High Resolution Triple Axis X-Ray Diffraction Analysis of II-VI Semiconductor Crystals

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The objective of this research program is to develop methods of structural analysis based on high resolution triple axis X-ray diffractometry (HRTXD) and to carry out detailed studies of defect distributions in crystals grown in both microgravity and ground-based environments.

HRTXD represents a modification of the widely used double axis X-ray rocking curve method for the characterization of grown-in defects in nearly perfect crystals. In a double axis rocking curve experiment, the sample is illuminated by a monochromatic X-ray beam, and the diffracted intensity is recorded by a fixed, wide-open detector. The intensity diffracted by the sample is then monitored as the sample is rotated through the Bragg reflection condition. The breadth of the peak, which is often reported as the full angular width at half the maximum intensity (FWHM), is then used an indicator of the amount of defects in the sample.

In HRTXD, both the incident and the diffracted X-ray beams are conditioned by highly perfect (and typically multiple reflection) X-ray optics to reduce the angular and spectral divergences in both beams. The importance of this arrangement can be appreciated by considering the effect of structural defects on the diffraction process. Compositional variations and/or strains in the crystal lattice will locally change the lattice parameter and will be manifested in variations from the exact Bragg condition in a direction perpendicular to the lattice planes. In contrast, the presence of a mosaic spread in the sample or local angular distortions in the diffracting planes will alter the angular position at which a misoriented region of a crystal diffracts. This effect will be manifested as redistribution of the diffracted intensity in a direction that is parallel to the reflecting planes. By conditioning the incident and diffracted X-ray beams in both angle and wavelength in a HRTXD experiment, the strong, perfect-crystal contribution to the Bragg reflection peak can be discriminated from the weaker, off-peak scatter generated by structural defects. This allows the scattering from defects to be measured directly. HRTXD also has the advantage that it can be applied to crystals with very high defect densities, unlike other methods such as transmission electron microscopy and X-ray topography.

The resulting HRTXD data are commonly plotted as equal-intensity contours as a function of parallel and perpendicular deviations from the exact Bragg condition. These so-called "reciprocal space maps" permit an assessment of the nature and density of the structural defects in the diffracting crystal to be determined either by visual examination or more quantitatively by modeling from diffraction theory. This approach is highly relevant to microgravity research because it aids in the development of our understanding of the effects of microgravity on crystal growth and the incorporation of defects.

In the present work, we have been interested in characterizing the defect structures in crystals of ZnSe, CdTe, HgCdTe, and HgZnSe that were grown at the NASA Marshall Space Flight Center. Our principal results may be summarized as follows:

ZnSe: The cleaved surface of a ZnSe crystal grown by physical vapor transport (PVT) showed extensive diffuse scatter indicative of grown-in defects. A "surface streak" perpendicular to the (220) diffracting planes was clearly visible, which is consistent with the presence of both relatively good structural perfection and a smooth surface. Similar results were obtained from the growth facet of a different ZnSe crystal grown by PVT. In contrast, chemical-mechanical polished ZnSe crystals consistently exhibit extensive isotropic diffuse scatter and no surface streak, indicating severe polished-induced damage to the surface. Work is currently in progress to better understand the chemical-mechanical polishing process in ZnSe. We have also observed a small but significant spatial variation in the grown-in defect density in ZnSe, depending on the spatial position of the irradiated volume with respect to the gravity vector during crystal growth.

CdTe: Our most recent examinations of CdTe have been of a vapor-grown crystal without contact with the ampoule sidewalls. The presence of a well-defined surface streak was noted, although the intensity due to a mosaic distribution spanned approximately one-half degree in this sample.

HgCdTe: A recent analysis of both the first-to-freeze (34% Hg) and last-to freeze (8% Hg) ends of an HgCdTe crystal grown by the traveling heater method from a tellurium zone showed evidence for large and angularly-discrete mosaic blocks. In each block, little evidence of a surface streak can be observed, suggesting a low degree of structural perfection.

HgZnSe: Like the HgCdTe sample described above, a HRTXD analysis of HgZnSe grown at the NASA Marshall Space Flight Center demonstrated the presence of angularly-discrete mosaic blocks within the irradiated volume of the crystal and little evidence of surface streaks within those blocks. However, we also observed a systematic change in lattice parameter (presumably due to changes in the mercury content) as a function of angular deviation from the mean Bragg position, indicating a possible correlation between defect structure and chemical composition of the crystal. Work is in progress to more fully understand this observation.