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USAXS: A Tool to Probe the Structure and Dynamics of Complex Fluids
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For small-angle X-ray scattering (SAXS) experiments requiring high angular resolution and dynamic range, the Bonse-Hart camera [1] is still an attractive technique. The low divergence and high flux of an undulator beam permits to use a crossed-analyser configuration, and obtain the unsmeared high resolution scattering data on an absolute scale (without requiring a calibration standard) within an acquisition time of a few minutes. However, Bonse-Hart double-crystal diffractometers with multibounce channel-cut crystals show rocking curves which depart considerably in their wings from dynamical diffraction theory [2]. The aim of this work is to improve and eventually suppress this parasitic scattering background originating from the surface of the crystals. This will make the ultra small-angle X-ray scattering (USAXS) technique suitable for studying weakly scattering biological samples and very dilute systems such as aerosol particles in a flame. This will be demonstrated using bio-colloids exhibiting hierarchical structures on the nanometer to micron scale. Furthermore, an improved USAXS set-up will allow to perform time-resolved experiments in the USAXS region and to exploit the coherence of the beam for scattering and imaging.

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Keywords: USAXS, complex fluids, microstructure and dynamics

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Anomalous SAXS and WAXS for the Structure of Pt/Ru Catalyst Nanoparticles
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With the high flux (3×10^{11} photons/s) and wide photon spectrum (5-35 keV) provided by the newly constructed superconducting wavelength shifter at the National Synchrotron Radiation Research Center, Taiwan, we study the structure of Pt and Pt/Ru nanoparticles embedded in carbon grains for fuel cell applications, using anomalous small angle X-ray scattering (ASAXS) and wide angle X-ray scattering. Using photon energies near 11.5 and 22.1 keV for the L and K absorption edges of Pt and Ru, respectively, in ASAXS, we have extracted the structural information for the Pt nanoparticles, including a mean dia. of 2.3 nm and a polydisperisty of 25%. Whereas the ASAXS result for the Pt/Ru nanoparticles indicates clearly a core-shell structure with Pt taking the core position. On the other hand, at a temperature above 450 °C WAXS reveals a sign of core-shall structural inversion for the Pt/Ru nanoparticles of an average dia of 3.3 nm. The structural inversion results in a significant growth in particle size, from a diameter of 3.3 nm to 5.0 nm, due to the reorganization and fusion of the nanoparticles. The coordinating numbers of Pt and Ru extracted from EXAFS data for the nanoparticles support the core-shell structure revealed by ASAXS and WAXS.
Keywords: anomalous scattering, small-angle X-ray scattering, nanoparticles

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Interface Structure in Solid Oxide Fuel Cells by Anomalous/High-Energy SAXS/WAXS
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Control of solid oxide fuel cell (SOFC) microstructure and chemistry is needed to optimize SOFC performance and cost. Within the composite electrodes, the variation in the triple-phase-boundary (TPB) morphology (where the gas-, electron- and ion-conducting phases all meet), as a function of distance from the electrolyte layer, is particularly significant in defining electrochemical performance. Thus, characterization of the void and phase microstructures within the anode and cathode, at sufficient resolution to infer a quantitative characterization of the TPB interface, is highly desirable. This has become possible by utilizing the high brilliance and high x-ray energies available at a 3rd generation hard x-ray synchrotron source.

Anomalous ultrasmall-angle x-ray scattering (USAXS), close to the Zr absorption edge, has been combined with high-energy small-angle and wide-angle scattering (HE-SAXS and HE-WAXS). For the first time this has provided correlated variations in void size distribution, interface surface area, and solid phase, to below 10 micrometers spatial resolution, and has enabled the ion-conducting YSZ phase to be distinguished from the voids and electron-conducting phase (LSM or Ni). From thses data, TPB properties can be inferred.

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Keywords: small-angle X-ray scattering, interface structure, solid oxide fuel cells

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Structural SAXS Studies of the Human Amyloid Precursor Protein
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The amyloid precursor protein (APP) gives rise to the β-amyloid peptide, considered to be a causal factor for Alzheimer's disease. The soluble extracellular domain of APP released by α-secretase cleavage (sAPPα) has several important physiological functions. Several APP fragments have been structurally characterized at atomic resolution, but the structures of APP and full-length sAPPα have not been determined. In this work, *ab initio* reconstruction of molecular models from solution X-ray scattering (SAXS) data for the two main isoforms of sAPPα (sAPPα_{695} and sAPPα_{770}) provided models with enough resolution to identify distinct domains. Using the *ab initio* models and molecular docking tools, the fragments whose structures are known at the atomic level were optimally fit within the models of full-length sAPPα, allowing localization of important functional sites (glycosylation, protease inhibition and heparin-binding sites). Furthermore, SAXS and analytical ultracentrifugation (AUC) results indicate that both sAPPα isoforms are monomeric in solution. AUC measurements further show that sAPPα_{695} forms a 2:1 complex with heparin, in agreement with SAXS results. Possible implications of such complex formation for the dimerization of sAPPa and biological signalling are discussed in terms of the structural models proposed.
Sponsors: FAPESP, LNLS, CNPq
Keywords: SAXS, human amyloid precursor protein, molecular model reconstruction

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Diffuse Scattering of Pt-Rh in Grazing Incidence
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The near-surface microstructure of Pt-47 at.% Rh was investigated by diffuse scattering under grazing incidence at 730°C and UHV condition. The (110) and (111) surfaces investigated had a crystallographic misorientation of less than 0.05°. The samples were polished, then sputtered and aged prior to the diffraction experiments done at the Materials Science Beamline 4S of the SLS. The incident

angle was 0.7 or 0.9 times the critical angle for total reflection. Thus, a depth of 2 and 3 nm, respectively, of the top-layers was probed.

