P.17.01.1
Acta Cryst. (2005). A**61**, C447
***In-situ* X-ray Analysis under Controlled Potential Conditions: An Innovative Setup and its Application to the Investigation of Ultrathin Films Electrodeposited on Ag(111)**
Francesca Loglio[a], Maria Luisa Foresti[a], Andrea Pozzi[a], Fabio D'Anca[b], Roberto Felici[b], Francesco Borgatti[b], Massimo Innocenti[a], Emanuele Salvietti[a], Anna Giusti[a], *[a]Department of Chemistry, University of Florence, via della lastruccia,3 50019 Sesto F.no Firenze. [b]OGG-INFM c/o ESRF, BP220, F-38043, Grenoble, France.* E-mail: logliof@unifi.it

An innovative setup to combine electrochemical and in-situ Surface X-ray Diffraction (SXRD) measurements is described. The electrochemical cell has a different design from the other ones commonly used in the beamlines. In particular, the cell arrangement allows the sample surface to stay completely immersed into the solution under controlled potential conditions even during the SXRD measurements. The X-ray beam goes through the liquid (about 1 cm) and the cell walls. However, because of the high X-ray energy, the beam attenuation is negligible and because of the detector arm slit positions, the diffuse scattering induced by the liquid is still low enough to detect the minima of the Crystal Truncation Rods (CTRs). The cell is connected to a special circuit for the alternate fluxing of the electrolyte solutions in the cell. The whole setup can be remotely controlled from outside the experimental hutch by a dedicated computer. The first measurements obtained on S films deposited at underpotential, and on CdS films of increasing thickness are reported.
Keywords: *in-situ* analysis, surface X-ray diffraction, electrochemical cell

P.17.02.1
Acta Cryst. (2005). A**61**, C447
Mixed Metal Oxide Nanoparticles – Synthesis and Characterisation
Paul Franklyn, David Jefferson, *Atomic Imaging Group, Department of Chemistry, University of Cambridge, Cambridge, England.* E-mail: pjf35@cam.ac.uk

Many naturally occurring mixed metal oxide materials are known, some of which have also been synthesized at high temperatures and pressures in laboratories. Several of these oxides offer potential applications as catalysts. Syntheses involving high temperatures and pressures are, however, detrimental to the efficiency of catalysts as such conditions lead to the formation of large particles, with a corresponding decrease in active surface area. Additionally, in purely scientific terms such mixed oxide species are interesting to study as the surface energetics of nanoparticles of mixed metal oxides may serve to stabilize phases and states that would not be stable in the bulk material.

In the work presented here mixed metal oxide nanoparticles containing titanium and tungsten were synthesized. The aim was to obtain materials that were new crystallographic forms containing tungsten and titanium ions within the same crystal matrix and not a solid solution of tungsten oxides and titanium oxides.

Nanoparticles were synthesised using the gel-polymer method as well as co-precipitation and were characterised using TEM, PXRD and XPS. The results indicate that the tungsten ions are located within the anatase matrix and have pronounced effects on the physical properties of the material.
Keywords: mixed oxides, nanoparticles, TEM X-ray structure determination

P.17.02.2
Acta Cryst. (2005). A**61**, C447
Fivefold Twinning of Diamond
Sam Ick Son[a], Su Jin Chung[b], *[a]CRD center of Samsung SDI, Yongin, 449-577, Korea. [b]New Materials Evaluation Center, Korea Research Institute of Standards and Science, Taejon, 305-600 Korea.* E-mail: samicks@hanmail.net

Among the low pressure diamonds grown by acetylene flame, various fivefold twinned particles in a form of pentagonal dipyramid, icosahedron or Kepler-Poinsot's hollow icosahedron were observed[1]. The relations between each twin domains were investigated by the electron back scatter diffraction (EBSD) method. The twin boundaries in the fivefold twins were observed by high voltage high resolution electron microscopy.

