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EXPERIMENTAL ESTIMATION OF DYNAMICAL SCATTERING EFFECT ON ELECTRON CRYSTALLOGRAPHY

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In order to analyze crystal structures of fine particles and thin films, electron crystallography is highly advantageous because of its larger cross section than those for X-ray and neutron. However, electron diffraction intensity is known to vary drastically as a function of thickness owing to the dynamical effect that can be estimated in principle when measured actual thickness of samples. In this study, such correlation between the dynamical effect and thickness was examined in the cases of organic crystals of perylene and bis(dimethylglyoximato)platinum whose crystal structures are known. A plate-like crystal was grown epitaxially on alkali-halide single crystalline substrates with a uniform thickness between 20 nm and 150 nm by controlling deposition amount. The electron diffraction intensities were recorded on an imaging plate at accelerating voltages of 80-1000 kV, and the corresponding dynamical scattering intensities were calculated by a multi-slice method. As a result, observed intensities do not fit with simulated ones for the thickness measured with an atomic force microscope, but agree with intensities simulated at thinner thickness in both samples; about 25 nm for perylene and about 1.3 nm for bis(dimethylglyoximato)platinum. This fact indicates that dynamical coherent thickness is remarkably thinner than the measured one, so that crystal structures were successfully analyzed so far in the frame of kinematical scattering in many cases of organic crystals [1, 2].

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Keywords: ELECTRON DIFFRACTION, DYNAMICAL EFFECT, THICKNESS

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CHARGE DENSITY MEASUREMENT IN MgB_2 BY CONVERGENT-BEAM ELECTRON DIFFRACTION

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The aim of this work is to make very accurate measurements of the ground state charge density in MgB_2, and so understand the role of the valence electrons in the bonding of MgB_2. The role of the free electron contribution to superconductivity, the charge density state in the MgB_2 crystal and band theory calculations will also be tested. We do this through accurate measurements of the low-order structure factors (which are most affected by bonding), using both electron and X-ray diffraction. Measurement accuracy must be below 1% to see bonding effects. Several methods have been used to measure low order structure factors, the most common being Bragg X-ray diffraction and the quantitative (QCBED) method [3]. These two methods are complimentary. X-ray diffraction suffers large extinction, but can measure the high order reflections very accurately, which are sensitive to atomic coordinates and temperature factors, and can be used for crystal structure determination. The QCBED method takes advantage of the small probe size in a transmission electron microscope (smaller than a mosiac block), and the enhanced sensitivity the Mott Formula gives to electron scattering to bonding. Large perfect crystals are not needed. By combining the two methods, a complete and accurate data set can be obtained.

Comparing X-ray and electron scattering factors for MgB_2 by band calculation. These show a very large percentage change in electron structure factors (up to 75%) for low orders due to bonding. The magnesium atoms are purely ionic, but boron atoms show strong covalent bonding. Experimental data collection is in progress.

Keywords: CBED, MgB_2, CHARGE DENSITY

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SAPPHIRE BY COMBINED ELECTRON AND X-RAY DIFFRACTION

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Quantitative convergent beam electron diffraction (QCBED) data have been collected from numerous crystals of sapphire using different electron energies, crystal thicknesses and crystal orientations. Many-beam dynamical electron diffraction calculations (Bloch-wave and multislice) have been used in pattern-matching refinements yielding multiple measurements of structure factors. The reproducibility provides a test of the reliability of QCBED under many different conditions, including the different theoretical formalisms.

The error in the reproducibility of strong, low-order structure factors by QCBED applied to sapphire, was of order 0.2%. These structure factors correspond to the reflections most strongly affected by extinction in single-crystal kinematic X-ray diffraction experiments. The QCBED technique was also used to measure a number of weak structure factors, less likely to be affected by extinction in the X-ray experiments. The error in reproducibility of these measurements was of order 1%. Synchrotron X-ray diffraction determined structure factors for sapphire were corrected for scale and extinction using the QCBED results. The QCBED values were then substituted for the corresponding synchrotron X-ray data. The combined data set was used to perform multipole refinements of the static deformation electron density. Comparisons to the results of Density Functional Theory (DFT) and *ab-initio* Periodic Hartree-Fock (PHF) calculations were made. The experimental results agree very strongly with published results of DFT (where the generalised gradient approximation (GGA) and full-potential linear augmented plane wave (FPLAPW) method were applied) whilst there is less agreement with those of PHF calculations.

Keywords: **COMBINED QUANTITATIVE CONVERGENT BEAM ELECTRON AND SINGLE CRYSTAL SYNCHROTRON X-RAY DIFFRACTION DATA, ACCURATE STRUCTURE FACTOR MEASUREMENTS, DEFORMATION ELECTRON DENSITY MAPS**

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COMBINATION OF ELECTRON DIFFRACTOMETRY, IMAGING PLATES (IP) AND ELECTRON DIFFRACTION STRUCTURE ANALYSIS (EDSA)

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Electron diffractometry studies have proved that correct intensity values are of decisive importance for most detailed and reliable structural data to be obtained. Electron diffractometry based on direct intensity measurements by stepwise scanning of the diffraction pattern against a fixed detector has provided by now the most precise values presented in an absolute digital form. Their linearity in a wide range about (10^6 e/sec) was verified in special experiments (at variable intensities of the primary beam). However the intensity measurements need long experiments and are problematic for not stable objects. The use of IP is free from this disadvantage providing quantitative intensity distributions of any complicated patterns at a short single exposure. The combination of both approaches makes EDSA most efficient presenting the atomic positions, interatomic bonds and atomic states by space distributions of the inner crystalline electrostatic field. The first experiments with the use of texture patterns of a number of various hydroxides and phyllosilicates have revealed a good agreement of results obtained with the two techniques and opens a new life for the EDSA.

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Keywords: **ELECTRON DIFFRACTOMETRY, IMAGING PLATES (IP), TEXTURE PATTERNS**