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Our Hot Filament Chemical Vapor Deposition (HFCVD) apparatus, modified by the introduction of a powder flowing system, allows the use of different kinds of solid powders as reactants. By the use of $Fe(NO_3)_3$ in acetone as a catalyst, and carbon nanoparticles as reactants, we successfully synthesized Single Wall Carbon Nanotubes (SWNTs). We proved able to control orientation of the deposit as well as the area of growth [1]. By slightly varying experimental conditions, we obtained a nanocrystalline diamond coating on SWNT wall [2]. The use of diesel soot as SWNT precursor has been investigated, too: highly oriented growth of tubular structures through catalysed reaction occurs all over the substrate with abundance of coiled, intriguing structures. Commercial graphite with powders' size in the micron range, in the same experimental conditions, lead to Multi Wall Carbon Nanotube rich deposits. Selected area growth of nanotubes can be achieved by means of catalyst dispersions on Si/SiO_2 patterned substrates or from selectively sputtering metallic iron

[1] Orlanducci S., Sessa V., Terranova M. L., Rossi M., Manno D., *Chem. Phys. Lett.*, 2003, **367**, 109S. [2] Terranova M.L., Orlanducci S., A.Fiori, Tamburri E., Sessa V., Rossi M., Barnard A.S., *Chem Mater.*, *submitted*.

Keywords: nanotubes, nanostructures, chemical vapor deposition

P.16.10.1

Acta Cryst. (2005). A**61**, C444

"Latent" Phase Clusters (Kvatarons) as Growth Units

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In a number of our works (Askhabov, 1998-2004) we have described a set of ideas and principles dealing with structural organization of substance in the nanorange. This ideas have been collectively referred to as "kvataron concept". Central in this new concept is the idea that there are specific nanosize clusters, which we call kvatarons, arising under non-equilibrium conditions. Physically, kvatarons are pre-crystallization clusters of the transient ("latent") phase. It has been found that clusters more than ~1.2 nm in size can become potential centers of crystallization. Only such clusters contain a minimal number of atoms necessary for crystal nucleation. At the same time, crystals grow by smaller clusters (kvatarons), which are transformed to "two-dimensional" nuclei already on the growing crystal surface. We have proposed a new theory of crystal growth, were kvatarons are the basic growth units. Kvatarons are ideal as growth units. Chemical composition of kvatarons is the same as that of crystals. Topologically kvatarons are close to structural modules of crystals. Variability of the structure allows kvatarons to join any crystal surface. A growing crystal itself actively influence on kvataron crystallization on the crystal. The kvataron model accounts for all fundamental aspects of crystal growth: growth kinetics, surface evolution, defects formation, impurity entrapment, etc.

This work supported by Russian Foundation for the Basic Research, project no. 05-0565112.

Keywords: nucleation and crystal growth mechanism, clusters, kvatarons

P.16.10.2

Acta Cryst. (2005). A**61**, C444

Numerical Modeling of Interaction of Particles with Solidifying Crystals

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The thermal fields for the movement of a solidification interface towards a spherical particle was dynamically modeled for studying the deformation of the interface in relation with different thermal properties of the particle with the matrix solid-liquid. Finite element methods were employed in an axi-symmetric model of the system. Both particle and matrix were considered of similar densities and heat capacity. The convective force in the liquid phase was not considered. The results show that a concave interface is found when the particle has a larger thermal conductivity than the matrix, and a convex interface is found in the opposite case.

In addition, the drag force on a particle being pushed by a crystal was calculated with a fluid flow model. The force was calculated for different pushing configurations and the results compared with the values given by the modified Stokes equation [1]; which show that the model value are slightly larger than those the calculated with the equation. This difference predict an equilibrium separation for pushing lower than the computed by modified Stokes expression when a Lifshitz-Van der Waals model for the repulsion force [2] is used.

[1] Uhlmann M.A., Chalmers B., Jackson K.A. *J. Appl. Phys*., 1964, **35**, 2986. [2] Dzyaloshinskii E., Lifshitz E.M., Pitaeskii L.P., *Soviet Physics*, 1961, **73**, 153-176.

Keywords: computer simulation of solidification, melt growth, pushing

P.16.10.3

Acta Cryst. (2005). A**61**, C444

Crystal Growth and Morphology Prediction of Two Quinacridone Polymorphs

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Quinacridone – a widely used organic pigment – is known to have three polymorphs. Two of them, the beta-polymorph and the gamma-polymorph, grow as very thin platelets.

Vapour growth starting from both polymorphs was performed by vacuum sublimation in a specially constructed furnace. During characterization of the polymorphic phases by DSC recrystallization was observed. This led to the formation of beta-crystals. The new polymorph was formed via the vapour phase.

To explain the observed morphology of the crystals computer simulation of crystal growth was performed. Both the attachment energy model and kinetic Monte Carlo simulations were used to predict the crystal morphology starting from the crystal structure. The crystal structure of the gamma-polymorph is known from the Cambridge Structural Database and the crystal structure of the beta-polymorph was predicted using the Polymorph Predictor of Accelrys.

The attachment energy model based on the Hartman Perdok theory fails to predict the large aspect ratio of the platelet morphology for the polymorphs. The kinetic Monte Carlo simulations use the actual crystal structure and growth mechanism; they predict the morphology of both polymorphs successfully.

Keywords: polymorphism, crystal morphology, crystal growth computer modelling

P.16.10.4

Acta Cryst. (2005). A**61**, C444-C445

MONTY: An Algorithm for Predicting Growth Rates for any Crystal Structure in any Orientation

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A crystal growth simulation program, based on the Monte Carlo algorithm, is presented. The experimental crystal structure is input for the algorithm. It is modeled by a set of molecular interactions, which are obtained from molecular mechanics calculations. The mother phase is parameterized by its bulk thermodynamic properties. As a result, besides the growth rate, the microscopic surface structure can be studied under various growth conditions. [1].

Two examples are presented. The program was used to simulate the growth of two polymorphs of aspartame; the extreme aspect ratio of the experimental needle crystals is well-predicted [2]. The second example involves naphthalene. There it is shown, using the spiral growth option in the algorithm, that it is impossible to grow crystals of naphthalene at moderate supersaturations without the presence of screw dislocations. This is supported by using AFM [3].

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)**
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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

P.17.02.1
Acta Cryst. (2005). A**61**, C447
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

P.17.02.2
Acta Cryst. (2005). A**61**, C447
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

P.17.03.1
Acta Cryst. (2005). A**61**, C447
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

P.17.03.2
Acta Cryst. (2005). A**61**, C447-C448
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