

depending on the experimental condition [1-4]. The deficient line is black and the excess line white on photograph. The deficient-excess unindexed line is black on one side and white on the other side of the line. In the present work for the first time the contrast reversal along the unindexed line is obtained. The specimens were single crystalline silicon films prepared by chemical etching of bulky crystals. The transmission electron diffraction patterns were obtained in an EG-100M electron diffraction camera at an accelerating voltage of 100kV with the primary electron beam almost parallel to [111] axis. In the obtained Kikuchi patterns the unindexed line runs along the middle line of the Kikuchi band. The deficient unindexed line in the vicinity of the strong and spot reflections changes the contrast and transforms into excess line. The experimental conditions of unindexed line contrast reversal are founded. It is shown that the contrast is reversed when unindexed line passes through or in vicinity of an intense spot reflection. The contrast reversal of unindexed line is explained within the framework of the Kikuchi patterns formation mechanism with due regard for the double Kikuchi diffraction [5].

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Keywords: Kikuchi lines, electron diffraction, reflection

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Electron nanocrystallography: Advancements toward automated structure solution

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The synthesis of new nanocrystalline structures demands new rapid methods of solving their crystal structures. Our goal is real-time structure solution at the electron microscope, based on automated acquisition of three-dimensional electron diffraction data with subsequent phasing of the data set and presentation of a unit-cell potential map that displays atomic positions and even species. To achieve this we must consider: 1) translation of the specimen during automated tilting; 2) automated recognition of zone-axis orientations; 3) multiple-scattering artifacts; 4) indexing methods; 5) absolute intensity scaling of the data; 6) scaling of data collected at different orientations; and 7) the phase problem. Initially, we have focused on issues 3) through 7) following manual acquisition of three-dimensional diffraction data from a known test crystal (the MgAl₂O₄ spinel structure). Data was collected by two techniques, both of which minimize multiple-scattering artifacts: precession electron diffraction (PED) and kinematic convergent beam electron diffraction (CBED) using an in-column Omega energy filter. After indexing and scaling, experimental structure-factor magnitudes were obtained from the patterns. These provide input to the charge-flipping algorithm [1], which works well with relatively poor-quality electron diffraction data or powder diffraction data [2], to solve the phase problem and obtain the correct crystal structure. Solutions for PED and kinematic CBED data are presented for comparison with each other and with simulations. Further development requires automated, scripted control of specimen tilt and data acquisition.

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Contribution of electron precession to the identification of a new zirconium hydride

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A new metastable zirconium hydride designated as zeta-hydride, was identified and characterized in Zircaloy-4 alloys submitted to hydrogen cathodic charging or autoclave corrosion tests. Its crystal structure was obtained by combining TEM experiments and theoretical calculations. Using the electron precession microdiffraction technique, it was possible to identify slight differences of intensity between some weak extra reflections which prove that the highest "ideal" symmetry (the symmetry which takes into account both the position and the intensity of the reflections on a pattern) of this hydride is 3m. This symmetry is in agreement with a hexagonal lattice (with lattice parameters $a=0.33$ nm and $c=1.029$ nm) and with the Laue class -3m belonging to the trigonal crystal system. Then, the Zr₂H stoichiometric formula of the hydride was inferred from observations of plasmon peaks on EELS patterns. Finally, a structural model of the hydride, with space group P3m1, was deduced from ab-initio structure calculations and its validity was confirmed by subsequent dynamical simulations of the electron diffraction patterns.

Keywords: electron diffraction, crystal structure analysis, electron microscopy and diffraction

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A study of structure properties of ZnS nano-crystals using electron crystallography

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We have characterized the structure properties of two types of ZnS nano-crystals by electron crystallography. For determination of their initial structures, we have performed XRD analysis for ZnS crystals of 5 nm and 10 nm which were synthesized by same route. Their real crystallite sizes were about 5.9 nm and 8.1 nm and their crystal systems were hexagonal and cubic, respectively. Their quantitative structures, however, could not be determined because of their weak diffraction intensities. To overcome the intensity problem, the structure of ZnS nano-crystals was resolved by using EF-PED (Energy-Filtered Precession Electron Diffraction) and HREM (High Resolution Electron Microscopy) utilizing a HVEM (High