High-Resolution Three-Dimensional Imaging of Dislocations

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Dislocations and their interactions govern the properties of many materials, ranging from work hardening in metals to device pathology in semiconductor laser diodes. The geometry of a dislocation network reveals key information such as dominant slip mechanisms, dislocation loops, presence of locks, and stair-rod dislocations. Fifty years ago (*1*), transmission electron microscopy (TEM) enabled individual dislocations to be imaged and led to a far greater understanding of the relationship between the defect structure of materials and their mechanical and electronic properties. Conventional electron micrographs are, however, two-dimensional (2D) projections of three-dimensional (3D) structures, and even stereo microscopy cannot reveal the true 3D complexity of defect structures. Ludwig *et al*. (*2*) were the first to reconstruct a true 3D image of a dislocation network by using x-ray topography. However, dislocations separated by less than ~10 μm are difficult to visualize and limit the resolvable dislocation density to 10^6 cm^{-2}. We describe an electron tomographic method that yields 3D reconstructions of dislocation networks with a spatial resolution three orders of magnitude better than previous work. We illustrate the method's success with a study of dislocations in a GaN epilayer, where dislocation densities of 10^{10} cm^{-2} are common (*3*).

Hetero-epitaxial films of Mg-doped, *p*-type α-GaN (wurtzite) were grown on sapphire, with the lattice mismatch resulting in dislocations threading through the film in the [0001] direction (*3*). During growth and subsequent rapid thermal annealing to activate the Mg dopants, partial delamination of the film led to near-surface cracks and associated dislocation networks. In TEM, weak-beam dark-field (WBDF) imaging (*4*) is able to resolve individual dislocations separated by only a few nm. A series of WBDF images, recorded about a single tilt axis, can be used for a 3D tomographic reconstruction of a dislocation network if the diffraction conditions are kept constant throughout the tilt series. By ensuring the $\langle 11\bar{2}0\rangle$ zone axis was parallel to the goniometer axis, we maintained a constant weak-beam condition along the entire tilt range. Images were recorded every 5° with the $11\bar{2}0$ reflection and setting the $33\bar{6}0$ reflection near the Bragg condition. More than 90% of the dislocations had a Burgers vector component parallel to $\langle 11\bar{2}0\rangle$ and were therefore visible (*5*). Modified back-projection techniques were used to reconstruct the 3D dislocation network (*6*).

An oblique view of the reconstructed dislocation network is shown in Fig. 1, and an animated version is also available (movie S1). Although somewhat disturbed by a persistent "dust" arising from unwanted additional diffraction contrast (e.g., thickness fringes), the reconstruction shows the 3D structure of the dislocation network. Threading edge and mixed dislocations that bound small-angle grain boundaries of individual domains (labeled as D in Fig. 1) have a slight oscillatory appearance caused by dynamical diffraction effects associated with dislocations inclined to the beam. In-plane dislocations are very clear owing to their near-constant visibility throughout the tilt series, but dense dislocation bundles (labeled as B), associated with the crack tip, are not resolved. These dislocations are spaced ~10 nm apart, thus establishing a resolvable dislocation density of ~10^{12} cm^{-2}. The 3D reconstruction shows that the in-plane dislocations are caused by threading dislocations turning over (T) and gliding in response to the stress field of the dislocation bundle. The reconstruction also shows that the in-plane dislocations have parallel but not identical slip planes. A jog of an in-plane dislocation by a threading dislocation is also seen (J). Unannealed films did not show dislocation turn over and glide, suggesting that this mechanism requires thermal activation.

Three-dimensional reconstructions afford new perspectives on dislocation geometry, for example, the measurement of minimum distances between dislocations, the dislocation curvature (and the balance of line tension by local stresses), and the angles subtended by dislocations at surfaces (free or interfacial), which allow investigation of surface-dislocation forces. WBDF dislocation tomography is applicable to all crystal systems, but variations in the diffraction condition through the tilt series and materials with large elastic anisotropy can lead to distorted reconstructions. We anticipate that WBDF tomography will be useful for many dislocation-related issues, such as providing key input for models of 3D dislocation dynamics, a better understanding of dislocation interactions (e.g., pile-ups, cross-slips, jog formations, and kinks), and the visualization of dislocations under indentations.

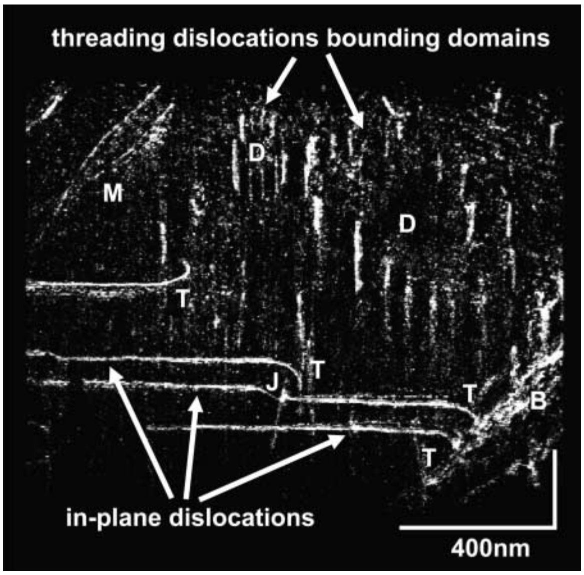

Fig. 1. An oblique view of a WBDF tomogram of a GaN film showing walls of threading dislocations surrounding domains (D), a dislocation bundle (B) associated with a crack, and threading dislocations that turn over at T to become in-plane dislocations and terminate at the specimen surface. Each turn over T occurs at a different height in the film, and one has interacted with a threading dislocation, causing a jog (J). Dislocations of mixed character (M) are also visible.

References and Notes

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5. Materials and methods are available as supporting material on *Science* Online.
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