For the bulk microstructure, no long-range order has been experimentally established in Pt-Rh owing to small interactions and sluggish kinetics. Only recently, weak diffuse maxima were found at 1 ½ 0 positions. For the near-surface microstructure, typically 600 positions were measured, with about 500 counts in 200 s. After calibration and subtraction of the inelastic scattering contributions, the remaining elastic scattering was decomposed into short-range order scattering, size-effect scattering, and Huang scattering. Local order was revealed for both surfaces, but there were differences, too. For the (110) surface, the diffuse maxima were located at 1 ½ 0 positions as for the bulk microstructure, for the (111) surface they were at ½ ½ ½ positions. In the evaluation, surface segregation was considered negligible, an assumption that seemed justified in view of the measured and calculated segregation profiles.

Keywords: grazing incidence diffraction, alloys, short-range order

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Shape, Polydispersity and Aggregation Number of Alkyl Sulphate Micelles

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Based on the thermodynamic consideration that the micellar cores are compact and consist entirely of portions of the hydrocarbon chains, the length of the latter should limit at least one micellar dimension. Due to the fluctuations in the local electrostatic field, the hydrocarbon chains in ionic micelles with realistic aggregation numbers can fit the above spatial constraints only if the micellar shape is a prolate or oblate ellipsoid.

The recent methods of interpreting experimental data are ambiguous in this respect in that the results they provide may be compatible with each of the above given types of micellar shape. One of the reasons for this is that the scattering pattern from monodisperse spheroids can be fitted e.g. by a set of polydisperse spheres. The present work is devoted to demonstrate that the scattering patterns from alkyl sulphate micelles can be well fitted by assuming either monodisperse prolate- or polydisperse oblate ellipsoidal micelles. Because the two types of micelle have significantly different mean aggregation numbers, the decision on the shape and polydispersity may be based on independent information of the mean aggregation number stemming e.g. from thermodynamic theories.

Keywords: micelles, interfaces, scattering

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Evolution of Nanocrystallinity in Periodic Mesoporous Anatase Thin Films

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Within the last few years, many periodic mesostructured forms of titania denoted meso-TiO_2 have been produced based upon the self-assembly method. Besides the usual benefits of the self-assembly method including high surface area and uniform pore size and shape, the crystallinity and crystallite size of the anatase composing the channel walls of meso-TiO_2 are an equally important factor since potential applications rely upon the intrinsic properties of titania governed by the extent and nature of its crystalline phase. Although crystallite growth, during the film calcination step, within the mesostructured titania framework should be considered the critical step in the formation of meso-TiO_2 thin films, the issue of crystallite growth has yet to be identified as a major determining factor with respect to the properties of meso-TiO_2 thin films and their applicability to electroactive and photoactive devices.

Herein we report the first kinetic study of the intrachannel wall phase-transition of amorphous titania to nanocrystalline anatase for periodic mesoporous titania thin films, monitored by time-resolved *in-situ* high temperature X-ray diffraction (HTXRD).[1] Structural transformations associated with the phase transition are further probed by high-resolution scanning electron microscopy (HRSEM) and transmission electron microscopy (HRTEM). The model found to be most consistent with the kinetic data involves 1-D diffusion controlled growth of nanocrystalline anatase within the spatial confines of the channel walls of the mesostructure. The observation of anisotropic, rod-shaped anatase nanocrystals preferentially aligned along the channel axis implies that the framework of the liquid crystal templated mesostructure guides the crystal growth.

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Keywords: crystal growth, *in situ* temperature diffraction, thin-film material

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Sol-gel Synthesis and Study of $LaPO_4$ Doped with Cerium(III) and Ytterbium(III) Thin Films

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In this research nanocrystalline thin films of Lanthanium phosphate doped with cerium(III) and ytterbium(III) were prepared.

Lanthanium oxide reacted with hexa hydrated cerium nitrate or ytterbium oxide dissolved in nitric acid and mixed with chelating agent for the metal ions (i.e. water-ethanol solution containing citric acid). The required amount of Poly ethylene glycole (PEG) as cross-linking agent and diamonium hydrogen phosphate were also added using a sol-gel method. The product was then dried on silica glass substrate. Heating on dried films for 5 hours in the temperature range of 800-1000°C results in formation of Lanthanium phosphate doped with cerium(III) and ytterbium(III). X-ray diffraction (XRD) and FT-IR spectroscopy, SEM and Fluorimetrie of doped lantanium phosphates were presented.

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Keywords: sol-gel, lanthanium phosphate, cerium and ytterbium

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Pulsed Laser Deposition Growth of thin Hydroxyapatite Layers on Titanium Substrates

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Hydroxyapatite-based ceramics are largely applied as coatings on metallic components of prostheses for inducing osteoblasts apposition and subsequent regrowth. However the ceramics-metal interfaces are often the seat of residual stresses with amplitude primarily depending on the deposition technique and the coating conditions. The amplitude of this stress can be very high causing a failure mechanism at the interface. Our aim is to validate new methods of laser ablation deposition (PLD), making it possible to control the residual stresses in ceramic layers and adherence to titanium substrates. We present a