

**Session II, Use of synchrotron radiation, Wednesday, November 25****L5****X-RAY DIFFRACTION STRUCTURAL BIOLOGY USING SYNCHROTRON RADIATION****Uwe Müller***Helmholtz-Zentrum Berlin für Materialien und Energie, Albert-Einstein-Str. 15, 12489-Berlin, Germany*

Over the past decades, X-ray diffraction of biological samples, also called macromolecular crystallography (MX) has been developed to the most powerful method to gain insights into the 3D structural space of biological macromolecules, like protein, protein-protein and other complexes. One key for these advances is the use of highly

brilliant X-rays produced by electron storage rings to investigate crystals of large unit cells or micrometer sized crystals.

Within this presentation, an overview of this technique and its current applications are provided.

L6**A SHORT OVERVIEW OF TOMOGRAPHIC IMAGING CAPABILITIES AT SYNCHROTRONS IN EUROPE AND ELSEWHERE****R. Mokso***Department of Solid Mechanics, Lund University, Ole Romers vag 1, Lund, Sweden
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Tomographic imaging aims to map the 3D electron density in a non-destructive manner. The method is relevant to study materials of almost all types including biological systems. State-of-the-art X-ray imaging instruments are used to create virtual representations of materials down to tens of nanometre spatial resolution. Perhaps the biggest asset of synchrotron imaging instruments is their excellent temporal resolution at this length scale [1]. The limits in the temporal domain are mainly set by the detector and the me-

chanics. For the spatial resolution the biggest concern is the radiation dose resulting from the interaction of X-ray radiation with the studied matter. I will give an overview of recent achievements in micro- and nano-scale tomographic imaging with emphasis on the specific capabilities of imaging beamlines in Europe.

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L7**APPLICATION OF PAIR DISTRIBUTION FUNCTION FOR STUDY OF HIGHLY-DISORDERED COMPOUNDS****Jozef Bednarčík***P.J. Šafárik University in Košice, Institute of Physics., Park Angelinum 9, 041 54 Košice, Slovakia
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Compared with crystalline counterparts, metallic glasses (MGs) have some superior properties, such as high yield strength, hardness, large elastic limit, high fracture toughness and corrosion resistance, and hence are considered as promising engineering materials. Fe- and Co-based amorphous alloys have been the subject of considerable research interest and activities for the last decades due to applications related to their outstanding soft magnetic properties. Structurally, metallic glasses can be classified as disordered materials. X-ray diffraction (XRD) using high-energy photons has proven to be well suited for describing the structure of highly disordered systems such as MGs.

Time-resolved in situ XRD experiments may nowadays be performed at high-brilliance synchrotron radiation sources for a variety of conditions which help to elucidate the structure-property relations.

In this contribution structural changes occurring in an $\text{Fe}_{72.5}\text{Cu}_1\text{Nb}_2\text{Mo}_2\text{Si}_{15.5}\text{B}_7$ alloy during a combination of constant rate heating (20 K/min) and isothermal holding at 500 and 520 °C will be investigated using in situ high-energy X-ray diffraction. It was found that the ferromagnetic-to-paramagnetic transition of the amorphous phase is revealed as a change in the slope of the thermal expansion curve when heating a sample at a constant rate up to 520



C. Real space analysis by means of the atomic pair distribution function (PDF) demonstrated that the rate and extent of the thermal expansion strongly depend on the interatomic separation. The PDF proved to be a reliable method for the description of crystallization kinetics. Further it allows determination of sizes of ultrafine nanocrystals with grain sizes well below 8 nm and thus makes

observation of early stages of nanocrystallization possible. This contribution presents results showing how pair distribution function can be successfully used for tracking the ferromagnetic-to-paramagnetic transition of amorphous phase in the vicinity of the Curie point.