In the HRTEM image, one of the fivefold cyclic twin boundaries is different from the others. All four twin boundaries are coherent Ó 3 boundaries where as the other only one is Ó 81 boundary which consists of a series of edge dislocations to make up for the mismatching angles which arise after five successive cyclic twinning. There should be at least six Ó 81 boundaries in a twinned diamond icosahedron.

In the twinned Kepler-Poinsot's hollow icosahedron, the indented negative trigonal faces are formed from {100} faces of cube. The convex edges of the hollow icosahedron could be confirmed as Ó 3 boundaries while the concave edges as Ó 9 boundaries.

[1] Son S. I., Chung S. J., *Z. Krist.*, **219**, 2004, 494.
Keywords: fivefold twin, twin boundary, diamond

P.17.03.1
Acta Cryst. (2005). A**61**, C447
3D-visualization for Structure of Large CaF_2 by Step-scanning Section Topography
Taihei Mukaide[a], Takashi Noma[a], Kazuhiro Takada[a], Hidenosuke Ito[a], Kentaro Kajiwara[b], *[a]Leading-Edge Fusion Research Center, Canon INC. [b]SPring-8, JASRI.* E-mail: mukaide.taihei@canon.co.jp

The crystal defects affect solid state properties such as optical property. A fluorite (CaF_2) is an attractive material for the ultraviolet optics due to its high transparency for the short-wavelength light. Accounting for the relationship between the optical characteristics and crystal defects, it is important to know the distribution of the defects in the large crystal. In this study, we performed to measure internal structure of large size of fluorites single crystal block using white-beam X-ray topography.

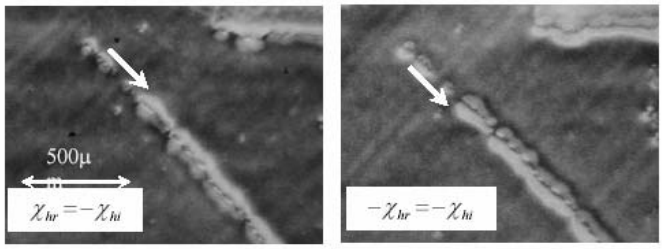
The experiments were performed at BL28B2 of SPring-8. The white X-ray beam from the bending magnet was shaped to the sheet-like beam of 30mm (horizontal) and 0.1mm (vertical) by the slits. The fluorite samples were grown by the Bridgman- Stockbarger method and cut into several sizes (for example 60mm of diameter and 60mm of thickness or 100mm of diameter and 40mm of thickness). The X-ray imaging detector was used to detect the diffracted X-rays from the sample. The section topographs were measured at intervals of 0.1mm in the vertical direction.

The three-dimensional images were reconstructed from the section topographs. Using this method, we can obtain the defect structure inside the large single crystals.
Keywords: X-ray topography, crystal defects, three-dimentional reconstruction

P.17.03.2
Acta Cryst. (2005). A**61**, C447-C448
X-ray Topography by using Resonant Scattering
Riichirou Negishi[a], Tomoe Fukamachi[a], Masami Yoshizawa[a], Shengming Zhou[b], Takaaki Kawamura[c], [a]*Saitama Institute of Technology*. [b]*Shanghai Institute of Optics & Fine Mechanics*. [c]*Univ. of Yamanashi*. E-mail: negishi@sit.ac.jp

Contrast variations of defect lines in X-ray topography are observed by changing X-ray energy very near the absorption edge of a crystal. X-rays from synchrotron radiation are used in the observation, which makes it possible to observe the X-ray topography using resonant scattering [1]. The variations of the defect contrasts using GaAs 220 reflection in the Laue case are observed by changing resonant condition near the K-absorption edges of both Ga and As. A typical example is as follows. A dark line defect is observed when the imaginary part of X-ray polarizability χ_{hi} is zero. The line becomes double and two lines with the same contrast are observed when the real part of X-ray polzarizability χ_{hr} is zero. The double line shows different contrast when $|\chi_{hr}|=|\chi_{hi}|$The bright and dark contrasts are

reversed by changing the phase of χ_{hr} and χ_{hi} as shown in the figure below. This clearly shows that such a change of contrast using resonant scattering should be quite useful to analyze characteristics of defects in a crystal.



