

MM 45: Nanostructured Materials VI

Time: Thursday 15:00–16:00

Location: H 0111

MM 45.1 Thu 15:00 H 0111

Analysis of sorption strains in mesoporous silica by in-situ x-ray diffraction — •JOHANNES PRASS, DIRK MÜTER, JOHN DUNLOP, STEFAN SIEGEL, CHENGHAO LI, and OSKAR PARIS — Max Plank Institut für Kolloid und Grenzflächenforschung, 14424 Potsdam, Germany

Sorption and capillary condensation of fluids in ordered mesoporous silica (SBA-15 and MCM-41) were studied by in situ small-angle X-ray diffraction using synchrotron radiation. Diffraction peaks resulting from the pore lattice were evaluated in terms of lattice strains. Intensity, position and width of the diffraction peaks show pronounced changes in the capillary condensation/evaporation regime. At complete pore filling, an expansion of the lattice is observed which can be explained by the decrease of total interfacial energy. A contraction of the pore lattice is observed for the nearly filled pores for both branches of the sorption isotherm, which is attributed to liquid menisci due to capillary condensation of fluid in the pores. Data measured for fluids with different surface tension and silica materials with different pore width allow to vary elastic strains in the pore lattice and to compare them with simple models.

MM 45.2 Thu 15:15 H 0111

Modeling experimental SAXS data from fluid sorption in ordered mesoporous silica — •DIRK MÜTER¹, SUSANNE JÄHNERT², GERHARD FINDENEGG², and OSKAR PARIS¹ — ¹Department of Biomaterials, MPI of Colloids and Interfaces, Potsdam — ²Stranski Laboratory of Physical and Theoretical Chemistry, TU Berlin

Experimental small angle x-ray scattering (SAXS) data from an in-situ sorption experiment of dibromomethane (CH_2Br_2) in 2D hexagonally ordered mesoporous silica (SBA-15) are evaluated using two complementing modeling approaches. The SAXS data were measured as a function of relative vapor pressure of dibromomethane at room temperature. The integrated intensities of up to 10 measured Bragg reflections were simultaneously fitted with a superposition of analytical functions derived from an improved model for the formfactor of the pore/liquid system [1]. By this the pore morphology can be described, and the sorption process can be followed quantitatively in terms of liquid film formation and capillary condensation. However, a discrepancy of the total amount adsorbed is noticed in comparison to the sorption isotherm measured by a microbalance. To account for this difference we have studied a model in which additional micropores are randomly inserted in the mesopore walls of the silica matrix. Numerical Fourier transformation of this model system shows that the micropores contribute mainly to diffuse scattering while adsorption into these additional pores improves the agreement with the microbalance

measurements.

[1] G. Zickler et al., *Phys. Rev. B* 73 (2006) 184109.

MM 45.3 Thu 15:30 H 0111

Elastic properties of argon adsorbed in nanoporous glass — •KLAUS SCHAPPERT and ROLF PELSTER — Universität des Saarlandes, FR 7.2 Experimentalphysik, Campus E 2.6, D-66123 Saarbrücken

We have performed isothermal adsorption and desorption experiments on argon in nanoporous Vycor. The effective shear modulus has been determined measuring the amount of adsorbed argon as well as the ultrasonic velocity near 12.5 MHz. Since fluids cannot sustain shear stress, argon only affects the effective shear modulus at temperatures below its freezing point ($T_{f,conf} < T_{f,bulk}$). Depending on the filling fraction, three different behaviours are observed: (i) The first adsorbed argon layers do not contribute to the measured shear modulus. (ii) In an intermediate range the shear modulus increases linearly with increasing argon content. (iii) At a characteristic filling fraction an abrupt enhancement of the shear modulus is observed. We discuss these phenomena in terms of capillary sublimation and crystallization.

MM 45.4 Thu 15:45 H 0111

Li diffusion in nanocrystalline and nanoglassy β -spodumene $LiAlSi_2O_6$ — •ALEXANDER KUHN, MARTIN WILKENING, and PAUL HEITJANS — Institute of Physical Chemistry and Electrochemistry, Leibniz University Hannover, Callinstr. 3-3a, 30167 Hannover

Recently nanocrystalline and nanoglassy ceramics have been a subject of growing research interest. In the present study β -spodumene $LiAlSi_2O_6$ and the corresponding glass were used as a model system. The influence of particle size on Li ion dynamics was studied by ⁷Li NMR spin-lattice relaxation rate measurements. As a general trend, the Li diffusivity in the glassy sample is larger than that in the μ -sized crystalline material [1, 2]. However, as the particle size decreases, the nanocrystalline material shows enhanced diffusivity whilst the opposite effect is found for the nanoglassy ceramics. The nanocrystalline and nanoglassy samples were prepared by high-energy ball-milling of the coarse grained starting material and the corresponding glass, respectively. A possible structural model trying to explain the observed convergence of the diffusivities of the nanocrystalline and nanoglassy samples is discussed.

[1] F. Qi, C. Rier, R. Böhmer, W. Franke and P. Heitjans, *Phys. Rev. B* 72 (2005) 10430.[2] W. Franke and P. Heitjans, *Ber. Bunsenges. Phys. Chem.* 96 (1992) 1674.