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Phase Identification by Selected Area Electron Diffraction

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Selected area electron diffraction (SAED) is an extremely useful technique for obtaining structural information from materials examined by transmission electron microscopy (TEM). The structural information derived from electron diffraction patterns in combination with chemical information from energy dispersive spectroscopy or electron energy loss spectroscopy helps in identification of phases. Despite the usefulness of SAED, there are limitations especially in comparison with the more fully developed technique of x-ray diffraction. For instance, SAED information is commonly limited to one orientation. D-spacings are extracted and compared to the d-spacings in a structural database. Structural information obtained from one orientation, however, does not necessarily uniquely identify a phase, or find related phases, and therefore ambiguities in identification commonly arise even with associated chemical information. In this work, consideration is given to an alternative approach to phase identification by SAED.

An alternative approach to phase identification using lattice matching techniques and converse transformation analysis was proposed over a decade ago by Karen and Mighell [1; and references cited therein]. By this approach, reciprocal space is sampled in three dimensions. Three d-spacings from three linearly independent directions and the angles between them are determined and used to define an arbitrary unit cell. Multiple 'primitive' cells are determined in this way and these cells subsequently analyzed by converse transformation algorithms where the type of relationship is deduced by the nature of the matrix relationships [1]. The results of the lattice analysis procedure can then be compared against a database containing crystalline information about phases of known materials. Given an appropriate tolerance for the magnitude of the d-spacings and angles, it is possible to uniquely identify a phase. The converse transformation procedure can reliably be used in spite of rather large experimental errors that can be routinely associated with lattice parameters determined from electron diffraction data. A modified and limited approach to the identification of unknown phases based on reduced cells and assuming small experimental errors, has been successfully implemented for single-crystal x-ray diffraction [2].

Application of the lattice analysis approach to identification by selected area electron diffraction has been hampered by the need to obtain multiple SAED patterns and to determine angular relationships between directions in the patterns. This information can be much more easily determined if simple tilt around crystallographic directions of interest is possible. This can be done if a crystallographic axis of interest is aligned with a sample holder tilt axis. Such tilting is possible with conventional TEM sample holders (double-tilt and single-tilt, rotate holders) only if, by chance, the sample is oriented along a sample holder axis. Typically, the sample will not be correctly oriented (Fig. 1) and, hence, the geometric relationships between directions in two diffraction patterns are difficult to determine. The availability of a new holder with three degrees of freedom for orientation has made possible the systematic three-dimensional sampling of reciprocal space by SAED. The holder, Gatan Model 925, is a double-tilt, rotate holder [3]. Previous work has shown that it allows for

systematic characterization of reciprocal space by tilting around directions of interest in reciprocal space [4]. Comparison between calculated and measured angles between diffraction patterns from different zones show agreement within 1°. Given the capability of this double-tilt, rotate holder, we are presently evaluating the application of the lattice analysis approach, using converse transformation analysis, to phase identification using SAED. By this process, cell data obtained from the double-tilt, rotate holder will be compared to a database containing cell data from known crystalline phases [5]. We intend to investigate the application of the approach initially on higher symmetry materials (cubic, tetragonal and orthorhombic). The tolerances necessary for such an approach will be determined. If proven successful for SAED, we envision automating the identification system using previously developed diffraction tools [6,7].

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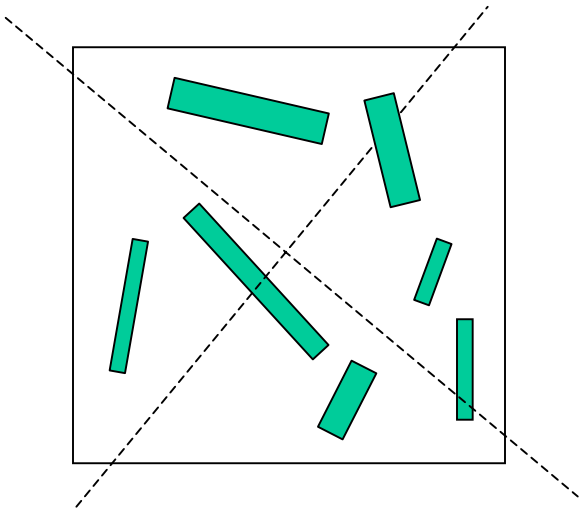

Figure 1. Sketch of grid square containing fibers. Dashed lines represent projection of tilt axes of a double-tilt TEM sample holder. The crystallographic axes of the fibers that are oriented along the fiber lengths are not aligned with the sample holder axes. Systematic tilt around either sample holder axis is therefore not possible. With the double-tilt, rotate holder, the fiber can be rotated to align the crystallographic direction of interest with a sample tilt axis allowing for simple tilting around the crystallographic direction of interest.

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