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Quantitative Convergent Beam Electron Diffraction

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Over the last few years, energy filtering, new digital recording systems, developments in theory and tremendous advances in computer power have opened up a new field of quantitative analysis of Convergent Beam Electron Diffraction (CBED) patterns [1-5]. Quantitative CBED techniques are still in an early stage of development and more research is required to fully understand their potential. This paper intends to give a review of the quantitative CBED methods used to determine low order structure factors from inorganic crystals and discuss some of the future prospects. Different examples of QCBED used in materials science will be shown.

The major advantage of the CBED method is the nanoscale electron probe size, which can give information from minuscule regions beyond the reach of other diffraction methods. Using such a small probe, one can almost always find a perfect crystal region where the theory of dynamical diffraction in a perfect crystal area is valid. Atomic resolution transmission electron microscopy (TEM) of the region the data is collected from can ensure that no defects are present. Another advantage is the fast electrons' strong interaction with matter, which gives rise to dynamical diffraction effects, making electron diffraction very sensitive to the crystal potential and the related charge distribution. This makes electron diffraction a powerful method for accurate measurements of low order structure factors (Fourier components of the crystal potential) and the study of crystal bonding.

Quantitative analysis consists of appropriate processing of experimental data and extraction of quantitative information from the processed data using a refinement method. This is done by pixel-by-pixel comparisons of experimental and theoretical intensities, obtaining the best fit by adjusting the parameters in the theoretical model using a goodness of fit criterion [2]. The intensity in one point in each disk in the experimental CBED pattern corresponds to exactly one diffraction condition (one incident beam direction), which makes the intensity distribution well suited for comparisons with theory. The theoretical intensities are usually based on the Bloch wave dynamical theory of high energy transmission electron diffraction [1]. A limited number of beams are included in the calculations, and additional ones are accounted for using the Bethe perturbation. Parameters refined are structure factors, absorption potentials, sample thickness, beam direction and scaling. Several diffraction geometries have been proposed [3-5]. Figure 1 shows an example of a systematic row CBED pattern with extracted line scans and the corresponding fit.

In quantitative work, energy filtering is crucial. Electron diffraction theory refers only to the elastically scattered electrons. Absorption is included in the calculations, but only as a removal of flux from the elastically scattered wave. Another important point, which has to be handled with great care, is the removal of the point spread function (PSF) in the digital recording systems (slow scan CCD cameras or imaging plates) [6,7]. The PSF comes from the averaged response of a parallel detector to a point signal.

In the refinement of low order structure factors, one needs prior information about the crystal structure. Lattice parameters, Debye-Waller factors, atom positions and operating voltage of the microscope are all fixed parameters in the refinements, and have to be known in advance. They remain fixed and errors will transfer to the refined values as systematic errors and limit the accuracy. Many of these parameters can be found using other CBED techniques [1].

The charge density associated with bonding is a very small fraction of the total charge in a solid. The bonding charge densities in a representative covalent crystal are typically of the order of 0.01% of the total charge around the core regions of the atoms. This illustrates the big challenge that the QCBED methods face and how accurate they need to be. The aim is refinement of the structure to such an extent that the deformation charge density can be found and predictions can be made about the nature of bonding, whether covalent, ionic or metallic. QCBED can give a small number of low order structure factors to very high accuracy (approaching 0.1 %). Bragg X-ray and QCBED methods complement each other [8]. Using the advantage of each method, QCBED can be used to measure accurately the absolute values of a few low-order structure factors and then be combined with X-ray results for weak and high order reflections. This has e.g. been done successfully in the case of CuO_2 [9]. [10]

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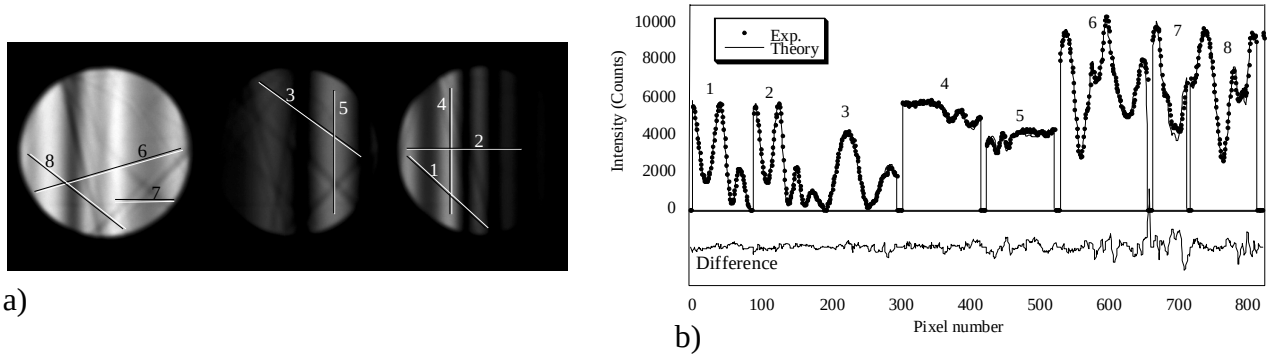

a) b)

Figure 1. a) Experimental CBED pattern from TiAl showing the (101) systematic row. Extracted line scans are indicated. b) Experimental line scans and best theoretical fit. The difference between theory and experiment is shown.

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