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