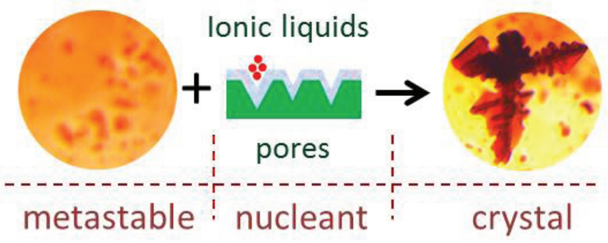

Figure 1. Mineral particles with porous surface and ionic liquid coating induce protein crystallization in a metastable zone.

Keywords: solid-supported crystallization, crystallization from metastable zone, heterogeneous nucleant

MS13. New instrumentation, methods and approaches in inorganic crystallography

Chairs: Anton Meden, Damien Jacob

MS13-O1 Complex metal hydrides: When powder diffraction needs help

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'Real life' energy-related materials such as solid-state hydrogen storage compounds or components of electrochemical cells are usually polycrystalline, poorly crystallized, highly reactive and dynamic systems. Powder diffraction at modern high brilliance X-ray sources is the most useful tool to investigate such systems because it is easy, fast and extremely versatile with respect to measurement conditions as well as in situ setups. However, powder diffraction applied to these systems rapidly reaches its limits due to the bad crystallinity of samples prepared by mechanochemistry as well as due to the method itself. We will show how a complementary approach combining powder diffraction with non-diffraction methods such as vibrational spectroscopy, thermal analysis and supported by ab initio solid state calculations allows overcoming these limitations[1].

The investigation of samples containing several novel crystalline phases of uncertain chemical composition appears as a problem with no unique solution. We will show that a purposeful use of various samples of different nominal compositions, powder diffraction patterns measured while thermally decomposing each phase (T-ramping)[2], direct space methods for structure solution and refinement by ab initio solid state calculations does provide detailed structural information of novel compounds even for hydrogen atoms[3].

The recently developed possibility of performing mechanochemistry, usually a "black box" approach, on reactive samples under the beam will also be discussed shortly. Ball milling mapped by in situ powder diffraction provides a means of "watching" the synthesis of novel materials, and it is the latest tool we have applied to follow the reactions of complex hydrides inside the reaction jars, capturing intermediate phases and tuning the milling conditions to selectively favour phase yields[3].

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Keywords: powder diffraction, metal hydride

MS13-O2 Decomposing electron diffraction signals from multi-component microstructures

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Recent development of spatially resolved (scanning) diffraction in the TEM offers the capacity to study phase and crystallographic orientation with nanometre spatial resolution [1]. However despite the relatively small sample thickness in TEM there is still the possibility of two or more distinct phases contributing to a diffraction pattern in the dataset. One solution is to use multivariate statistical analysis [2] approaches to separate out the unique diffraction signals (or components) from the different phases in the system. This can provide useful information about the general diffraction signal from a phase by removing the small variations in diffraction patterns arising from local variations in the sample thickness or bending, allowing a more robust pattern match for regions of the same structure and orientation. It can also provide a robust method for localising phases within a scanned area allowing completely embedded phases to be separated from within the scanned diffraction data.

This approach will be shown through a number of different studies, starting with the analysis of twinning and epitaxial growth in semiconductor nanowires and continuing through to the separation of phases in partially oxidised zirconium, leading to confirmation of the existence of a proposed intermediate oxide [3] structure found at the metal-metal oxide interface.

The onward development of this approach is to isolate and track individual phases through a tilt-series of measurements, allowing tomographic reconstruction of the individual phases combined with understanding of the complete 3-D orientation relationships within the microstructure. This will be shown through a study on a nickel superalloy sample highlighting the coherent interface between matrix and precipitates within the microstructure.

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Keywords: Scanning electron diffraction, principle component analysis, orientation tomography