L8

IN-SITU X-RAY SCATTERING STUDY OF PULSED-LASER DEPOSITION OF LuFeO₃/Pt SYSTEMS

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Growth of multiferroic epitaxial layers of hexagonal LuFeO₃ has been studied by X-ray scattering in-situ during pulsed-laser deposition (PLD). For this purpose we used a unique PLD chamber attached to the NANO-beamline of the Institute of Photon Science and Synchrotron Radiation (IPS) attached to the KARA storage ring (Karlsruhe, Germany). The LuFeO₃ layers have been deposited on single-crystalline sapphire substrates, in some samples we also deposited thin Pt interlayers prior to the LuFeO₃ growth. In the first part of the talk I report on grazing-incidence small-angle scattering (GISAXS) measured in-situ during the Pt deposition. From the data we determined the Pt growth kinetics, in particular we study the dependence on the island-to-layer percolation threshold on the substrate temperature [1]. In the second part I discuss the parameters of the LuFeO₃ thin layers and their development during deposition [2]. The layers are mosaic and we studied the angular mosaic spread and mean size of the mosaic blocks as functions of the substrate deposition temperature and of the Pt thickness underneath. The final part of the talk is devoted to the study of growth oscillations during the LuFeO₃ deposition; the oscillations were recorded by mea-

suring the time dependence of the intensity of the quasi-forbidden reflex (0003) of LuFeO₃. From the data we determined the growth rate and the height of the Ehrlich-Schwöbel diffusion barrier as functions of the laser repetition frequency [3].

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L9

FUTURE DATA ANALYSIS SERVICES AT EUROPEAN AND NATIONAL PHOTON AND NEUTRON FACILITIES

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The most powerful X-ray laser facilities as well as multiple 4th generation synchrotron light sources are in user operation across the Europe. Preparation for the European spallation source is ramping up, more photon and neutron

(PaN) facilities are planning upgrades. With the excellent brightness, larger and faster detectors enormous volumes of scientific data are produced. The variety of experiments as well as PaN user communities are broadening, publish-

ing practices and academic institutions requirements for scientific data management, including interest in open-data, are increasing. In late 2018, within the European Open Science Cloud (EOSC) initiative, several major European PaN facilities started a project called PaNOSC [1], which was complemented by ExPaNDS [2] project at the national research PaN institutes a one year later. Both projects aim for expanding scientific data catalogues and analysis services in order to make scientific data at PaN facilities comply with the FAIR data principles. This includes adjustments to scientific data policies, extension of scientific data retention period, tools to search for datasets of possible scientific or scholar interest, improvements of data accessibility, data formats and metadata catalogues and finally a possibility to reproduce the scientific results by means of remote data analysis services. Within the wide scope of the projects several application use cases have been chosen to prototype all the services including data analysis. The selected scientific use cases cover multiple methods including crystallography, for the ExPaNDS project in particular serial crystallography [3], CryoEM and powder diffraction [4], but also other techniques as small angle scattering, reflectometry or ptychographic X-ray computed tomography [5]. The use cases represent

several types of PaN sciences analysis workflows including Python Jupyter notebooks, conventional high-performance distributed computing, cloud-like containerization for data-science and remote desktops for visualization. The idea is to match the environments to run scientific software with archived datasets and records in metadata catalogues. The projects outcomes include definition of application interfaces and a functional prototype that can be deployed at research facilities, can be interconnected with other tools, developed and extended in a sustainable way, allowing to bridge more scientific data into EOSC.

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L10

PHASE TRANSFORMATIONS IN Ti-15Mo SINGLE CRYSTAL INVESTIGATED BY SYNCHROTRON X-RAY DIFFRACTION

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In metastable titanium alloys, the transformation from the high-temperature phase (bcc) to the low-temperature phase (hcp) can be suppressed upon quenching to room temperature [1]. Due to the thermodynamically metastable nature of the phase, particles of the so-called phase form in the material by a diffusionless shuffle mechanism already during quenching. The crystallographic structure of phase is either trigonal or hexagonal, depending on the completeness of the transformation, and there are four crystallographic orientations (variants) of the phase [2] particles are coherent with the parent phase, their size ranges from a few nanometres [3] to a few tens of nanometres and they were found to be spatially weakly ordered in a cubic array [4]. With increasing temperature, particles evolve and grow by a diffusion-assisted process until they reach a stability limit at which the precipitation of phase becomes the dominant transformation.

