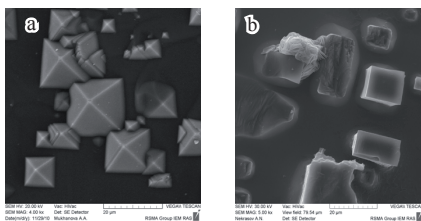


Poster Sessions

up to $1 \cdot 10^3$ nuclei in cm^3 . The aggregates are similar to the natural “diamondite” type and consist of microcrystals sized from 10-20 nm to 50-100 μm . Composition of the experimental growth carbonate melt-carbon solutions is chemically similar to the natural parent medium [2] that determines the physicochemical mechanism of the “carbonate-synthetic” diamond formation and, respectively, peculiarities of the face growth and morphology. By kinetic indication, it is possible to determine regions (1) of spontaneous nucleation and crystallization of diamond (“region of labile carbon oversaturation”) and (2) of diamond seeded growth (“region of metastable carbon oversaturation”) within the PT region of diamond thermodynamic stability. A boundary between the two regions is positioned of less than 0.5 GPa to the diamond-graphite equilibrium lines creating a narrow region of diamond seeded growth between the boundaries. Within the diamond-seeded growth region, morphological peculiarities of growing faces and crystallization steps and fronts are kinetically-dependent. The crystallization fronts are variable from polycentric and roughly blocked to smooth with nano-dimensional growth steps. The interaction of crystallization fronts can impose the effects of the layers overgrowth. All the new growing layers, independently of the seed face (111) with smooth layers or (100) with semi-octahedral hills, have the “octahedral” orientation for growth layers (fig. a). The “cubic” morphology demonstrated in the fig. b is firstly obtained; it is important that the orientation of the growing layers in the case is “cubic”. This uncommon result is obtained at highest temperature of 2400°C.



Support: Grants of RF President MK-913.2011.5, RAS Presidium P02, RFBR 10-05-00654 and 11-05-00401.

[1] A.V. Spivak, A. Litvin Yu., *Diamond Relat. Mater.* **2004**, *13*, 482-487. [2] A. Litvin Yu. In Ohtani, E., ed. *Advances in High-Pressure Mineralogy: Geological Society of America Special Paper* **2007**, *421*, 83-103.

Keywords: carbonate-synthetic diamond, morphology, seeded growth

MS35.P06

Acta Cryst. (2011) **A67**, C457

Synchrotron topography of (Nb,Yb):RbTiOPO₄ single crystals and related materials

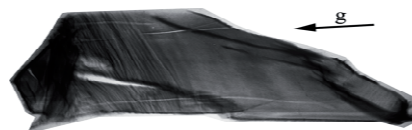
Joan J. Carvajal,^a Balaji Ragothamachar,^b Vishwanath Sarkar,^b Xavier Mateos,^a Michael Dudley,^b Francesc Diaz,^a Magdalena Aguiló,^a ^a*Physics and Crystallography of Materials and Nanomaterials (FiCMA-FiCNA) and EMaS, University Rovira i Virgili, Tarragona (Spain).* ^b*Stony Brook Synchrotron Topography Facility, Department of Materials Science & Engineering, Stony Brook University, Stony Brook, NY (USA).* E-mail: joanjospe.carvajal@urv.cat

Rubidium titanyl phosphate, RbTiOPO₄ (RTP) is isostructural with the well-known potassium titanyl phosphate, KTiOPO₄ (orthorhombic, Pna2₁). Its non-linear optical properties make it suitable as a frequency doubling material when doped with active lanthanide ions such as Yb³⁺ [1]. However, the distribution coefficient of Yb³⁺ in RTP crystals is very low and the codoping with Nb⁵⁺ is required to increase the concentration of Yb³⁺ in the crystals through a mechanism of electrical

compensation of the matrix [2]. Laser action has been demonstrated at $\sim 1 \mu\text{m}$ for these crystals [3]. The (Nb,Yb):RTP crystals are grown by the top-seeded solution growth (TSSG) technique using self-fluxes to avoid the presence of undesired ions in the solution that can affect the physical properties of the crystals. However, the crystals exhibit a tendency to crack during removal from the furnace after growth. Such cracked crystals are obviously not suitable for optical and spectroscopic applications.

Synchrotron white beam X-ray topography (SWBXT) has been employed to characterize the structural defects in these crystals to investigate the cause of cracks. X-ray topographs recorded from both doped and undoped crystals reveal defect features such as growth striations, growth sector boundaries, dislocations, grain boundaries, and especially, inhomogeneous strain that might be responsible of cracking. The distribution of these defects have been mapped in each type of crystal along the three main crystallographic directions and compared. Defect distribution was also analyzed with respect to the growth conditions in order to gain insights into the growth mechanism and the origin of cracks. Results from these studies will be presented.

