

Voltage Electron Microscope). EF-PED data resulted in the basic crystallographic information of ZnS nano-crystals: $P63mc$, $a = 3.83\text{\AA}$ and $c = 6.26\text{\AA}$; $F-43m$, $a = 5.41\text{\AA}$, respectively. As a result of HREM analysis, their crystallite shapes were turned out to be nano-rods and nano particles, respectively and the nano-rod structure was elongated to the [001] direction. The size and shape dependency related to crystal structures of ZnS nano-crystals has been discussed.

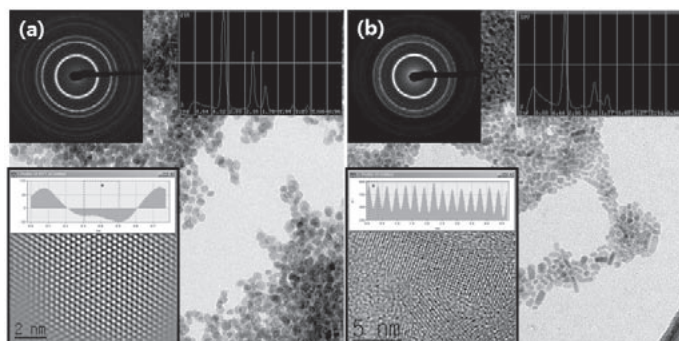


Fig. 1. EF-PED and HREM analysis of ZnS nano-crystals.

(a) Cubic structure of ZnS nano particles. (b) Hexagonal structure of ZnS nano-rods

Keywords: electron crystallography, energy-filtered precession electron diffraction, ZnS

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Electrostatic potential analysis of the orbital-ordered phase of spinel oxide FeCr_2O_4 using CBED

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A structure analysis method using convergent-beam electron diffraction (CBED), which was developed by Tsuda and Tanaka[1,2], enables us to directly determine the electrostatic potential from a nanometer-scale specimen area. The electron density distribution can be also obtained from the electrostatic potential through the Poisson's equation. We have applied the method to a spinel oxide FeCr_2O_4 , which undergoes a structural phase transformation at 135K from a cubic spinel structure of the high-temperature phase to a tetragonal structure of the low-temperature phase with Jahn-Teller(JT) distortion[3]. A ferro-type orbital-ordering of 3d electrons of Fe atoms is expected in the low-temperature JT-distorted phase of FeCr_2O_4 [4]. FeCr_2O_4 single crystals were grown by the chemical vapor transport(CVT) method. Energy-filtered CBED patterns of the low-temperature phase were obtained at a specimens temperature of 90K using an energy-filter transmission electron microscope(EFTEM) JEM-2010FEF with an accelerating voltage of 100kV. Atom positions, Debye-Waller factors and low-order structure factors were refined by nonlinear least squares fitting between the experimental CBED patterns and dynamical diffraction calculations using an analysis software MBFIT[1]. The electrostatic potential and electron density distributions were reconstructed from the refined parameters. Clear anisotropy of the electrostatic potential has been found, which is attributed to the ferro-type ordering of the $3z^2-r^2$ orbitals of the Fe atoms.

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Keywords: CBED, electrostatic potential, spinel

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Microstructural characterization of YPO_4 : Li by transmission electron microscopy

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Xenotime YPO_4 is an yttrium phosphate with a tetragonal structure and can be doped by ions of diverse diameters in order to control its electrical conductivity. The purpose of this work is to present an study about the microstructural characterization of YPO_4 by electron transmission microscopy when this yttrium phosphate is doped with lithium. Samples were obtained by the following solid state reaction: $\text{Y}_2\text{O}_3 + (\text{NH}_4)_2\text{HPO}_4 \rightarrow \text{YPO}_4 \cdot x\text{Li}$ Powder samples were studied in a JEOL 100 CX with point resolution of 2.7angstrom. Brighth field and Diffraction techniques were used to establish that $\text{YPO}_4 \cdot x\text{Li}$ has an orthorhombic structure with lattice parameters: $a=b=9.7$ angstrom and $c=6.01$ angstrom, this means that interstitial lithium ions can create a distortion in the tetragonal structure of yttrium phosphate.

Keywords: xenotime, orthorhombic yttrium phosphate, electron microscopy

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Determination of chiral indices of carbon nanotubes using electron diffraction pattern

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Carbon nanotubes (CNTs) show physical properties of a metal or a semiconductor depending on these structures. Thus, it is important to investigate the structure of a single CNT expressed by a chiral index (n, m) [1]. Transmission electron microscopy is a powerful tool to investigate structures of single CNTs. In the present study, the chiral indices are determined by using electron diffraction patterns obtained from single double-walled CNTs (DWCNTs) and single single-walled CNTs (SWCNTs). Electron diffraction patterns were obtained from 200 nm areas containing a single DWCNT or SWCNT by using a transmission electron microscope of JEM2010 at an accelerating voltage of 100 kV. Intensities of the diffraction patterns were recorded on imaging plates. Determination process of chiral indices is as follow. 1.The chiral angles and diameters of the CNT are estimated from the diffraction patterns. 2.The chiral indices (n, m) of the CNT are estimated from the angles and the diameters. 3.Diffraction patterns of chiral indices (n, m) were simulated using a simulation program "MBFIT" [2, 3]. 4.The simulated diffraction patterns are compared with the experimental patterns. 5.Chiral indices (n, m) were determined from the best agreement between the experimental and the simulated patterns. The determination of chiral indices enables us to calculate the joint densities of states of the CNTs and to compare electron energy-loss spectra of the CNTs with joint density of states of the CNTs calculated from chiral indices (n, m).

