MM 13: Topical Session Nanoanalytics using Small-Angle Scattering I

Time: Tuesday 10:15–11:30 Location: IFW A

Invited Talk MM 13.1 Tue 10:15 IFW A

Small-Angle Neutron Scattering in Materials Science — •Pavel Strunz[1], Debashis Mukherji[2], Gerhard Schumacher[3], Ralph Gilles[4], and Albrecht Wiedenmann[5] — [1]NPI and Research Centre Řež, CZ-25068 Řež near Prague — [2]TU Braunschweig, IfW, Langer Kamp 8, D-38106 Braunschweig — [3]Helmholtz-Zentrum Berlin, Glienicker Str. 100, D-14109 Berlin — [4]TU München, Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II), Lichtenbergstr. 1, D-85747 Garching — [5]ILL, 6, rue Jules Horowitz, BP 156, F-38042 Grenoble Cedex 9

A complementary use of Small-Angle Neutron Scattering (SANS) can be considered for microstructural investigations. In special cases, it can even be indispensable as it provides information not accessible by other methods. Due to the low absorption of neutrons by a majority of elements, it is a suitable method for bulk characterization as well as for in-situ studies at extreme conditions. Neutrons enable relatively easy contrast variation which helps to resolve details of microstructure. A sensitivity to magnetic inhomogeneities has to be pointed out as well.

Examples taken from the research of Ni superalloys and ceramic thermal barrier coatings demonstrate the application of in-situ SANS in investigation of high-temperature materials. The evolution of precipitates in Ni-Fe-based superalloy and formation of nanopores in YSZ coating was observed. Another study employs contrast variation for characterisation of nanoparticles produced by extracting precipitates from a bulk alloy. The existence of a core-shell structure of the nanoparticles was confirmed by SANS.

MM 13.2 Tue 10:45 IFW A

In-situ SANS study of domain formation during condensation and evaporation of fluids in SBA-15 — •Maxim Erko[1], Johannes Prass[1], Dirk Wallacher[2], Astrid Brandt[2], and Oskar Paris[1] — [1]Department of Biomaterials, Max Planck Institute of Colloids and Interfaces, D-14424 Potsdam, Germany — [2]Helmholtz Centre Berlin, Glienicker Straße 100, D-14109 Berlin, Germany

Ordered mesoporous materials such as SBA-15 are ideal model systems to study the sorption and condensation behaviour of fluids in confined geometry. A particularly interesting question relates to collective phenomena such as the formation of domains of unfilled and filled regions that extend over much larger spatial distances than the typical pore size. We have studied the formation and dissolution of such domains upon condensation and evaporation of contrast matching H2O/D2O and Perfluoropentane (C5F12) in SBA-15 silica with in-situ small angle neutron scattering (SANS), using the DEGAS sorption system at the BENSC neutron facility at Helmholtz Centre Berlin. The SANS data can be modelled by randomly distributed domains of filled and unfilled mesopores on a lattice as described by the classical Laue-scattering. Thus, the SANS data in the capillary condensation / evaporation regime can be fitted by an analytical model with only one free parameter, i.e., the filling fraction of mesopores. The quantitative values of this parameter are compared with the results from the independent volumetric sorption experiment, and the differences are discussed with respect to the metastability of the fluid in the adsorption regime.

MM 13.3 Tue 11:00 IFW A

Analytical Determination of Micellar Nanoreactor Structure by Means of SANS — •Roza Bakeeva[1], Alexandr Kuklin[2], and Vladimir Sopin[1] — [1]Kazan State Technological University, Kazan, Russia — [2]Joined Institute for Nuclear Research, Dubna, Russia

We have studied the reaction of decomposition of toxic phosphorus esters (O-4-nitrophenyl-O,O-dimethyl-tiophosphate) in micellar media, containing compounds with nucleophilic properties (potassium fluoride KF and sodium hydroxide NaOH). These additives are electrolytes and their addition brings about the reducing a surface potential micelles and it is possible to control the changing of size and form of micelles. In this instance micelle is considered as nanoreactor.

The SANS method was used to determine the characteristic parameters of nanoreactors - micelles (radius, shape, aggregation number). Neutron scattering curves were obtained on YuMO small-angle scattering setup (two-detector version), using a high-efficiency neutron pulse source (IBR-2 reactor). The shape of scattering associates (micelles) was determined by Porod invariant and Guinier plot analyses, as well by simulation using the FITTER program package The most appropriate model of a particle is a sphere for system CTAB + D2O, CTAB + KF + D2O and a cylinder for system CTAB + KF + NaOD + D2O.

The obtained data have shown that introduction KF brings about the compression of micelles CTAB, but addition of NaOH stimulates the sphere to cylinder transition that in the significant measure defines a difference in their catalytic characteristics.

MM 13.4 Tue 11:15 IFW A

Lots wife problem in biomineralization: An exploration of colloidal protein-minerals particles with SANS — Alexander Heiss[1,2], Vitaliy Pipich[1], Willi Jahnen-Dechent[2], and •Dietmar Schwahn[1] — [1]IFF des Helmholtz Forschungszentrum Jülich — [2]IBMT der RWTH Aachen

The serum protein fetuin-A is an important inhibitor of calcification in mammals. In vitro experiments using SANS and TEM demonstrated, that fetuin-A mediates the formation of colloidal protein-mineral composites (CPP) in aqueous supersaturated calcium phosphate solutions. Colloidal particles, very similar to those in vitro synthesized secondary CPPs, were also found in ascites from a patient with calcifying peritonitis, a rare clinical complication. Here we show that SANS contrast variation provides detailed information on the topology and the composition of protein-mineral colloids.

Fetuin-A gives rise to a two-stage process of colloid formation. The primary CPPs are spherical and have a diameter of 50 nm, whereas the secondary CPPs have an ellipsoidal shape and a diameter of 200x100 nm. Contrast variation SANS in combination with singular value decomposition technique revealed that the primary CPPs appear as a homogeneous protein-mineral composite whereas the secondary CPPs consist of a compact octacalcium phosphate nucleus covered by a fetuin-A monolayer. Moreover, the analysis of the SANS data indicated that in fact most fetuin-A was still in solution as monomers loaded with calcium and phosphate ions.