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Measurement of Crystal Symmetry using HARECXS

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Convergent Beam Electron Diffraction (CBED) has long been used to measure the symmetry of crystalline materials because of its sensitivity to changes in structure. By quantitative comparison of calculated CBED patterns with experimental data it is possible to determine crystal symmetry and atomic site occupation [1]. In a similar manner, electron channeling induced x-ray emission can be used to study crystal site occupancy[2,3]. In this work we have used high angular resolution of electron channeling x-ray spectroscopy (HARECXS) in a zone axis orientation to demonstrate that crystal symmetry can be directly observed as a compliment to the CBED technique.

The sample chozen for this work was a single crystal specimen of GaAs whose projected crystal structure in the {100}, {111}, and {110} planes is illustrated in Figure 1. It can be seen that in the {100} {111} zones of the crystal structure both Ga and As are symmetrically distributed, however in the {110} zone there is a noticeable asymmetry in the structure, this break in symmetry along one direction of the lattice should be directly measurable using HARECXS [4].

HARECXS measurements were carried out on a FEI Tecnai F20, equipped with an EDAX SUTW Si(Li) detector operated at an incident beam energy of 200 kV. A single crystal sample of GaAs was cleaved and ion milled and then was plasma cleaned in an Ar plasma for 15 minutes in a South Bay Technology Plasma cleaner. The latter, process being done to mitigate the effects of carbon contamination during temporally extended measurement of ~27 hours which was done to achieve statistically significant data. All measurements for this work, were carried out with the incident probe was adjusted to achieve parallel illumination conditions using the condenser-objective lens, and employing a ~ 50 micron C2 aperture to define a region of interest on the sample of approximately 100nm in diameter. After carefully adjusting the beam tilt pivot points of the instrument, a custom computer program was used to accurately tilt the incident probe in a 2D raster pattern over the fixed region of interest on the specimen, while simultaneously recording an X-ray spectrum at each location of the 133 x 75 point matrix. At normal incidence the XEDS count rate was ~ 1.2 Kcps while the nominal thickness of the area of interest was ~ 400 nm.

Figure 2 presents the results of measurements of the Ga Kα and As Kα x-ray emission lines as a function of orientation. One can immediately observe in these data sets the asymmetric nature of the HARECXS intensity distribution for both Ga and As respectively. In this data one can readily observer the overall asymmetry of the two atomic species, in addition, we note the fine details in the individual channeling maps differ markedly. Work is currently in progress to compare these experimental results with calculations.

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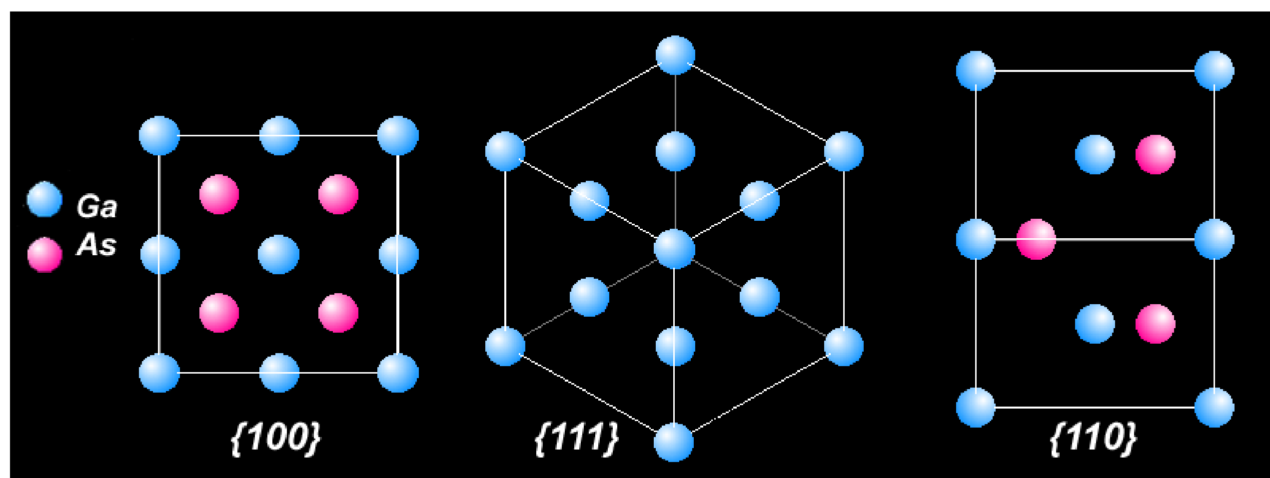

Figure 1. Projected structure of GaAs as viewed along the {100}. {111} and {110} planes.

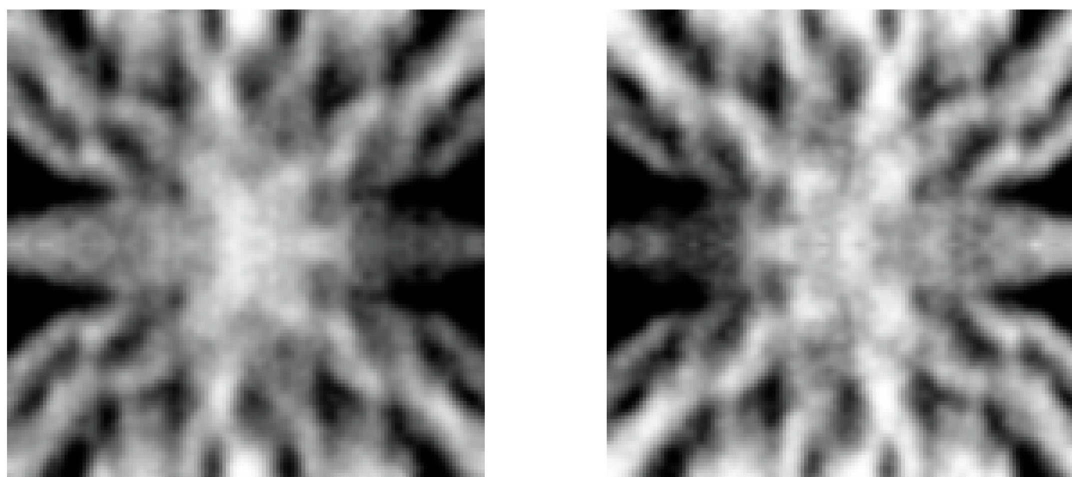

Figure 2. Two Dimensional HARECXS Intensity maps for the {110} zones of GaAs. for the Ga Kα (Left) and As Kα (Right) emission lines.

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