The main goal of this research is to investigate the sequence of transitions occurring in single-crystals of Ti-15Mo (in wt.%) alloy during linear heating by synchrotron X-ray diffraction. For this purpose, a single crystal was prepared using the floating zone method [5], solution treated above the β -transus temperature and quenched to water to produce a well-defined starting condition for the heating experiments.

X-ray diffraction was measured at the high-energy beamline ID11 at ESRF, Grenoble. The photon energy was 60 keV and the primary beam was parallel to [100] direction in the studied crystal. The heating was performed in a dedicated furnace equipped with a quartz chamber, allowing to reach high vacuum (approx. 10^{-6} mbar). The diffracted signal was collected in situ during linear heating with the rate of 5°C/min. An example of a measured diffraction pattern together with calculated pattern explaining the origin of individual peaks is displayed in Fig. 1.

The set of measured data allowed us to determine the sequence of phase transformations by following the evolution in regions around selected diffraction spots during linear heating, see Fig. 2. The intensity in these regions first decreases with increasing temperature, but the reflections do not disappear completely. On the contrary, around 673 K (400 °C), the peaks sharpen and their intensity increases – this is most visible in Fig. 2 c) which shows an spot closest to the Ewald sphere (Figs. 2 a) and b) display peaks further from the Ewald sphere, so the intensity increase is not readily apparent due to their sharpening). At 833 K (560 °C, denoted by a black vertical line in Fig. 2), all reflections disappear, suggesting a complete dissolution of the phase. The intensity observed at the highest measured temperatures corresponds to phase diffraction spots which lie close to the original reflections.

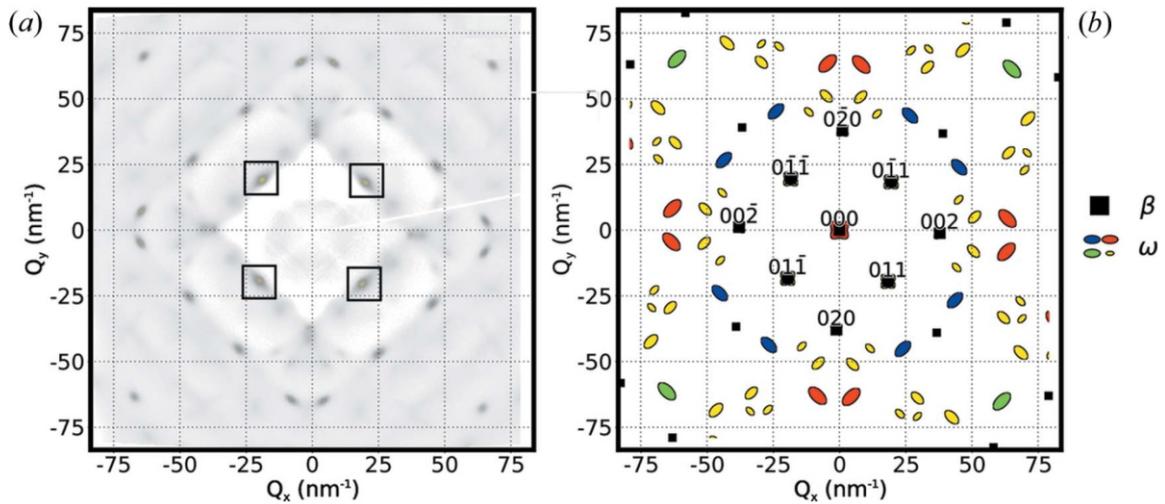


Figure 1. a) Measured diffraction pattern at room temperature and b) calculated pattern showing the positions of observed and diffraction spots (square and ellipse symbols, respectively).