[1] J.J. Carvajal, V. Nikolov, R. Solé, Jna. Gavalda, J. Massons, M. Aguiló, F. Diaz, *Chem. Mater.* **2002**, *14*, 3136-3142. [2] J.J. Carvajal, J.L. Garcia-Muñoz, R. Solé, Jna. Gavalda, J. Massons, X. Solans, F. Diaz, M. Aguiló, *Chem. Mater.* **2003**, *15*, 2338-2345. [3] X. Mateos, V. Petrov, A. Peña, J.J. Carvajal, M. Aguiló, F. Diaz, P. Segonds, B. Boulanger, *Opt. Lett.* **2007**, *32*, 1929-1931.



Keywords: synchrotron white beam X-ray topography, top-seeded solution growth, RbTiOPO₄

MS35.P07

Acta Cryst. (2011) **A67**, C457-C458

The CrystalHarp™ - an advanced high throughput capillary plate for protein crystallization

Mareike Kurz,^a Beat Blattmann,^a Christophe Briand,^a Paul Reardon^b Andres Käch,^c and Markus G. Grütter,^a ^a*Institute of Biochemistry, University of Zürich, Zürich (Switzerland).* ^b*SWISSCI AG, Neuheim, (Switzerland).* ^c*Center for Microscopy and Image Analysis, University of Zürich, Winterthurerstrasse, Zürich (Switzerland).* E-mail: m.kurz@bioc.uzh.ch

Counter diffusion crystallization in capillary is an easy, cost-effective and practical procedure for obtaining protein crystals for *in situ* X-ray data analysis. This method is used simultaneously for screening, incorporation of heavy atoms and cryo protection in a single capillary [1], [2].

We present a novel capillary crystallization plate, based on the counter-diffusion method and designed for 48 high throughput-screening experiments. Due to the plate's design only 20 μl of protein is needed to load the plate very quickly. A liquid handling robot can facilitate the pipetting of the individual well conditions. The crystallization plate is compatible with any incubation and imaging systems and allows *in situ* X-ray diffraction measurements. Alternatively, an individual capillary can be removed from the CrystalHarp™ and the crystal can be analysed 360° *in situ* by X-ray diffraction.

For initial plate validation, crystallization experiments with six commercially available soluble proteins, two soluble in-house proteins

and two in-house membrane proteins were performed. In all cases crystals could be successfully grown based on known vapour-diffusion conditions. In one case the CrystalHarp™ was also successfully used for screening new crystallization conditions.

As a proof of principle, diffraction studies on all ten proteins crystallized in the CrystalHarp™ were undertaken at the SLS. We can show, that crystals grown in capillary diffract to a similar resolution as the ones grown by vapour diffusion. So far, for three proteins (lysozyme, insulin and the membrane protein AcrB), complete datasets were collected and the structures solved by molecular replacement [3], [4].

Additionally, we used the CrystalHarp™ for initial high pressure freezing experiments to circumvent the cost-intensive and tedious process of searching for the correct cryo protectant [5]. As a proof of principle lysozyme crystals were used. Crystals were both, conventionally snap-frozen in liquid nitrogen and under high pressure of 200 MPa. We find, that crystals frozen in the conventional way without the addition of cryo protectant get damaged during the freezing process, resulting in ice ring formation and loss of resolution. In comparison, crystals grown in the exact same condition but frozen under high pressure are clearly not damaged and also results in the abolishment of ice rings in the diffraction pattern.

[1] J.M. Garcia-Ruiz, *Methods in Enzymology*, **1997**, 368. [2] J.D. Ng *et al.*, *J Struct Biol*, **2003**, 142(1), 218-231. [3] M. Cianci *et al.*, *Acta Crystallogr D*, **2008**, 64. [4] K.M. Pos *et al.*, *FEBS Lett.* **2004**, 30, 564(3). [5] C.U. Kim *et al.*, *Acta Cryst D*, **2005**, D61, 881-890.

Keywords: high-throughput counter diffusion, in situ diffraction, high pressure freezing

MS35.P08

Acta Cryst. (2011) A67, C458

A three-polymorph system with k2- and t2-types of phase transformations

Ilija A. Guzei,^a Erica M. Gunn,^b ^a*Department of Chemistry, University of Wisconsin-Madison, Madison, (USA)*. ^b*School of Pharmacy, University of Wisconsin-Madison, Madison, (USA)*. E-mail: iguzei@chem.wisc.edu

