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Structural Studies of Nanocrystalline Metals
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Microstructure of metals (Cu, Fe, Mg, Cu with Al_2O_3) obtained by high-pressure torsion and its evolution with post annealing was studied by conventional powder diffraction in symmetric Bragg-Brentano geometry. This included evaluation of lattice parameters and texture but mainly line profile analysis in terms of both approximate modified Williamson-Hall method and total powder pattern (or multiple line profile) fitting [1-3]. Typical line-broadening anisotropy was explained by dislocation line broadening and elastic anisotropy. Dislocation density in the order of 10^{15} m^{-2} and crystallite size (~ 50 - 500 nm) were determined. The method was completed by X-ray film technique (area detection), diffuse scattering in the transmitted wave, transmission electron microscopy and life-time positron annihilation spectroscopy. Common features and differences for the microstructure evolution of studied metals are discussed.

Films made of small amount of colloidal Au nanoparticles prepared with pre-calculated size were investigated in parallel beam geometry. The diffraction line profile analysis also revealed strong line broadening anisotropy and indicated not only small crystallite size but also the presence of stacking faults and dislocations. The results were confirmed by conventional and high-resolution TEM and UV/vis spectroscopy

[1] Scardi P., Leoni M., *Acta Cryst*.,2002, **A58**, 190-200. [2] Scardi P, Leoni M., Dong Y.H., *Eur. Phys. J*.,2000, **B18** , 23-30. [3] Ribárik G, Ungár T., Gubicza J., *J. Appl. Cryst.*, 2001, **34**, 669-676.
Keywords: powder diffraction, line profile analysis, severe plastic deformation

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Size Anisotropy and Lognormal Size Distribution in the Powder Diffraction Whole Pattern Fitting
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The approach developed by Popa and Balzar [1] to model the size broadening in powder diffraction patterns by samples with lognormal size distribution of spherical crystallites can be easily extended to include size anisotropy if the crystallite shape is approximated by an ellipsoid.

In comparison with the existing approaches using ellipsoids to describe the size anisotropy, this approach uses a peak breadth symmetrized according to the crystal Laue class.

The proposed model was tested on a zinc oxide diffraction pattern measured in a Bragg – Brentano geometry. The model is compared with the previously proposed model using spherical harmonics to describe the size anisotropy [2].

[1] Popa N. C., Balzar D., *J. Appl. Cryst*.,2002, **35**, 338-346. [2] Popa N. C., *J. Appl. Cryst.*, 1998, **31**, 176-180.
Keywords: powder diffraction, size effect, anisotropy

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Development of a NIST SRM 1979 Nano-Crystallite Size Standard for Broadening of X-ray Line Profiles
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The development of traceable material standards is critical in the advancement of nano-technology. A NIST nano-crystallite size Standard Reference Material (SRM) 1979 has been developed for this very purpose, assuring improvement in the measurement accuracy of the size distribution and shape of nano-crystallites from X-ray line profile analyses and electron microscopy techniques.

SRM 1979 will consist of two materials prepared with the use of flow reactor technology to permit large, 1 kg batches. Production methods were chosen to minimize the presence of structural defects that may contribute to line broadening. The first material sample is ceria (cerium (IV) oxide, CeO_2) with an (approximate) average spherical crystallite size of 20 nm over a size range of 5–35 nm. The second is zinc oxide (ZnO) with approximately cylindrical crystallite morphology of 80 nm and a size range of 60–100 nm.

The certification of the SRM has also seen the development of a Bayesian/maximum entropy method. This analysis takes full account of the form of the instrumental, background and statistical noise contributions embedded in the diffraction data. As well as providing the most probable solution, the method produces a full error analysis of the size distribution— a critical element in certifying SRM 1979.

The X-ray analysis presented here will be compared with the results of direct observations of SRM 1979 using TEM imaging, and a discussion based on this comparison will be presented.
Keywords: nanocrystallites, standard reference material, line profile analysis

MS61 Structures Phase Transitions and Properties at High Pressure

Chairpersons: Mohammed Mezouar, Nozomu Hamaya

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Single Crystal Studies of the Incommensurate Composite Structure of Rb-IV
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A number of elements have recently been shown to have a composite incommensurate high pressure phase. This phase has a so called 'host-guest' structure type comprising a tetragonal 'host' framework with channels along the fourfold c axis. Within these channels are 1D chains of atoms that form a 'guest' structure that is incommensurate with the host. We have observed host-guest structures in the alkali and alkaline-earth metals Rb, Sr, Ba, and in the group Va elements Bi, Sb and As [1]. Among these elements Rb is unique in having a 16-atom host structure, and transition to a disordered phase comprising liquid-like guest chains [2].

The 1D guest chains in Rb-IV are more than 7Å apart [3], and this raises the question as to the nature of the host-chain and chain-chain interactions in the ordered phase, and how this interaction weakens or vanishes at the transition to the disordered phase. The strength of the host-chain interaction can be inferred from the intensity of the *hklm* modulation reflections. Although these extremely weak reflections could not be detected with the powder techniques used previously to determine the structure of Rb-IV [3], they should be detectable using single crystal methods. In this talk I will present new results on the full modulated structure of Rb-IV using x-ray diffraction data collected from high quality single crystals of Rb-IV at ~18GPa.

[1] McMahon M.I., Nelmes R.J., *Z. Kristallogr.*, 2004, **219**, 742-748. [2] McMahon M.I., Nelmes R.J., *Phys. Rev. Lett*., 2004, **93** (5), 055501. [3] McMahon M.I., Rekhi S., Nelmes R.J., *Phys. Rev. Lett.*, 2001, **87** (5), 055501.
Keywords: rubidium, incommensurate composite structure, high-pressure crystallography

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Structural and Vibrational Studies of Solid Ammonia to 120 GPa
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