Combined Synchrotron White Beam X-Ray Topography and High Resolution Triple Axis X-Ray Diffraction Characterization and Analysis of Crystals Grown in Microgravity and Ground-Based Experiments

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Research Objectives and Relevance to Microgravity

The objective of the research proposed here is to carry out detailed studies of defect and general distortion distributions in crystals grown in both microgravity and ground-based environments (e.g., using modified Bridgman or Vapor Transport techniques) using a combination of synchrotron white beam X-ray topography (SWBXT) and high resolution triple crystal X-ray diffractometry (HRTXD). This unique combination of techniques is expected to reveal detailed differences between crystals grown in these two different environments and to enable a meaningful assessment to be made of the influence of a microgravity environment on various aspects of the quality of crystals grown therein. SWBXT and HRTXD are complementary analytical techniques which are most sensitive and useful at contrasting ends of the "scale" of defect densities in crystals. By applying both techniques to the same crystals, it will be possible to provide detailed and quantitative assessments of the defect structure from regions that range from highly perfect (where SWBXT is expected to be the superior characterization tool) to highly imperfect (where HRTXD should be the superior method). This unified and continuous view of the defect structure should lead to valuable insights into the effect of growth conditions on the defect generation process. The proposed research program will draw upon the extensive experience that has been gained at the State University of New York (SUNY) at Stony Brook and the University of Wisconsin at Madison in the analysis of defects in semiconductors using advanced methods of X-ray diffraction. Prof. Michael Dudley of SUNY-Stony Brook has worked closely with the crystal growth community at NASA's Marshall Space Flight Center in applying methods of SWBXT to numerous semiconductor crystals grown both on earth and in microgravity. His counterpart, Prof. Richard Matyi of the University of Wisconsin at Madison, has been actively engaged in demonstrating the capabilities of HRTXD to a variety of semiconductor materials. By combining these complementary techniques it should be possible to obtain a deeper understanding of the process of defect generation than would be possible with either technique alone, or perhaps by any structural probe.

The significance of the research described here is that it will directly determine the influence of a microgravity environment on the detailed defect and distortion distribution in crystals produced in flight experiments, and will enable direct comparison to be drawn with crystals produced in ground-based experiments. Confirmation will be obtained that effects resulting from the limited

flight times available for microgravity crystal growth experiments do not exert control over the microstructure of the crystals grown, potentially detracting from the intended assessment of the influence of the magnitude of the gravity vector on these processes. Determination of the influence of cooling rate on the defect microstructure of crystals is crucial for selection of experimental conditions under which the effects of the gravity vector on crystal growth quality can be usefully investigated. Once such selection has been optimized, differences in microstructure observed in microgravity grown crystals may be safely attributed to the influence of the gravity vector and not to artifacts related to compressed growth schedules.

Significant Results to Date and Future Plans

To date, three materials have been studied cooperatively using SWBXT and HRTXD: ZnTe, CdZnTe, and ZnSe. Further work is under way on these materials in addition to HgZnSe and HgCdTe. (Some of this work was initiated as funded programs commenced under NRA-93-OSSA-12. Parallel work also continues under Universities Space Research Association-funded programs 3536-03 and 3537-04). Comparison of results obtained by these two techniques reveals good agreement. For example, in ZnTe SWBXT reveals a well defined cellular structure of dislocations with subgrain diameters in the range of 300 mm and relative tilts of the order of 5-10 seconds of arc. This agrees well with HRTXD results. In ZnSe, a much more even distribution of dislocations was revealed by SWBXT, with no cellular structure being discernible, a result again corroborated by HRTXD. The clarity of the dislocation images present on reflection topographs was strongly dependent on the state of strain of the crystal surface. Clear images were obtained through cleaved surfaces whereas blurred images, indicative of surface strain, were obtained through polished surfaces. This was corroborated by HRTXD with diffuse scatter, indicative of the presence of a high density of uniformly distributed dislocations, being present in both cases, but with the surface streak only being discernible from the cleaved surface. In the case of CdZnTe, SWBXT and HRTXD revealed that the defect structure was very much dependent on the local cooling rate, with the best quality regions being obtained at the slowest cooling rates. The best quality regions were revealed to have dislocation densities less than 10^3 cm^{-2}, with no subgrain structure observable, and with a few Te precipitates observed. In future work, a complete description will be sought of the type and distribution of all defects present in the crystals to be examined. Crystals will initially be examined in boule form using both SWBXT and HRTXD in reflection geometry in order to reveal the overall distribution of defects and distortion around the cylindrical surface of the crystal. This will help determine the optimal wafering geometry to be adopted for the next stage of the research. Once the boules have been suitably wafered, SWBXT and HRTXD will be used, in sequence, to examine the defect and distortion distributions in each of the wafers. Information so gathered will then be compiled to reconstruct to complete three-dimensional defect and distortion distributions in the as-grown boule. This reconstructed information can then be used to compare with the predictions of growth models. A preliminary attempt at the latter has already been published.