Session XI

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X-RAY LABORATORY AT THE INSTITUTE OF MACROMOLECULAR CHEMISTRY CAS: INSTRUMENTAL EQUIPMENT AND RECENT PROJECTS

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The laboratory of X-ray and Neutron Structural Analysis at the Institute of Macromolecular Chemistry of the Czech Academy of Sciences (currently part of the department of NMR Spectroscopy & X-ray and Neutron Diffraction) was founded in 1965 with a primary focus on studying the structure of polymers in different forms. By utilizing X-ray diffraction, we can investigate not only the crystal structure of the polymer, but also the amorphous regions, providing valuable information about the crystallinity of the sample, crystallite sizes and orientation of the polymer chains, which are related to the mechanical properties and thermal behaviour of the polymer. This technique can also be used to observe phase transitions of block copolymers in selective solvents, even *in-situ*.

To perform these experiments, our laboratory is equipped with two instruments – the GNR EXPLORER powder XRD and a custom SAXS instrument originally built by MolMet and later upgraded by SAXSLAB. The GNR EXPLORER can also be used for XRR measurements, making it possible to determine thickness, density, and surface roughness of thin films. Our SAXS instrument can cover a wide q-range (from 0.005 to 3.6 $Å^{-1}$) thanks to adjustable sample-to-detector distance and includes a sample stage for measurements at non-ambient temperatures. It is also capable of GISAXS measurements, which are useful for investigating the morphology and ordering of polymer domains within thin films.

Some of our notable recent projects include an XRD study of polypyrrole - barium ferrite magnetic cryogels for water purification [1], an XRD study of PEDOT / maghemite adsorbent for the removal of Reactive Black 5 from aqueous media [2], or a GISAXS study of block copolymer thin films with added magnetic nanoparticles for high-resolution lithography technologies [3]. Our laboratory also cooperates in longer-running research at the IMC such as special purpose polymers (e.g. for drug-delivery systems [4]), polymers with special optical and mechanical properties (e.g. tough and transparent elastomers [5]), polymer-based membranes for solid electrolytes [6], or biodegradable polyurethane foams [7].

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"ORDINARY DAY OF A POWDER DIFFRACTIONIST"

(X-ray powder diffraction at the Institute of Inorganic Chemistry of the CAS in Řež)

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X-ray powder diffraction (XRPD) is an indispensable technique for any chemist, mineralogist and any other scientist or engineer. The laboratory of XRPD exist in the Institute of Inorganic Chemistry of the Czech Academy of Sciences for more than 25 years. This talk will introduce this laboratory and its instrumentation.

Few case studies will demonstrate the everyday activities of the Laboratory, some hints for performing the XRPD analyses, preparation of samples and measurement techniques, among them those connected with projects addressed to the Institute, e.g. projects in the ALMA Laboratory and the NanoEnviCz infrastructure.

Last but not least, some unusual samples passed through our systems will be also presented.

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BRAGG COHERENT DIFFRACTION IMAGING OF NANOPARTICLES PREPARED BY GAS-PHASE AGGREGATION

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In presented work, silver and hafnium metallic nanoparticles prepared by the aggregation from the gas phase were studied by Bragg coherent diffraction imaging.

Bragg coherent diffraction imaging (BCDI) is a lensless technique that enables the reconstruction of the nanocrystal in 3D with high sensitivity to the presence of microstrains in the crystal structure. It is based on the scattering of the coherent beam from monocrystalline nanoparticle and the measurement of a series of 2D reciprocal space cuts around the Bragg diffraction to obtain the 3D diffraction pattern. Afterwards, the phase retrieval algorithm, consisting of the iterations between the reciprocal and real space and constraints applications, is employed to recover the phase information.

Studied nanoparticles were prepared by gas aggregation cluster sources combined with magnetron sputtering of single metallic targets. This physical preparation method is environmentally friendly, scalable to industry demands, provides high cleanness of the process and tunability of nanoparticles structure, and therefore it is highly interesting for industrial applications. The structure of silver nanoparticles was analysed after annealing to 300 °C, i.e., above the temperature of the initial coalescence step. Hafnium nanoparticles were studied as-deposited, and different morphologies were detected. The shape of the strain fields suggests that their presence could be connected with the high complexity of the preparation method. The changes in morphology and inner strain field of Hf nanoparticles were tracked by BCDI in-situ during annealing up to 200 °C in an air atmosphere. BCDI results are combined with outcomes from additional characterization methods, namely in-situ powder X-ray diffraction and electron microscopies, for a complete description of the nanoparticle oxidation process.

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