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NEW TECHNIQUES FOR TEM NANO-ANALYSIS : PRECESSION DIFFRACTION AND 3D DIFFRACTION TOMOGRAPHY FOR STRUCTURE DETERMINATION AND (EBSD-TEM LIKE) HIGH RESOLUTION PHASE/ORIENTATION MAPS

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Precession electron diffraction (PED) is a new promissing technique for electron diffraction patterns collection very close to kinematical condition (like in X-ray diffraction) allowing this way to solve ab-initio crystal structures of nanocrystals. PED intensities help to solve nanocrystal structures (inorganic metals, ceramics, minerals up to polymers, organic structures, pharmaceuticals and even proteins), even in cases where X-ray synchtrotron data may fail to solve the structure.

On the other hand, another exciting development in electron crystallography is the 3dimensional diffraction tomography technique which consists in an automatic collection of a series of randomly oriented diffraction patterns in precession mode of the same crystal through the whole TEM angular range, usually from -45 to +45 , at 1 angular intervals. The resulting 3D electron diffraction set of reflections can be visualized as clear 3D picture of the reciprocal cell of the crystal; exciting applications like direct cell determination, crystal defect such as twinning or streaking or industrial applications like polymorph screening are possible now.

A new exciting application has also been developed for an EBSD-TEM like phase and orientation maps for nanocrystals. PED precession interface may perform a scanning with a small step (1-35 nm, depending on TEM source) through a sample area (example 5x5 m^2), resulting in a collection of a large number of diffraction patterns which are compared one by one by cross-correlation techniques with a series of generated diffraction patterns (templates) of all possible orientations of known phases existing on the scanned area.The resulting high quality, high resolution (1-2 nm) orientation and phase maps obtained in TEM are much superior to equivalent EBSD-SEM orientation maps. Moreover, there is no need for specific surface specimen preparation (like in EBSD-SEM), because with this technique all diffracting crystals have enough signal to produce high resolution orientation maps. Such orientation and phase maps may be produced in few minutes in any materials, making the technique highly attractive for high throughput EBSD-TEM structure analysis.

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Abstract

Electron diffraction is the only available diffraction method applicable to investigation of single crystals of sub-micrometer size. Strong dynamical scattering effects occurring during the interaction of electrons with crystals result in a situation, where standard electron diffraction can be used to infer geometric information about the crystal, but not for quantitative structure analysis. The situation is changing with the advent of a new method called Precession electron diffraction, which partly suppresses the dynamical effects, and allows for solution of crystal structures from electron diffraction data. The newly established laboratory for electron diffraction at the Institute of Physics AS CR aims at introducing and developing the method of precession electron diffraction so that it becomes a standard crystallographic tool that can complement and compete with established diffraction methods.

Abstrakt

Elektronová difrakce je jedinou dostupnou difrakční metodou pro zkoumání krystalových struktur jednotlivých krystalů submikrometrových rozměrů. Silné dynamické efekty vznikající při difrakci elektronů na krystalech způsobují, že standardní elektronovou difrakci lze využít pouze pro získání geometrické informace o krystalu, a nikoli pro kvantitativní strukturní analýzu. Tuto situaci mění nová metoda precesní elektronové difrakce, která potlačuje dynamické difrakční jevy, a umožňuje řešit