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ENHANCEMENT OF HIGH-RESOLUTION ELECTRON MICROSCOPY BY ELECTRON DIFFRACTION

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The electron diffraction pattern (EDP) formed on the back focal plane of objective lens and the image formed on the image plane are related each other by Fourier transform. In addition, the kinematical diffraction theory and weak-phase object approximation hold approximately for very thin samples, such as those affording structure images with atoms or atomic clusters as black dots. Hence, the amplitudes of structure factors can be obtained from the EDP, while phases can be obtained from the corresponding image but in a lower scattering angle range than that for the amplitudes. To combine the EDP provides the possibility to enhance the image resolution to the diffraction limit. The enhancement to high-resolution electron microscopy is not only due to the electron diffraction data, but also due to various kinds of diffraction analysis methods. The direct method has been employed to improve the image resolution and the Fourier synthesis technique to refine the obtained structure model. To enlarge the thickness range of observed crystals, a method of electron diffraction intensity correction was set up by referencing the Wilson statistic and the heavy atom method to reduce the deviation of intensities from the corresponding squares of structure factors. Besides, the dynamical diffraction effect correction is important in crystal defect determination at atomic scale by medium-voltage field-emission electron microscope.

Keywords: ELECTRON DIFFRACTION HIGH-RESOLUTION ELECTRON MICROSCOPY DYNAMICAL DIFFRACTION EFFECT

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PHASE RETRIEVING AND EXTENSION BY COMBINATION OF TIE AND GERCHBERG-SAXTON ALGORITHM

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The phase in the image plane of a transmission electron microscope always lost in the recording process. Recently, the technique of Transport of Intensity Equation (TIE) was used to resolve the phase retrieval problems from the two displaced intensity information. The TIE technique involves solving a Laplacian type of equation utilizing two images recorded with different focused values. We propose a new method of solving this equation. Basically, the Laplacian operator in TIE equation can be converted to be a convolution operator and therefore the phase information up to the information limit in the image plane can be obtained using regular de-convolution process- a maximum entropy de-convolution method. The phase information can be further extended beyond the information limit using Gerchberg-Saxton algorithm. Basically, Gerchberg-Saxton algorithm consists of two projections operated in real and reciprocal spaces cyclically. A generalized maximum entropy method (Kullback-Leibler cross entropy) is used as a real space (*P*1) projection. After *P*1 projection, not only the phases within the input spatial frequencies are improved, but also the phases in the higher frequencies are extrapolated. The optimum solutions from *P*1 projection can be further improved by a *P*2 projection that square root of diffraction intensities from a diffraction pattern are then substituted to complete a cycle operation of Gerchberg-Saxton algorithm. Application examples of the resolution extension for HRTEM image in the case of $NiSi_2$/Si interface and $\sigma = 5$ TiO_2 grain boundary will be shown in this talk.

Keywords: GERCHBER SAXTON ALGORITHM, TIE EQUATION, PHASE EXTENSION

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MAPPING CRYSTALLOGRAPHIC DISTRIBUTIONS IN THE TRANSMISSION ELECTRON MICROSCOPE

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Mapping of crystal orientations in the scanning electron microscope through use of electron backscatter diffraction patterns (EBSP) is now common practice. The spatial resolution limit is 20 nm. Below this limit it is necessary to use the transmission electron microscope. However, the normal operational mode of the TEM is not suited for collection of the large sets of diffraction data needed to produce a crystallographic map. It has been necessary to develop a new technique. The electron beam is tilted to enable dark field imaging from the first ring. A series of images are captured into computer memory as the beam is stepped around the ring at 2° intervals. The beam is tilted to successive rings in the first and second Laue zones and corresponding sets of dark field images recorded. The images are interrogated off-line to determine at what tilt rotation settings each pixel brightens. A pixel brightens only at the Bragg condition. Hence examination of all images enables the operating Bragg reflections to be determined for each pixel. The crystal orientation at that point is then calculated. Data collection time is 5 to10 minutes. The images are recorded at 256 x 256 resolution. Computer software developed for generating orientation maps from EBSPs in the SEM is used to generate the TEM based maps. The technique has been used in the examination of nano crystalline material, cross sections of semiconductor devices, and in highly deformed metals.

Keywords: DARK FIELD IMAGING, ELECTRON DIFFRACTION, ORIENTATION MAPPING

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REFINEMENT OF CRYSTAL STRUCTURAL PARAMETERS AND CHARGE DENSITY USING CONVERGENT-BEAM ELECTRON DIFFRACTION

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We have developed a method to refine crystal structural parameters using convergent-beam electron diffraction, which is applicable to nanometer-scale crystal structure analysis (K. Tsuda and M. Tanaka, Acta Cryst., A55 (1999) 939). The method is based on the fitting between theoretical calculations and experimental intensities of energy-filtered two-dimensional CBED patterns containing both of zeroth-order Laue-zone (ZOLZ) reflections and higher-order Laue-zone (HOLZ) reflections. The use of HOLZ reflections is essential for the present method because small displacements of atoms can be sensitively detected using HOLZ reflections with large reciprocal vectors. ZOLZ reflections can be utilized for determining low-order structure factors, which are very sensitive to valence electrons. For this purpose, we developed an energy-filter transmission microscope JEM-2010FEF, which can take energy-filtered CBED patterns covering high angles to include HOLZ reflections, and an analysis program MBFIT to refine structural parameters, which is based on many-beam Bloch-wave calculations and nonlinear least-squares fitting.

Using the method, we refined the atom positions, anisotropic Debye-Waller factors and low-order structure factors of the rhombohedral phase of $LaCrO_3$, which is a perovskite-type material for interconnector of solid oxide fuel cells. Clear anisotropy of the thermal vibration of the oxygen atoms was successfully detected for the first time. From the low-order structure factors, the charge transfer from the La and Cr atoms to the O atoms was visualized. The anisotropic charge density of the orbital-ordering phase of $LaMnO_3$ determined by the present method is also presented.

Keywords: CONVERGENT BEAM ELECTRON DIFFRACTION, STRUCTURE REFINEMENT, CHARGE DENSITY