

STEM-Based Characterization of Dislocations and Stacking Faults in Structural Materials

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The national initiative on Integrated Computational Materials Engineering holds great promise for accelerating the insertion of new materials in high performance structural applications. Achieving this aim relies upon the fidelity of materials models and their ability to capture the connectivity between processing, microstructure and performance. Advancements in our ability to characterize deformation mechanisms at finer length-scales are providing new insights into the governing deformation mechanisms in several important structural materials are being developed

This work will focus on the application of diffraction contrast STEM imaging, which has recently been demonstrated to hold significant advantages over conventional TEM imaging. While STEM has become an essential tool in materials science due to the ability to form a fine probe for chemical/composition analysis and high angle annular dark field (HAADF) imaging, the advantages of STEM for diffraction contrast imaging are less widely appreciated [1,2]. The present paper will demonstrate that the more “traditional” field of defect analysis also stands to benefit significantly from the use of advanced FEG-STEMs. This mode can provide image resolution and quality that is comparable, and in many cases superior, to that of the standard conventional TEM (CTEM) modes, while offering several important practical advantages [1-3]. The advantages include the suppression of auxiliary contrast effects (bend contours, etc.) and the ability to image in thicker specimens than is practical using conventional TEM at standard operating voltages. Additionally, CTEM visibility rules such as those for stacking faults and “ $g \cdot b$ analysis” for dislocations also remain in STEM mode provided the convergence condition and detector geometry are configured appropriately. These conditions will be described and image simulations that support these conclusions will also be presented.

Several examples of data acquired on an FEI Tecnai F20 field emission 200 kV S/TEM with a probe convergence angle of 5.9 mrad are shown in Figs. 1 and 2. Figure 1 shows an example of a “ $g \cdot b$ analysis,” demonstrating that dislocation invisibility can be achieved as in CTEM. An important added benefit of the STEM mode is the ability to form high contrast, bright-field images along low index zone axes - a mode which is not feasible with CTEM. This enables more straightforward determination of defect geometries, and greatly aids defect analysis since all defect content (with the exception of defects with net displacements parallel to the zone axis) can be viewed simultaneously. Quite remarkably, even when along a zone axis, specimen regions that are much thicker than possible with CTEM can be examined. An example of this benefit is shown in Fig. 2 where defects in a superalloy specimen that is 0.5 microns thick can still be clearly resolved. Quantitative analysis of defect strain field information is being enabled by simulation of STEM diffraction contrast images using the CTEM Soft program which can compute the Darwin-Howie-Whelan dynamical multi-beam equations for the systematic row and zone axis orientations over the range of incident beam directions given by the converged probe. In Fig. 3, a STEM dark field $g[-202]$ image of an $a[-101]$ near-edge superdislocation in an $Ni_3(Al,Ta)$ single phase g' alloy is shown. This dislocation is expected to dissociate into four Shockley partials. The simulated image (inset) results from

the assumed positions of the Shockley partials shown. The defect contrast is in excellent agreement with that observed. Image features as a function of dislocation core positions are presently under investigation and will be reported.

References

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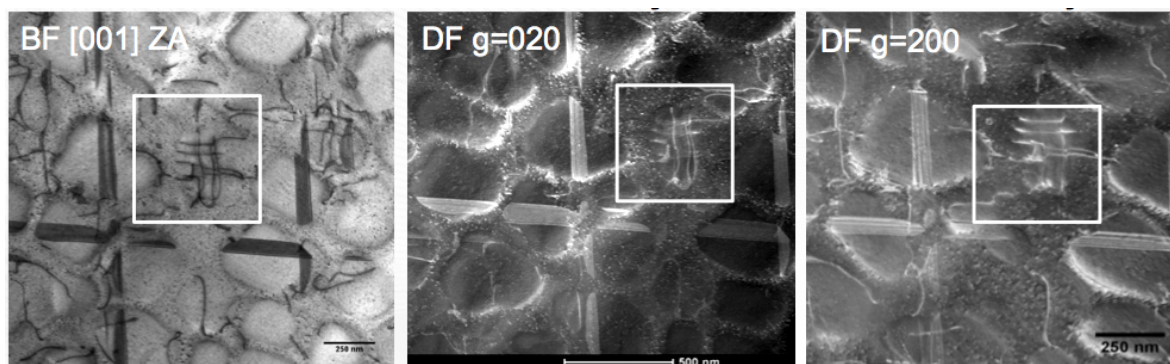


Figure 1. Example of a dislocation analysis in a Ni-base superalloy. Shown are zone axis [001] bright field and two dark field images using g_{020} and g_{200} . Inset region shows two different families of dislocations.

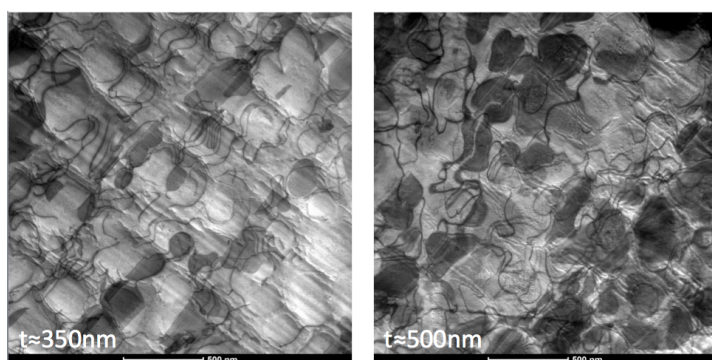


Figure 2. Examples of [001] zone axis images of dislocation and stacking fault structures in a Ni-base superalloy. The FIB foil thickness for each image are 350 nm and 500 nm, respectively.

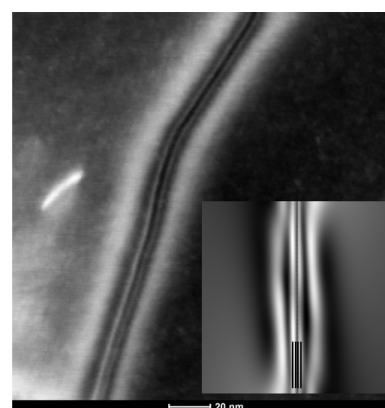


Figure 3. STEM dark field micrograph showing four-fold dissociation in Ni_3Al using $g[-202]$ diffraction vector obtained near the (111) glide plane. Inset shows a dynamical simulation of this defect with the Shockley partial dislocation cores marked by black lines.