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Keywords: carbon nanotubes, electron diffraction analysis, structure determination

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Crystal structure of nm-scale precipitates in Al alloys by electron diffraction and DFT calculations

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The macroscopical properties of Al alloys, such as hardness and formability, are to a large extent determined by their nanostructural properties. During precipitation hardening of Al alloys, several metastable phases are formed as nanometer-scale precipitates. An important aspect in studying the hardening effect of the different phases is the coherence strain between the precipitates and the surrounding matrix. It is essential to obtain an accurate model of the crystal structure of these precipitates, since this will allow for detailed modeling of the precipitate-matrix interface. The crystal structure of several of the hardening phases has been successfully determined, based on quantitative electron diffraction and verified by DFT calculations. Indeed, generally, electron diffraction and DFT calculations would lead to the same selected structure out of a number of candidate models, and structural relaxation in the DFT calculations gave only minor differences with the experimentally determined structure. However, most DFT calculations so far have assumed a 'bulk' precipitate structure, i.o.w. repeating infinitely in 3 dimensions, which was an acceptable approximation in most cases, but has proven to be inadequate for the smallest precipitates. Therefore, in more recent work, it has been decided to include the precipitate-matrix interface in the DFT calculations, where the initial model for the interface structure is based on a combination of electron diffraction and high resolution electron microscopy images.

Keywords: electron diffraction, DFT, alloys

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Structural studies of spin crossover compounds under extreme environmental conditions

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Spin crossover (SCO) is the switching between high and low spin states which arises from a variation in temperature or pressure or as a result of light irradiation. The understanding and resultant control of this process is fundamental to the potential applications of these systems in data storage and novel electronic devices. Results of this transition include a dramatic change in the metal-ligand bond distance and often striking changes in the crystal structure. The vast majority of SCO systems studied to date involve mononuclear

iron (II) species that are linked in a crystal through intramolecular interactions. There is an increasing trend towards the study of complexes with higher dimensionality such as 1D-3D coordination polymers and polynuclear metal complexes, largely due to increased cooperativity between iron centres through direct covalent bonding [1]. Application of hydrostatic pressure serves as a tool for modifying the local metal environment in a controlled manner [2]. High pressure studies have been shown to be invaluable in revealing the cooperative nature of these electronic structure transitions, which are accompanied by a significant change in volume. In Durham we are currently developing a set-up which will allow high-pressure low-temperature single crystal diffraction studies to be carried out. This poster will show our first results from single crystal experiments aiming to combine pressure and temperature on a spin crossover material. This research is intended to aid in the understanding of how sample environment impacts on the complex nature of the spin crossover phenomenon.

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Keywords: structural and magnetic phase transitions, diamond anvil cells, low-temperature crystallography

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Structural stability of the FeCr sigma phase under pressure to 77 GPa

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The Fe-Cr alloy system contains an intermediate phase of the equiatomic composition called the sigma phase. The crystal structure of the sigma phase is tetragonal with 30 atoms in the unit cell, space group $P4_2/mnm$, lattice parameters $a = 8.7995 \text{ \AA}$, $c = 4.5442 \text{ \AA}$, $c/a = 0.516$, Pearson symbol tP30 [1], whereas pure elements Fe and Cr both crystallize in the body-centered cubic (bcc) structure. The aim of this work was to investigate the stability of the FeCr sigma phase at high pressure. Diffraction studies of the FeCr sample with the sigma phase were performed using diamond anvil cell, image plate detector and synchrotron radiation at the Advanced Photon Source (Argonne National Lab). The highest pressure achieved in this study was 77 GPa. The sigma phase was found to be stable in this pressure range. The equation of state for the tetragonal structure was found to have the values of bulk modulus $K_0 = 217(5) \text{ GPa}$ and $K_0' = 5.8(2)$. Interestingly, the compressibility of the FeCr sigma phase is lower than that of the constituent elements: the bulk modulus K_0 for the bcc phases of Cr and Fe is 161 and 173 GPa, respectively. Structural stability of the sigma phase under pressure found in the present study supports the suggestion that electronic energy is one of the main factors responsible for the existence of this complex, low-symmetry structure. The stability of the sigma phase can be related to the Hume-Rothery effects based on the Fermi surface - Brillouin zone interactions [2]. Partial support from RFBR under grant 07-02-00901 is acknowledged.

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Keywords: high-pressure diffraction studies, binary alloys, phase stability