PS14.02.15 POROD SCATTERING OF NEUTRONS BY AN ANISOTROPIC MICROSTRUCTURE. G. G. Long[1], J. Ilavsky[1,2], A. J. Allen[1], [1]Ceramics Division, NIST, Gaithersburg, MD 20899, [2]Plasma Spray Laboratory, SUNY/Stony Brook, Stony Brook, NY 11794

The specific surface area of the pores and cracks that are found in complex microstructures, such as plasma-sprayed ceramic deposits, was investigated by means of small-angle neutron scattering as a function of an array of processing variables. The prevailing orientation of microcracks, which presumably form upon cooling, depends upon the spray direction. Voids formed between the lamellae, however, are independent of spray angle. The amount of microcracking that takes place depends upon the chemistry of the deposit and the spraing conditions.

The above results were derived from large-Q ($|Q|=(4\pi/\lambda)\sin\theta$) small-angle neutron scattering data, described by the single-particle diffraction Porod law. When the shapes are isotropically distributed, the Porod law is independent of the scattering particle shape, and independent of direction in the material. The preferentiallyoriented polydisperse systems of pores and cracks in a plasma-sprayed microstructure, however, will lead to anisotropic determinations of an "apparent" Porod constant. For these specific materials, there are two anisotropic directions and one isotropic direction, which makes possible a direct quantitative determination of specific surface areas as a function of orientation, when data from three orthogonal planes within the material are available.

PS14.02.16 SAXS INVESTIGATION OF SILICA AEROGELS OBTAINED FROM A SINGLE-STEP PROCESS BOOSTED BY FLUORINE ANIONS. Lucjan Pajak[a] and Andrzej Jarzebski[b], Silesian University, Institute of Physics and Chemistry of Metals, Bankowa 12, PL-40007 Katowice, Poland[a], Institute of Chemical Engineering, Polish Academy of Sciences, Baltycka 5, PL-44100 Gliwice, Poland[b]

The structure of aerogels depends on the rate of gelation (reaction of hydrolysis and condensation) which is markedly affected by the choice of catalyst. Fluorine anion catalysts dramatically accelerate this process but as yet have been used quite rarely in aerogel technology. A homogeneous series of aerogels synthesized using TEOS, EtOH and a complex catalyst comprising ammonium hydroxide and ammonium fluoride and dried at EtOH supercritical conditions, were investigated by means of the nitrogen adsorption method and SAXS. Since aerogels are hyperporous solids exhibiting disordered structure, the results were quantified using the fractal geometry approach. It was found that:
• aerogels obtained from the process exhibit polymeric rather than colloidal structure usually observed in aerogels obtained from the conventional basic process,
• primary particles are markedly smaller than observed in conventional basic aerogels,
• lighter samples show a pure mass fractal structure extending over a decade   in the length scales from 40 Å upwards with D_m= 1.8-2.3 depending on   water content,
• small water content leads to the rough surface fractal structure consistently with poisoned-Eden growth mechanism [1],
• mass/volume fractal characteristics obtained from SAXS and power-law behaviour of pore volume vs pore diameter differ substantially despite a certain similarity in values of D_m/D_V.

[1] K.D. Keefer, D.W. Schaefer, Phys. Rev. Lett. 56, 2376 (1986)

PS14.02.17 DETERMINATION OF THE MICRO-STRUCTURE OF HYDRATING CEMENT PASTE BY SMALL ANGLE NEUTRON SCATTERING. T M Sabine, W K Bertram, L P Aldridge, F Eichhorn[*] and I Joffe[*], Materials Division, ANSTO, Sydney, Australia, [*]BENSC, Hahn-Meitner Institute, Berlin, Germany

SANS data from two types of cement show that there is a significant difference in the development of the micro-structure during hydration. Data on hydrating OPC (ordinary Portland cement) and DSP(cement containing silica fume) were collected on the V 12 neutron interferometer at BENSC.

In analysing the data the square of the structure factor, $[F(q)]^2$, is expanded in powers of the variable x_j which is defined by the equation

$$x_j = \frac{1}{\sqrt{1 + \frac{1}{3}(qR_j)^2}}$$

R_j is a parameter with the dimensions of length specifying an average size or correlation length in the scattering object.

The first, second and fourth powers of x_j give excellent approximations to the classical solutions for the rod, disc and sphere respectively while non-integral powers provide the fractal type solutions.

After inclusion of multiple scattering, absorption in the detector crystal, and background, the model is refined by least-squares methods against the raw experimental data.

The results show clearly that the inhomogeneities in OPC are spheroidal while those in DSP are disc like. The growth in size of the inhomogeneities with time is correlated with the evolution of heat.

PS14.02.18 EVIDENCE OF ASSOCIATED STATES DURING REFOLDING OF CYTOCHROME c. Dan Segel[1], Sangita Seshadri[2], Hirotsugu Tsuruta[3], Anthony L. Fink[2], Keith O. Hodgson[3,4], Sebastian Doniach[5]. Department of [1]Physics, [4]Chemistry, [5]Applied Physics, Stanford University, Stanford, CA 94305; Department of [2]Chemistry, University of California, Santa Cruz, CA 95064; [3]SSRL, Stanford University, Stanford, CA 94309

Preliminary time-resolved small angle x-ray scattering (SAXS) studies of cytochrome c refolding show association of the protein sample to form possibly dimers at protein concentrations as low as 3.5mg/ml. These studies extend earlier results[1].

In these experiments SAXS data were taken upon 7-fold stopped-flow dilution from protein in 3.5M GdnHCl solution at pH7. We found the forward scattering intensity to decrease by a factor of 1.3 over a 21s observation period, indicating dissociation of dimers or higher order associates. Longer term kinetics studies of cytochrome c refolding were also performed on a manually mixed solution. Beginning 10min. after mixing, SAXS data showed a steady forward scattering intensity which is nearly twice that measured for the native protein dissolved in buffer at pH7. This suggests the formation of a long-lived, fully dimerized state.

The time-resolved data was analyzed in the Guinier region to obtain the time course of the radius of gyration. R_g in 3.5M GdnHCl at pH7 was found to be about 34.5Å, falling to 22.5Å on 7-fold dilution. R_g for the native solution is about 14Å. Thus the observed R_g of the refolded sample is consistent with dimer formation. Extraction of p(r), pair-distance distribution function, from the higher angle data is also qualitatively consistent with that generated using a simple dimer model.

[1]Transient association during the folding of cytochrome c, D.Eliezer, H.Tsuruta, H.Kihara, S.Doniach and K.O.Hodgson, submitted for publication.