Numerical fitting of measured diffraction peaks allowed us to estimate the temperature dependence of phase fraction (red points in Fig. 3) and interface area between the matrix and particles (blue points in Fig. 3). Both quantities depend linearly on the number of irradiated particles; the steeper decrease of S/S_0 above 523 K (250 °C) suggests that while particles grow, their number decreases (a process similar to Ostwald ripening). The increasing trend observed in Fig. 3 is caused by progressive coarsening of particles. As the temperature nears the stability limit of the phase at 833 K (560 °C), both the phase fraction and the interface area decrease rapidly. More information on this topic and a comparison with complementary data from electrical resistivity measurements can be found in [6].

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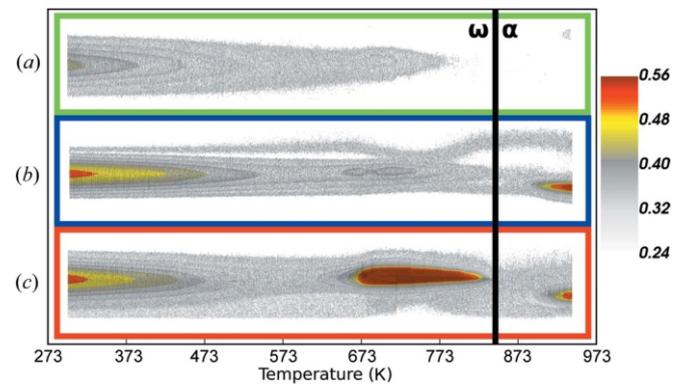


Figure 2. Evolution of a), b) and c) diffraction spots.

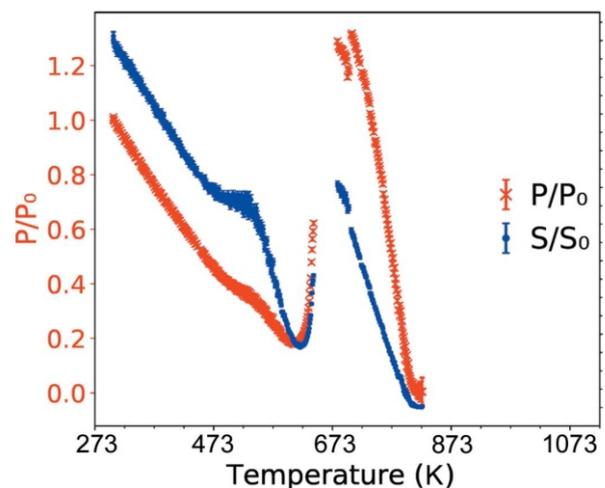


Figure 3. Evolution of phase fraction of the phase (red points) and the interface area (blue points).

L11

TEMPERATURE INDUCED STRUCTURAL EVOLUTION OF POROUS IMIDAZOLATE COMPOUNDS VIA SYNCHROTRON XRPD

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Zeolitic imidazolate frameworks (ZIFs) are an interesting class of metal-organic frameworks, structured by tetrahedrally configured transition metal cations bridged by imidazolate (Im). ZIFs are able to reproduce the zeolitic topology but also incorporate the electronic properties of the transition metal ions.[1]

In this research, novel high-temperature polymorph of sodium imidazolate, HT-NaIm was discovered. Solid-state NMR was used for initial elucidation of structural features, the crystal structure was determined by single-crystal X-ray diffraction, while the *in-situ* HT-XRPD experiments utilizing synchrotron radiation have been performed in order to gain the insight into the structural evolution and thermal stability which was additionally analyzed by differential thermal analysis and hot stage microscopy measurements. HT-NaIm exhibits pores of 50 Å³ that suggest possible application for gas sorption/separation. Once formed, high-temperature polymorph of NaIm retains its structure and remains stable at room temperature, what is important application-wise.

