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Tutorial review

Electron tomography of dislocation structures

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ABSTRACT

Recent developments in the application of electron tomography for characterizing microstructures in crystalline solids are described. The underlying principles for electron tomography are presented in the context of typical challenges in adapting the technique to crystalline systems and in using diffraction contrast imaging conditions. Methods for overcoming the limitations associated with the angular range, the number of acquired images, and uniformity of image contrast are introduced. In addition, a method for incorporating the real space coordinate system into the tomogram is presented. As the approach emphasizes development of experimental solutions to the challenges, the solutions developed and implemented are presented in the form of examples.

1. Introduction

The introduction and application of electron microscopy to the study of defects in materials in the 1950's opened new research directions with early successes being the direct observation of dislocations, including their motion, in aluminum [1,2]. With advances in instrumentation, including sample holders and recording media, computational hardware, and techniques, there have been significant advances in the spatial, temporal, and energy resolutions, which have opened new research avenues such that insight to the structural, compositional, electronic, and magnetic properties of materials are now possible. Instrumentation and recording media developments have transformed the instrument from exploring static to dynamic properties with temporal resolution spanning from picoseconds in ultrafast instruments to tens of milliseconds in conventional instruments [3–7]. Despite these advances, an electron micrograph is a projected image of the three-dimensional information as represented on the electron exit surface of the sample. That is, sample information in the electron beam direction is lost. This information can be recovered by applying stereomicroscopy techniques in which two micrographs acquired using the same conditions but separated by an angular tilt of ~15° are viewed simultaneously through a stereo-viewer [8] or used to form a red/blue anaglyph with the resultant image viewed through red/blue glasses. These approaches can provide three-dimensional information but are inflexible in terms of changing the viewing direction and options for visualizing the data are limited; Humphreys and Stewart solved this problem by constructing a wire model of the interaction, but this is a rare example [9]. An alternative approach is to recover the through thickness information using the tomography techniques pioneered by the biological community [10–13]. This approach essentially requires

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overlaying many images of the same object that have been acquired over a large angular range and using an algorithm, such as the weighted back-projection, WBP, or the simultaneous iterative reconstruction technique, SIRT, to form the three-dimensional tomogram. In the last decade electron tomography has begun to be applied in the physical sciences to explore the morphology of nanoparticles as well as magnetic domain structures [14–16]. To date there has been limited application of electron tomography to the investigation of defect structures [17–23]. The emphasis has been on dislocation structures in the nanometer regime [17–23] although more recent efforts are providing insight as to the dislocation structures at the atomic scale [23]. An example of the latter is the work of Chen et al. [23]. They achieved atomic resolution images of dislocations in a Pt nanoparticle by combining 3D Fourier filtering with high-angle annular dark-field scanning TEM tomography in which the center of mass method was used to align the tilt series and the equally sloped method to reconstruct it [24,25]. In this review, the application of electron tomography to the study of defects at the nanometer scale only will be highlighted and a practical method for its application presented.

The mathematical principles behind electron tomography, including the algorithms used in the reconstruction of the images, are described in detail elsewhere [12,13,26]. Here the emphasis is placed on the practical limitations relevant to forming a tomogram of defect structures in crystalline materials. Unlike serial-section tomography, which builds a 3D model from cross-sections, electron tomography uses through thickness projections as the source images. The specimen data in these projections do not map back directly to a single position in 3D space, but instead indicate how much of the electron beam has been transmitted or scattered along a straight line through the sample. According to the projection-slice theorem, the projection of an object at a given angle is a central slice through the Fourier transform of that object. By taking a sufficient number of projections over an appropriate angular range, a full description of the volume can be obtained. Correspondingly, by applying an inverse Fourier transform to the superposition of the Fourier transformed projections, a 3D real space reconstruction of the original object can be produced. To obtain an accurate representation of the volume, the acquired images must satisfy the projection requirement, i.e., the image contrast must vary as a monotonic function of the thickness and no more than one other physical characteristic. Most electron tomograms, however, are reconstructed by the simpler and less computationally demanding method of weighted back-projection [27], which is based on the fact that a point in 3D space can be uniquely defined by any three independent vectors that pass through it. If the specimen is regarded as an ensemble of many points, then each electron micrograph is effectively one of these vectors. By acquiring a sufficient number of projections of the specimen over an appropriate angular range, Fig. 1a, and aligning them, Fig. 1b, the information in the micrographs can be projected back onto the specimen center, recovering the 3D sample data. This concept can be visualized by considering Fig. 1c, in which the process is depicted as illuminating each aligned projection from behind and superpositioning the resultant beams to reconstruct the original specimen structure. Following the back-projection, a simple weighting function is applied to the Fourier transform to compensate for the fact that high-frequency/high-resolution features in the specimen change faster with tilt angle than low-frequency/low-resolution features [28]. Without this weighting filter, the relative under-sampling of the high frequencies causes fine spatial details to become blurred and obscured.

Ideally, projections should be acquired over an angular range of 180° in order to fully sample the entire Fourier space of the object. However, from a practical viewpoint, the angular range is limited by instrumentation and sample constraints. For example, the pole-piece gap of the objective lens restricts the dimensions of the sample holder with a concomitant restriction on the accessible angular range of tilt. This can be overcome by increasing the gap size, which will impact the resolution limit, or through design of novel holders [29]. In addition, some sample forms restrict the accessible angular range due to shadowing effects at high tilts. The instrument constraints are more important and are determining in terms of the maximum usable angular range.

The limited angular range affects the fidelity of the reconstructed tomogram as a volume of Fourier space is under-sampled; this is referred to as the "missing wedge" of information. As a consequence, the resolution of the

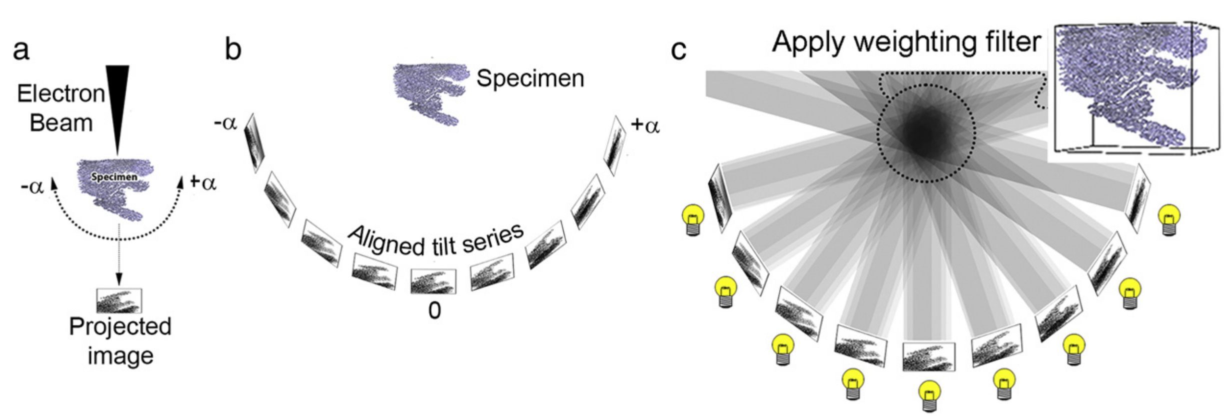

Fig. 1 – Weighted back-projection process. a. A 2D projected image of the 3D volume is collected; b. collection of such images at regular intervals over a wide angular range (±α); and c. back-projection of images with a weighting factor to reconstruct a tomogram of the original volume.