

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.

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

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

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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

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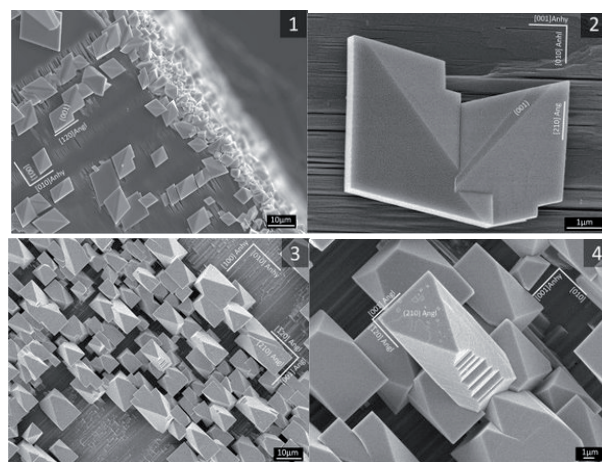
Epitaxial growth of Anglesite (PbSO₄) on anhydrite (CaSO₄) cleavage surfaces

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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].



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