5-(Pyridin-2-ylmethylene)-3-phenyl-2-methylthio-3,5-dihydro-4H-imidazole-4-one (1) exists in three polymorphic forms. Upon cooling from room temperature the orthorhombic Form I of 1 undergoes an enantiotropic k2 transition to the orthorhombic Form II at 262.9(5) K. A molten 1 can be cooled in a controlled fashion to generate a monoclinic Form III. Form III, once isolated, is indefinitely stable between 100 K and its melting point of 466 K. If crystals of Form III are in contact with seed crystals of Form I, a first-order Form III→Form I solid state phase transition of type t2 occurs upon heating with the onset between 420-448 K. The differences in the molecular geometries of the three polymorphs are small, but molecular packing is considerably different in the monoclinic lattice. Powder pattern similarity coefficients between the polymorphic pairs are interpreted to highlight the differences between the phases. The presentation describes the discovery of Forms II and III and the detailed study of the three polymorphs. Videos of a controlled single crystal growth from the melt and solid state phase transition Form III→Form I will be demonstrated.

Keywords: polymorphism, phase transition, melt

MS35.P09

Acta Cryst. (2011) A67, C458

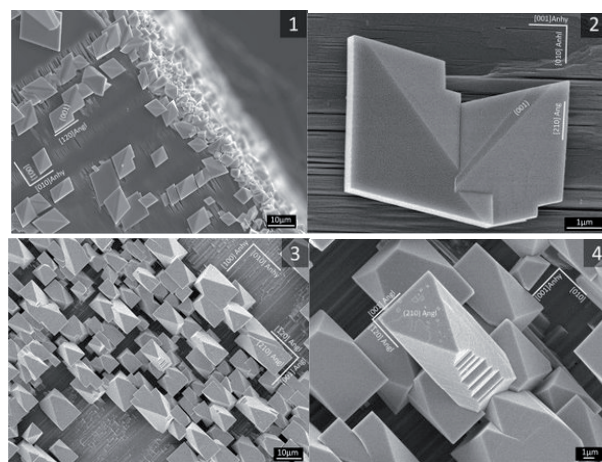
Epitaxial growth of Anglesite (PbSO₄) on anhydrite (CaSO₄) cleavage surfaces

Lurdes Fernández-Díaz,^{a,b} Juan Morales Sánchez-Migallón,^a José Manuel Astilleros,^{a,b} Amalia Jiménez,^c ^a*Departamento de Cristalografía y Mineralogía, Universidad Complutense de Madrid, Madrid (Spain)*. ^b*Instituto de Geociencias (UCM-CSIC). Facultad de CC. Geológicas, Madrid (Spain)*. ^c*Departamento de Geología. Universidad de Oviedo, Oviedo (Spain)*. E-mail: lfdiaz@geo.ucm.es

The interaction of CaSO₄ minerals with Pb-bearing aqueous solutions is an effective mean to remove this pollutant from the fluid as a result of the precipitation of anglesite (PbSO₄) [1]. Generally speaking, the ability of mineral surfaces to remove toxic metals through the formation of sorbates, that immobilize these pollutants in their structure, is strongly affected by the spatial arrangement and the crystallographic relationships between sorbant and sorbate. Gypsum (CaSO₄·2H₂O) crystals in contact with Pb-bearing solutions undergo partial dissolution, which is shortly followed by the precipitation of anglesite crystals randomly distributed on gypsum surfaces. Similar dissolution-precipitation processes occur during the interaction of anhydrite (CaSO₄) with aqueous solutions containing Pb. However, in this case anglesite crystals grow oriented on anhydrite surface. In this work we investigate the crystallographic relations between anglesite crystals and two of the main anhydrite cleavage surfaces, (100), and (001), as observed during the anhydrite dissolution-anglesite precipitation process.

On anhydrite (100) surface anglesite crystals dispose their (001) face parallel to the substrate, with $[010]_{\text{anhy}} \parallel \langle 120 \rangle_{\text{angl}}$. In the case of anhydrite (001) surface the matching occurs with anglesite (210) plane, with $[100]_{\text{anhy}}, [010]_{\text{anhy}} \parallel \langle 001 \rangle_{\text{angl}}, \langle 120 \rangle_{\text{angl}}$.

Figures 1 to 4 show anglesite crystals growing on anhydrite (100) and (001) surface in four orientations. These orientations are related to each other by symmetry elements that exist in the anhydrite planes, but are absent in the matching anglesite planes. The coalescence of crystals differently oriented lead to the formation of twins. This type of twinning has been envisaged as a type of transformation twinning that specifically occurs during solvent mediated transformations [2].



[1] J.M. Astilleros, A. Godelitsas, J.D. Rodríguez-Blanco, L. Fernández-Díaz, M. Prieto, A. Lagoyannis, S. Harissopulos, *Applied Geochemistry* **2010**, 25, 1008-1016. [2] A. Pinto, A Jiménez, M. Prieto. *American Mineralogist* **2009**, 94, 313-322.

Keywords: anhydrite, anglesite, epitaxial growth