditions.