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Studies of material structure and process with coherent diffraction and time-resolved X-ray imaging

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The success of structural science today is largely based on the use of X-ray diffraction on crystalline specimens. However, not all materials can be crystallized. This is especially true for membrane proteins and for larger multi-protein macromolecular assemblies. As the complexity of the biological systems being studied increases, their spatial dimensions also approach sub-micrometer scale which is on the borderline between what one can see directly with microscopic and imaging techniques and what can be studied using diffraction and scattering [1]. Coherent X-ray diffraction imaging technique has become an exciting field of research as it holds the potential for high-resolution imaging of nonperiodic structures at diffraction-limited spatial resolution, without being limited by manufacturing defects of X-ray optics. In this talk we will provide an overview of the coherent diffraction imaging program at the APS. In particular, we present recent research and development on the concept of crystal guard aperture [2] as a way to eliminate parasitic-scattering backgrounds in coherent diffraction imaging experiments, which is crucial to achieving high signal-to-noise ratios at high diffraction resolution. We have applied this method to study metallic nanofoam formation in dealloying process. Recent experimental and phase-retrieval results will be presented. This work is performed at the Advanced Photon Source at Argonne National Laboratory which is supported by DOE BES under Contract No. W-31-109-ENG-38.

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Keywords: coherent diffraction imaging, phase problem, nanofoam structures

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Studies of silica aggregate structure and its dynamics in rubber using time-resolved USAXS and XPCS

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Rubber filled with silica particles shows reinforcement effect, which causes various changes in mechanical and viscoelastic behavior such as the increase of elastic modulus, tear strength, and tensile strength and hysteresis loss. Understanding of the physical mechanism of the reinforcement effect is one of the most important unsolved issues in the field of elastomers. In the present study, we focus on the role of silica aggregate structure on the reinforcement. By using time-resolved two-dimensional ultra-small- and small-angle X-ray scattering (2D USAXS-SAXS), we have obtained the structural change of silica aggregates in uniaxially deformed rubber in real time. The measured deformation behavior of aggregates qualitatively explains the stress-strain behavior of filled rubber. In addition, we performed X-ray photon correlation spectroscopy (XPCS) in order to elucidate the viscoelastic properties of filled rubber, in particular, the origin of energy loss. We obtained information on the structural fluctuation of silica aggregates in rubber by using XPCS. The results have revealed non-diffusive behavior of silica dynamics in rubber. Furthermore, XPCS measurement during vulcanization process of rubber clearly monitored the slowing process of silica accompanied with the cross-link of rubber polymers.

Keywords: time-resolved 2D-USAXS, XPCS, reinforcement effect

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Time-resolved monitoring of nanocomposite growth using grazing incidence small-angle scattering

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Nanocomposite organic-metallic materials are of crucial importance for many areas in modern technology. To name just a few, are biosensoring [1], solar cell applications [2] or organic electronics [3]. In order to design the structure-function relationship, one must tailor the interface metal - organic layer. This ranges from selective contacting [4] to exploiting the plasmon resonances of the nanostructured metal layer [5,6]. Deposition techniques include solution-casting [7,8], and vacuum deposition [5,6]. Therefore, understanding the growth kinetics of the nanocomposite during deposition is indispensable for tailoring the nanocomposites' properties [6]. Grazing incidence small-angle x-ray scattering (GISAXS) [5,9,10] allows for in-situ monitoring of the full three-dimensional structure and morphology of nanocomposite thin films in real time. We combined in-situ gold sputter-deposition on different organic multilayer substrates with GISAXS at the beamline BW4 / HASYLAB. Our real-time investigations allow for identifying the different kinetic growth regimes during sputter deposition and for deducing the changing structure of the polymer-metal interface with high time-resolution [11].

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Keywords: time-resolved structural studies, nanocomposites, small-angle X-ray scattering

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Cctbx architecture and algorithms

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After about eight years of very active development, the Computational Crystallography Toolbox (cctbx) has evolved into a large collection of reusable, open source libraries covering many aspects of crystallography. The cctbx has roots in both small-molecule crystallography and macro-molecular crystallography and is used in both fields. The cctbx libraries are organized in a hierarchy of modules. It is possible to use subsets of the modules independently of other modules that may not be required for certain applications. We will present an overview of the cctbx architecture and the underlying software technology that has enabled the sustained growth of the libraries.
Links: cctbx.sourceforge.net, phenix-online.org

Keywords: crystallographic algorithms, object-oriented libraries, open source

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Identifying residues using 3D coordinates: An application of multiple APIs

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Given a small macromolecule of known sequence and the 3D atomic coordinates and atom types (but not the residue types) of all atoms, how should one assign the atoms to particular residues? This is a non-trivial structural homology problem. The Kabsch algorithm [1] can align structures of similar size, but it is not applicable when matching a small number of atoms from a residue template to a macromolecule. One could use the atoms to generate density and then trace the chain, but that fails to make use of the known information on atom types and requires a large complex program. We propose an alternate, simpler solution using a combination of CBFlib[2] and a linear algebra API to fit the molecule residue-by-residue starting with the largest and back-tracking when necessary. This approach to the problem is workable and suitable for embedding in graphics software. We created a program that uses these two APIs and in most cases identifies which atoms of a molecule belong to the specified residue types and produces a valid mmCIF file. Some issues that arose were variations in residue conformation, missing atoms and the similarity between the structure of some smaller residues to portions of other residues. The resulting program successfully assigns most of the test molecule atoms to the correct residues. The functionality of the program is being added to RasMol and PyMol.

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Magnetic structure determination combining nonpolarised and polarised neutron diffraction

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Determination of the ground state is a key starting point in studying static and dynamic properties of any system: quantum magnet, multiferroic, heavy fermion, superconductor. For complex magnetic structures a combination of several techniques often is necessary to derive the unique solution. I will present a number examples from frustrated magnets ($Cu_2Te_2O_5Cl_2$[1] and Cu3TeO6) and quadrupolar ordered systems (CeB_6[2], $Ce_2Pd_{20}Ge_6$) which demonstrate usefulness of combination of nonpolarised and polarised neutron single crystal diffraction. This complex task is made possible using the Cambridge Crystallographic Subroutine Library[3].

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Keywords: magnetic structures, neutron polarimetry, frustrated magnets, quadrupolar ordering

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An algorithm for determining crystal lattices in unknown polycrystalline compounds

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When recovering crystallographic orientations of individual grains in polycrystalline materials both unit cell parameters and Bravais lattice are utilized. These crystallographic properties are normally determined from the radial spectra of diffraction rings by Powder Diffraction Indexing programs. Afterwards, grains are located in reciprocal space by identifying copies of the Bravais lattice [1,2]. Here, a new method is presented for determining the crystallographic properties of unknown polycrystalline compounds. Effectively, the method reduces the polycrystalline data set into a single crystal data set. As the Bravais lattice is embedded multiple times in the data set (multiplicity is given by number of grains) the method seeks to recover the base lattice by rotating the whole data set and match it against itself. If, by chance, the rotation is close to the crystallographic mis-orientation between two grains the algorithm identifies the two lattices. A match against previously identified lattice solutions is made. Clearly, the probability of making a false match in either selection step is high. In the algorithm a number of filtering steps ensures that the final estimation of the base lattice is only constructed from high frequency lattice points. The unit cell and Bravais lattice can then be determined from the base lattice by a single crystal indexing program. The algorithm itself is implemented in [2]. The algorithm and results will be presented.

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Keywords: pattern recognition, indexing, polycrystals