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A new method of processing and analysing electron diffraction patterns is presented that may have numerous analogues in other areas. It is demonstrated here in quantitative convergent beam electron diffraction (QCBED), for very precise measurements of structure factors. The technique maximises the sensitivity of structure factor measurement from diffraction data by almost completely eliminating the diffuse background contributed by inelastic scattering processes, most notably, thermal diffuse scattering (TDS). This is demonstrated in fig. 1. The present work is an extension to [1] and covers both energy-filtered and unfiltered CBED.

[1] P.N.H. Nakashima, Phys. Rev. Lett. 99 (2007), 125506.

[2] Thanks to A. Prof. J. Etheridge, Prof. A. Moodie, A. Prof. A. Johnson, Dr. V. Streltsov, the Australian Research Council (DP0346828) and the Australian and Victorian Partnerships for Advanced Computing.

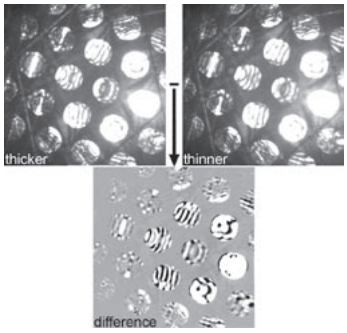

Fig. 1: Two zero-loss-filtered CBED patterns (6eV slit width) from different thicknesses of corundum. The background (outside the discs) in both patterns still contains significant signal due to inelastic scattering (mostly TDS), which is almost completely canceled in the difference pattern.

Keywords: inelastic scattering, thermal diffuse scattering, accurate structure factors

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Automatic space group determination using precession electron diffraction patterns

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A set of algorithms for automatic High Order Laue Zone (HOLZ) indexing and possible set of space groups extraction were developed and implemented in the "Space Group Determinator" program. The symmetry analysis is performed using Morniroli-Steeds tables [1,2]. The developed program becomes extremely useful for the analysis of precession electron diffraction patterns (PEDs) [3]. "Space Group Determinator" was successfully tested on both simulated and experimental diffraction patterns with different zone axis orientations [4]. There are several advantages using PEDs: - The intensities extracted from PEDs are less dynamical, especially for main zone axes; - There are more reflections with higher resolution visible (depending on the precession angle); - The width of a HOLZ band (if visible) can be significantly larger than on a corresponding SAED pattern. The last statement is especially important for the correct space group or set of space groups determination. The possibility to observe several reflection lines within HOLZ makes the plane lattice shifts extraction easier. The knowledge of FOLZ shift with respect to the ZOLZ and the possible differences in periodicities provides very important information. The corresponding plane shifts in a* and b* directions between ZOLZ and HOLZ can be used together with tables from [1] for finding lattice centering, glide planes and partial symmetry symbol. This information can be treated systematically and implemented in the automatic procedure.

1 J.P. Morniroli, J.W. Steeds, Ultramicroscopy 45 (1992), p. 219-239.
2 J.P. Morniroli, A. Redjaimia, S. Nicolopoulos, Ultramicroscopy 107 (2007), p. 514-522.
3 R.J. Vincent, P.A. Midgley, Ultramicroscopy 53, 3 (1994), p. 271-282.
4 P. Oleynikov, PhD thesis, Stockholm University, (2006), p. 73-78.

Keywords: space groups, electron diffraction, precession

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Determination of order parameter of single $L1_0$-FePd nanoparticle by nanobeam electron diffraction

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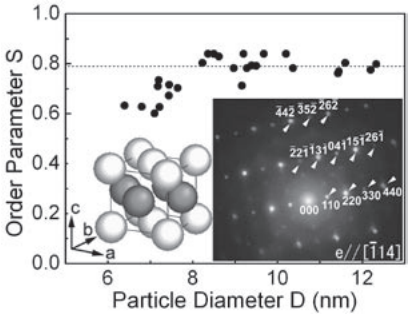
Long-range order (LRO) is the key issue in the structure-property relationships of the hard magnetic FePd nanoparticles with the $L1_0$ structure. In this study, we introduced a new technique to determine the LRO parameter of single FePd nanoparticle using nanobeam electron diffraction (NBD). The LRO parameter was determined by quantitative analysis of NBD intensities recorded by imaging plates together with intensity calculations considering the multiple scattering of electrons. In taking NBD patterns, hh0 systematic reflections were excited using a JEOL 3000F transmission electron microscope. Specimen thickness was evaluated by electron holography. The obtained LRO parameters of nanoparticles larger than 8 nm are distributed around the average LRO parameter (S=0.79) determined by selected area diffraction. In contrast, the LRO parameters gradually decrease as the particle size decreases below 8 nm (S=0.60-0.73). Experimental conditions required for NBD analysis are presented and the possible experimental errors are discussed. Attached figure shows the size dependence of the LRO parameters. A schematic of the $L1_0$ structure and an example of NBD pattern are shown in the inset.



Keywords: microdiffraction, ordered structures, nanoparticles

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Contrast reversal of unindexed Kikuchi lines

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Kikuchi patterns can contain the unindexed line which runs along the middle line of a Kikuchi band and cannot be indexed as a Kikuchi lines. It appears as an excess, deficient or excess-deficient line

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].

[1] Pfister H. Ann. Phys.Lpz. 11, 239, 1953.
[2] Uyeda R. and Nonoyama M. Japan. Journ. Appl. Phys. 4, 498, 1965.
[3] Kainuma Y. and Kogiso M. Acta Cryst. A 24,1. 81. 1968.
[4] Karakhanyan R.K. and Grigoryan P.A. Kristallografiya. 24, 1, p. 159. 1979.
[5] Karakhanyan R.K., Aleksanyan P.L., Manucharova J.K. Phys. Stat. Sol.(a), 121, p.K1. 1990.

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 $MgAl_2O_4$ 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.

[1] G. Oszlanyi and A. Suto. Acta Cryst., A60, 134 (2004).
[2] J.S. Wu, J. Spence, M. O'Keeffe, and K. Leinenweber. Nat. Mater., 5, 647 (2006).

Keywords: electron crystallography, precession, structure solution

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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 Zr_2H 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