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A comparison of the analysis of non-centrosymmetric materials based on ion and electron beams

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ABSTRACT

Techniques like electron backscattered diffraction (EBSD) and angle-resolved electron Rutherford backscattering (ERBS) are sensitive to the lack of inversion symmetry in crystals and hence can determine the absolute crystal orientation. In this paper we demonstrate for the case of GaP that medium energy ion scattering (MEIS) can also be used to obtain the absolute crystal orientation. A comparison between the 2D-backscattering intensities as a function of the detection directions for electrons (as is measured in EBSD or ERBS) and protons (as is measured in MEIS) is made and discussed in terms of diffraction and classical subchanneling respectively.

1. Introduction

In contrast to centrosymmetric semiconductors with the diamond structure (point group $m\bar{3}m$), the $\langle 001 \rangle$ axes cease to be proper 4-fold rotation axes for binary semiconductors like GaAs and GaP crystallizing in the cubic zincblende structure (point group $\bar{4}3m$). In the zincblende structure, a rotation over 90° around $\langle 001 \rangle$ is equivalent to an inversion operation, which can effectively interchange the relative position of the cations and anions.

An important consequence of the symmetry reduction in the zincblende as compared to the diamond structure is relevant, for example, when films of III-V compounds are grown on a Si(001) surface [1]. In this case, domains of two inequivalent crystal orientations related by an inversion operation can be formed in the overlayer, and defects will be found at the anti-phase domain boundaries. It is thus important to be able to characterize the absolute orientation of crystals experimentally, which requires a characterization method that is sensitive to the absence of a center of symmetry in the crystal structure.

For imaging applications in the scanning electron microscope (SEM), it was shown recently that electron backscatter diffraction (EBSD) can be used to map the distribution of anti-phase domains in thin GaP films [2]. Dynamical electron diffraction effects in the Kikuchi diffraction patterns which are observed in an EBSD measurement can provide sensitivity to non-centrosymmetric effects of the crystal structure. By correlating experimentally observed two-dimensional Kikuchi diffraction patterns with simulations using the dynamical theory of electron diffraction, the local absolute crystal orientation can be assigned to the measured sample region.

Moreover, in Electron Rutherford Backscattering (ERBS) experiments it was shown that, if one separates the contribution of the anion and cation based on their different recoils losses, the effect of the orientation has the opposite sign for scattering from the cation and anion [3]. As the strength of both signals depend on their atomic number Z, a small fraction of this asymmetry survives if one takes the sum of both intensities, as is done in conventional EBSD. This small difference is enough, however, to determine completely the overlayer orientation.

Ion channeling is a real-space technique that can probe the crystal structure. Here recoil effects are much larger (due to the smaller mismatch of the mass of projectile and target atom) and separation of the heavy and light atoms is more routinely obtained. Can ion-beam techniques be an alternative to EBSD for the determination of the overlayer orientation and what is the nature of the difference under these conditions? Here we explore this question using medium energy ion scattering (MEIS) with an electrostatic analyzer. Then it is possible to obtain two-dimensional intensity distributions, just as in EBSD, but only for the contribution of the heavier element. To be specific we studied a bulk GaP crystal using 100 keV protons as a projectile and focus on the deviations of the observed intensity, when the crystal is rotated by 90° along the [100] axis, i.e. if it is possible to determine the complete crystal orientation.

Asymmetries measured by ion channeling have been reported before for non-centrosymmetric crystals [4–6] for angular scans. Here we

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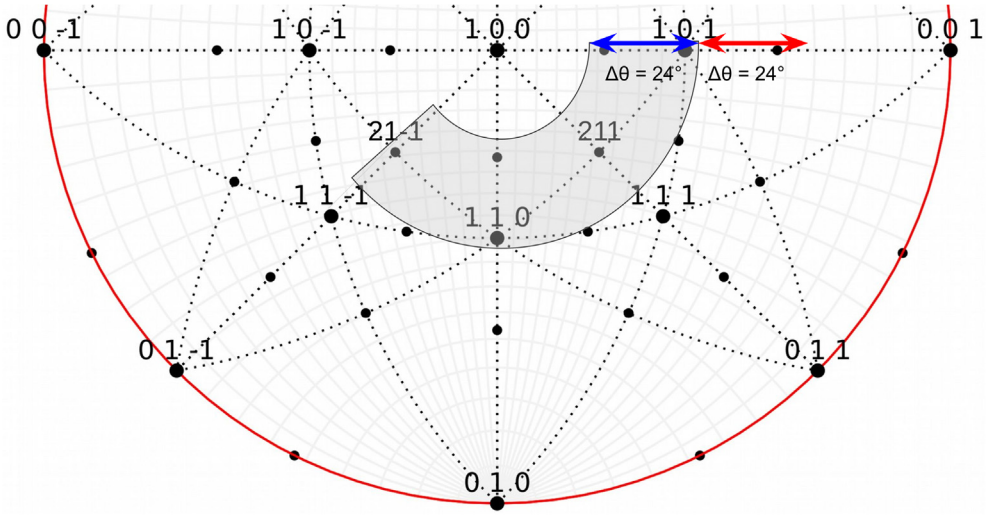

Fig. 1. Stereographic projection of the GaP crystal on the [100] direction. The shaded area corresponds to the region where the experimental cartography was performed. The red and blue arrows show the polar angle regions for a normal incidence beam and for a 25° tilt in the sample, respectively [9]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

measure complete two-dimensional intensity distributions over a range of (θ, ϕ) angles.