[1] Negishi R., Yoshizawa M., Zhou S., Matsumoto I., Fukamachi T., Kawamura T., *J. Synchrotron Rad*., 2004, **11**, 266.

Keywords: X-ray topography, resonant scattering, defect contrast

P.17.03.3

Acta Cryst. (2005). A**61**, C448

Characterization of Dislocations in Protein Crystals using Synchrotron White-beam Topography

Kenichi Kojima[a], Haruhiko Koizumi[a], Miki Shimizu[a], Masaru Tachibana[a], Kentaro Kajiwara[b], Hiroshi Sugiyama[c], [a]*Graduate School of Integrated Science, Yokohama City University.* [b]*SPring-8/JASRI.* [c]*PF/KEK.* E-mail: kojima@yokohama-cu.ac.jp

To determine three-dimensional structure of protein molecules using X-ray diffraction method and neutron diffraction method, various protein crystals are grown. In particular, large protein crystals (~2 mm) are required for neutron diffraction method since the brilliance of neutron radiation is weak. Moreover, the characterization of crystal defects, especially dislocations, in protein crystals is important for an understanding of their crystallization. Therefore, it is important to establish synchrotron white-beam topography of protein crystals, which is one of the most powerful methods for characterization of dislocations in the large protein crystals. The application of X-ray topography to protein crystals has been carried out by some groups. However, the topographic contrasts observed in protein crystals were poor compared with those in organic crystals of small molecules reported previously. We found that the thickness of protein crystals should be more than 0.4ξ (ξ: the extinction distance) to observe the clear images. Thus, we have succeeded in observing clear topographic contrasts not only in tetragonal hen egg-white (HEW) lysozyme crystals [1] but also orthorhombic HEW lysozyme crystals using large protein crystals (~2 mm). These dislocations structures will be discussed at the conference.

[1] Tachibana M., Koizumi H., Izumi K., Kajiwara K., Kojima K., *J. Synchrotron Rad.*, 2003, **10**, 416.

Keywords: x- ray topography, protein crystals, dislocations

P.17.04.1

Acta Cryst. (2005). A**61**, C448

Quantitative X-ray Diffraction Study of Welded Joints in Heat-resistant Steels

Jorge L. Garin , Rodolfo L. Mannheim, *Department of Metallurgical Engineering, University of Santiago de Chile, Santiago, Chile.* E-mail: jgarin@lauca.usach.cl

Welding of cast heat-resistant steels have attracted much attention because of their interesting high-temperature applications in the metallurgical and mining industry. However, welded joints in service at elevated temperatures can yield precipitation of intermediate complex phases such as sigma, chi and carbides. In order to compare the behavior of the material with its microstructural features, a quantitative characterization of the weldments was carried out by means of X-ray diffraction. For this purpose Rietveld analysis were performed on a series of arc-welded joints of heat-resistant steels of the HC (25Cr-3Ni) and HD (30Cr-6Ni) type.

The Rietveld refinements were performed based upon typical measurement and global parameters. The powder diffraction patterns of the weldments resulted in strong preferred orientation effects due to the uniaxial solidification of the weld metal-pool, which was corrected in the Rietveld refinement by using the March-Dollase function. The pseudo-Voigt function was used for the simulation of the peak shapes, while the background was modeled by a 3rd order polynomial in 2θ with refinable coefficients.

A total of five phases were identified and considered in the refinemet process, namely ferrite (Cr,Ni), austenite (Ni,Cr), sigma phase, $Cr_{23}C_6$ and Cr_7C_3.

