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***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)**
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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

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Mixed Metal Oxide Nanoparticles – Synthesis and Characterisation
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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

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Fivefold Twinning of Diamond
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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

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3D-visualization for Structure of Large CaF_2 by Step-scanning Section Topography
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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

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X-ray Topography by using Resonant Scattering
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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 200 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

Weinheim, Germany, 2002. [2] Boese R., Kirchner M. T., Billups W. E., Norman L. R., *Angew. Chem. Int. Ed.*, 2003, **42**, 1961.
Keywords: ionic liquid, cryo-crystallization, polymorphism

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Improved Crystal Detection of Protein Crystals by Bulk Contrast Enhancement
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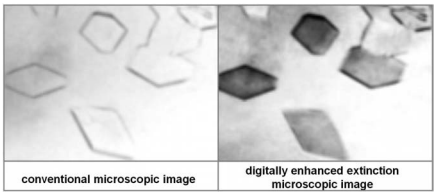
We present a computer-enhanced microscope that improves the reliable detection of small and colorless protein crystals in their native crystallization environment. Careful observation and evaluation of crystallization experiment pose a substantial burden on operator-based resources especially in high throughput crystallization operations. The presence of crystals is usually established by the observation of - frequently disguised - crystal facets, i.e. crystal edges in images. It is desirable to add a further contrasting method. Polarization microscopy does provide bulk color but this contrast is severely attenuated owing to the use of polymer-based birefringent crystallization trays.



We show how bulk contrast of micro crystals (grown in birefringent plastic trays and in lipidic cubic phase matrices) can be enhanced dramatically by digital processing of images that are captured with an automated extinction microscope (see figure). At first, images of crystallization experiments with different rotations of locked polarization extinction settings are captured. Then the colors are decomposed and numerical operations are applied on the respective grey-value matrices. The final combined false-color image shows protein crystals with enhanced bulk contrast. The configuration of the automated extinction microscope, its image processing algorithms and its usefulness for the detection of colorless protein crystals will be shown. We conclude that bulk contrast enhancement substantially aids the confident identification of crystals.
Keywords: crystal detection, image processing, lipidic cubic phase

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How a new Chemical Compatibility Test Facilitates Protein's Crystallization
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In early stages of a macromolecule's crystallization, when little information is known about a protein's solubility versus various chemicals, the selected strategy is to setup usual initial screens at protein concentration selected from past experience. Factors such as availability of protein or intrinsic protein physical properties can be used as guidelines, but again, they provide little help in selection of initial screens conditions.

During development of new optimization procedures and initial screens, we needed to find a new startegy which would address this question, and enable us to orient crystallization appropriately. Presented here is a new method to test a macromolecule's solubility against many chemicals which can be applied straightforwardly at experimental setup. Using this method, not only did we obtain a reasonable and necessary high level of precipitation in any selected initial screens, but results from this test can also be applied directly to optimization strategies like "Pro-Active" or "The Optimizer Series" presented earlier.

This strategy was applied to a series of 10 proteins, where solubility was tested against a series of salts, polymers, organics and buffers. From results obtained, initial screens and optimization methods were selected. This preliminary solubility evaluation, performed prior to crystallization setup, benefited not only initial screening results but also accelerated optimization process, using less protein compare to the classical optimization method.
Keywords: biomacromolecular crystallization, optimization, crystal growth apparatus design