2. Experimental procedure

The MEIS measurements were performed at the Ion Implantation Laboratory of the Federal University of Rio Grande do Sul. A 500 keV electrostatic accelerator provided an incident beam of H^+ with an energy of 99 keV. The GaP crystal was mounted on a 3-axis goniometer that allowed us to perform measurements with the outgoing trajectories along different polar and azimuthal angles. The pressure in the analysis chamber was about 10^{-7} mbar. Typical beam currents were less than 15 nA. The backscattered H^+ ions were analyzed with a Toroidal Electrostatic Analyzer (TEA). At the exit plane of the TEA a pair of micro-channel plates coupled to a position-sensitive detector was used to measure the scattering energy and angle for the detected ions [7,8]. The analyzer is mounted at 120° with respect to the incident beam. The TEA angular aperture is 24° and each angle bin corresponds to 0.08°. These configuration allows the analysis in a polar range from $\theta = 48°$ to 72°, as shown by red arrows in the Fig. 1. In order to perform the analysis in a different polar range we rotated the sample towards the detector by 25°. Then a polar range between 23 and 47° is measured, as shown by the blue arrows in Fig. 1. The overall energy resolution of the system is 450 eV for 99 keV H^+ ions.

Typical maps of ion scattering intensities as a function of the detected energies and scattering angles (the so-called 2D MEIS spectra) for 99 keV H^+ ions impinging on GaP crystal are shown in Fig. 2.

Blocking lines (reduced intensity at certain scattering angles) are evident in the top panel. Corresponding PowerMeis [10] simulations for an amorphous GaP are shown on the bottom panel. The region 1 corresponds to the surface peak of Ga and the region 2 to the surface peak of *P* superimposed to the Ga signal. The contributions from H^+ backscattering from Ga and from the combination of Ga and P are easily distinguished and were used to select the energy window for the cartography. In the energy range indicated as region 1 the H^+ was backscattered from Ga near the surface.

The difference between the experiment (top panel) and simulation in region 1 is due to channeling and blocking effects. In this experiment the incoming beam was along the surface normal, i.e. the ⟨100⟩ directions and the scattering yield away from the surface was reduced by the channeling effect (at all scattering angles). In addition, blocking causes the reduction of the ion scattering intensity for certain scattering angles (evident as vertical lines with reduced intensity). Since the simulation is for an amorphous GaP sample the ion scattering intensity is only affected by a slow decrease in the scattering cross section with increasing polar angle. The onset of the P contribution to the spectrum occurs several keV below the onset of the Ga contribution. The change of the onset with the scattering angle is described by the kinematic factors of Ga and P. If one adds add up measurements taken at different azimuthal angles (here varied from 0° to 90°, with a step of 5°), then the blocking effect are averaged out, as shown in Fig. 2 (middle panel). However, this procedure does not eliminate channeling effects as evident by the reduced intensity near 90 keV, below the Ga surface peak. The comparison between this experimental result and the simulation shows more clearly the contribution from Ga and Ga plus P signals. Also the cross-section dependence on the scattering angle is more clearly revealed in the simulation.

In order to obtain a map of the blocking directions of the GaP crystal, and thus to determine its stereographic projection, we followed the procedure described in detail in Refs. [11,12]. In short, the 2D-MEIS spectrum (as shown in Fig. 2 top panel, but now with the crystal rotated so the incoming beam is 25° away from the surface normal and generally not along a channeling direction) is measured. The intensity $I(\theta)$ in a selected energy range of $91.5 < E < 93.0$ is recorded. Then the crystal is rotated along the surface normal, and in this way the azimuthal (ϕ) angle is scanned over 142° with increments $\Delta\phi$ of 0.5°. The measured angular range is showed as a shaded area in the Fig. 1. The selected energy range corresponds to a backscattering depth of $\simeq 6 \pm 1.5$ nm for a scattering angle of 120°. In this energy window (region 1) only Ga contributes, as shown in Fig. 2. Changing the azimuthal angle also changes the direction of the incoming beam. For each azimuthal angle, blocking lines were observed on the 2D-MEIS spectrum at specific polar angles. For some azimuthal angle the incoming beam coincides with a planar direction and there was a clear channeling effect observable, causing reduced intensity for all polar values. By combining the $I(\theta)$ obtained at different ϕ angles, we obtain the $I(\theta, \phi)$ distribution that represents the cartography of the crystal.

3. Theoretical procedure

The MEIS cartography was compared with results of Monte-Carlo computer simulation implemented via the VEGAS code [13]. This program simulates the classical incoming and outgoing trajectories for a given scattering geometry. In the VEGAS code, the crystalline sample is described in terms of a two-dimension unit cell, which can fully retrieve layer-by-layer the crystalline structure when periodic conditions are