The main advantage of this processing was the use of the March-Dollase model for correction of the strong texture effects on the diffraction pattern of the weldments, which yield the lower R-values.

Keywords: Rietveld refinemet, welding , heat-resistant steels

P.17.04.2

Acta Cryst. (2005). A**61**, C448

Detection of weak X-ray Waves Scattered by the Crystal Subsurface Inclusions

Hakob P. Bezirganyan[a], Hayk H. Bezirganyan (Jr.)[b], Siranush E. Bezirganyan[c], Petros H. Bezirganyan (Jr.)[d], [a]*Department of Solid State Physics, Faculty of Physics, Yerevan State University, Yerevan, Armenia.* [b]*Faculty of Computer Science and Applied Mathematics, Yerevan State University, Yerevan, Armenia.* [c]*Department of Medical & Biological Physics, Yerevan State Medical University after Mkhitar Heratsi, Yerevan, Armenia.* [d]*Department of Computer Science, State Engineering University of Armenia, Yerevan, Armenia*. E-mail: hakob_bezirganyan@yahoo.co.uk

In presented theoretical paper a method is proposed appropriate for the non-destructive high-resolution investigations of the various kinds of non-diffracting subsurface nanosize inclusions based on the Grazing-Angle Incidence X-ray Backdiffraction (GIXB) technique [1, 2], which takes place in the conditions of specular vacuum wave suppression phenomenon [3]. Note that in the conditions of the reflected wave suppression mode [3] the specular wave (contrary to other existing X-ray diffraction methods) practically carries the information only about the non-diffracting subsurface inclusions.

Proposed method can be used to register relatively weak X-ray waves scattered by the non-diffracting subsurface inclusions or reflected by the surface regions, which aren't involved in backscatter diffraction process inside the thin crystalline film or the nanostructure.

[1] Bezirganyan H.P., Bezirganyan P.H., *Phys. Stat. Sol. (a)*, 1988, **105**, 345. [2] Bezirganyan H.P., *Phys. Stat. Sol. (a)*, 1988, **109**, 101. [3] Bezirganyan H.P., Bezirganyan H.H. (Jr.), Bezirganyan S.E., Bezirganyan P.H. (Jr.), *Opt. Comm.*, 2004, **238/1-3**, 13.

Keywords: inclusions in crystal, grazing incidence X-ray diffraction, X-ray backscatter diffraction

P.17.04.3

Acta Cryst. (2005). A**61**, C448-C449

Dislocations and Crystallite Size in Forsterite Produced at 11 GPa and 1400 ºC

Krisztián Nyilas[1], Hélène Couvy[2,3], Patrick Cordier[2,3], Tamás Ungár[1], [1]*Department of General Physics, Eötvös University Budapest, H-1518, POB. 32, Budapest, Hungary.* [2]*Bayerisches Geoinstitut, Universität Bayreuth, Germany.* [3]*Laboratoire de Structure et Propriétés de l'Etat Solide, ESA CNRS 8008, Université des Sciences et Technologies de Lille, Villeneuve d'Ascq, France*. E-mail: nyilas@metal.elte.hu

Synthetic forsterite is deformed at 11 GPa, 1400 °C in a multianvil high pressure apparatus at the Bayerisches Geoinstitut (Universität Bayreuth, Germany). X-ray diffraction patterns are measured by a special high resolution double crystal diffractometer with negligible instrumental effects. The monochromatised $K\alpha_1$ beam has a footprint on the specimen of 0.1x1 mm^2, enableing microbeam analysis. This condition provides diffraction patterns of the small specimens of the size of 0.2x2 mm^2. High resolution enables to carry out line profile analysis on the reflections well separated from those of platinum and corrundum unavoidable due to the small compact specimen structure. The dislocation densities are found to decrease with holding time at 1400 °C from about between $16x10^{14}$ m^{-2} to $0.04x10^{14}$ m^{-2}. Good correspondence of the dislocation structure determined by X-ray line