Additionally, new family of mixed bimetallic imidazolates AMIm₃ (A = Na, K; M = Mg, Mn) has been

synthesized and crystal structures were determined from powder X-ray diffraction data. Temperature aided decomposition during *in-situ* SR HT-XRPD experiments gave the information about structural changes and thermal stability of the prepared samples. All compounds have the imidazolate ligand connected to four metal cations forming a complex 3D network with channels running along the *c*-direction, thus showing the similar sorption potential because of the empty volume of around 30 C³ incorporated inside the channels (Figure 1).[2]

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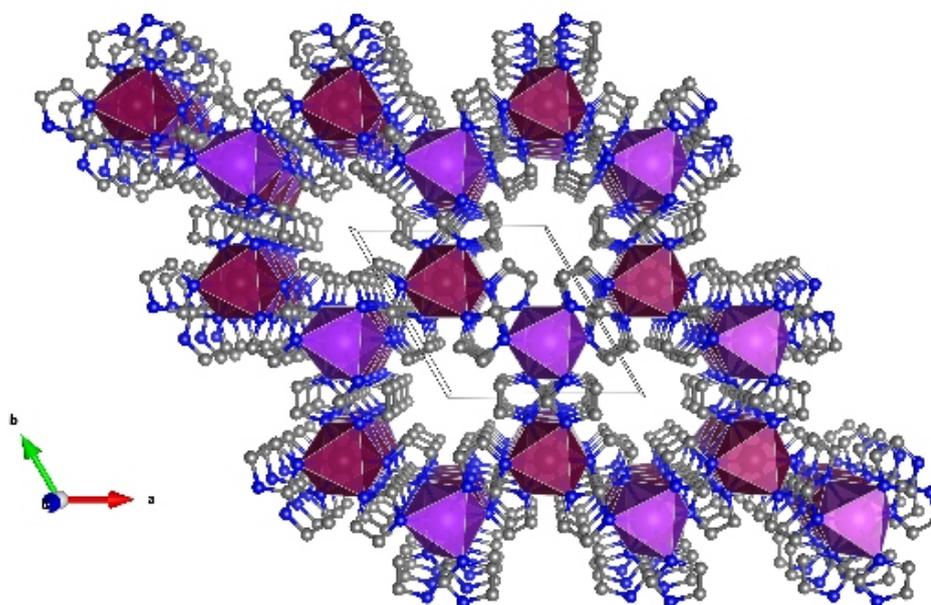


Figure 1. Extended crystal packing of KMgIm₃ showing channels along the *c*-direction.



L12

ESRF AND THE CZECH MEMBERSHIP

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The European Synchrotron Radiation Facility (ESRF) is the most powerful synchrotron source in Europe. The ESRF is one of the world's largest synchrotron science centres. Every year, 9000 scientists from 22 partner countries and from around the world travel to Grenoble to use its extremely brilliant X-rays for leading-edge research. The ESRF Upgrade Programme 2009-2018 maintains the world-leading role through a continuous quest for higher performance figures, meeting the needs of returning users, and attracting scientists from new disciplines [1]. ESRF-EBS (Extremely Brilliant Source) is the ESRF's 150M€ facility upgrade, over 2015-2022, bringing its scientific users a first-of-a-kind, low-emittance, high-energy

synchrotron light source and new, cutting-edge beamlines. With a revolutionary new storage ring concept that increases the brilliance and coherence of the X-ray beams produced by a factor of 100 compared to present-day light sources, ESRF-EBS represents a new generation of synchrotron and an extraordinary new tool for scientists to study the heart of matter.

The Czech republic is a member state of the ESRF, enabling Czech scientist to apply for beamtime. Our membership is financed through the MŠMT program Inter-Excellence and is secured for the period between 2018-2022.

1. www.esrf.eu