

**PS14.02.11 SMALL-ANGLE X-RAY SCATTERING OF ZEOLITE-SUPPORTED Pt CATALYSTS: ANCHORING AND FRAMEWORK EFFECTS.** H. Brumberger, J. Goodisman, R. Ramaya, Department of Chemistry, Syracuse University, Syracuse, NY 13244

SAXS measurements on Pt/Fe/LZY-52(NaY) catalysts\*, prepared with loadings ranging from ~ 1.4 to 3.4w/o Pt (1:1 Pt/Fe atomic ratio), calcined at 300, 400 or 500°C and reduced in H<sub>2</sub> at 400°C, yield average Pt particle sizes generally below those of Pt/LZY-52 at the highest calcination temperatures. This may be a consequence of "anchoring" by Fe<sup>2+</sup>, reported by Tzou *et al* (1).

Similar measurements on Pt supported by a different type of zeolite framework, Pt/LZ-M-5\*, which has a mordenite-type of structure, show that sintering of the metal appears to be severely restricted at all calcination temperatures and loadings. Average Pt particle sizes are appreciably smaller than those found under similar preparation conditions on the NaY or Fe/NaY supports, which have a three-dimensional channel system, while LZ-M-5 has a straight one-dimensional one.\*\*

Experimental and analytical details will be presented for these measurements.

\* LZY-52 and LZM-5 are Union Carbide Corporation catalysts. We are grateful to the company for research samples.

\*\* Supplier's data sheet.

(1) Tzou, M. S., Jiang, M. J. and Sachlter, W. M. H., *Appl. Catal.* 20, 231 (1986).

**PS14.02.12 TEMPERATURE-COMPOSITION PHASE DIAGRAM AND MESOPHASE STRUCTURE CHARACTERIZATION OF THE MONOVACCENIN/WATER SYSTEM.** Martin Caffrey and Hong Qiu, Department of Chemistry, The Ohio State University, Columbus, OH 43210

The temperature-composition phase diagram of monovaccenin (a C18:1c11 monoacylglycerol) in water was constructed using small- and wide-angle x-ray scattering/diffraction in the range of ca. 0 °C to 110 °C and ca. 0 % (w/w) to 60 % (w/w) water in the heating direction. The phases identified in this system include the lamellar crystalline phase, the lamellar liquid crystalline phase, the fluid isotropic (FI) phase, two inverted cubic phases (Q<sup>230</sup>, Ia3d; Q<sup>224</sup>, Pn3m), and the inverted hexagonal (H<sub>II</sub>) phase. The monovaccenin/water phase diagram reported herein is remarkably similar to that of the monoolein (a C18:1c9 monoacylglycerol)/water system. There are some important differences however that will be highlighted in the presentation. Since our long term objective is to establish the relationship between lipid molecular structure and lyotropic/thermotropic mesophase propensity by comparing phase diagrams for an homologous series of lipids, every effort has been taken to ensure that the current phase diagram represents equilibrium behavior and that the assorted phase boundaries have been determined accurately. The interpreted phase diagram is based on close to 500 discrete measurements in temperature-composition space recorded as a function of temperature in 5 °C increments and of composition in 4 % (w/w) water increments on average.

**PS14.02.13 SMALL ANGLE NEUTRON SCATTERING MICROSTRUCTURAL INVESTIGATION OF TWO MARTENSITIC STEELS FOR NUCLEAR APPLICATIONS.** R. Coppola(1), F. Fiori(2), E.A. Little(3), M. Magnani(4), (1) ENEA-Casaccia, C.P. 2400, 00100 Roma, (2) Istituto Nazionale di Fisica della Materia - Ist. Scienze Fisiche, Università di Ancona, 60131 Ancona, (3) University of Wales, U.K., (4) ENEA-Clementel - Bologna

This contribution presents the results of an investigation carried out using small-angle neutron scattering (SANS) to study the microstructure of two martensitic steels of interest for nuclear applications, namely: a) modified martensitic steel DIN 1.4914

(MANET - type) developed for fusion reactor applications (composition: C 0.17 Cr 10.5 Mo 0.50 Ni 0.85 Mn 0.60 Nb 0.20 V 0.25 Si 0.32 Fe to balance wt%). b) AISI 410 steel (C 0.15 Cr 13.00 Mn 0.45 Ni 0.47 Si 0.30 Fe to balance wt%). The present investigation is mainly focused on microstructural effects arising from the different Cr content, namely on the occurrence of C-Cr elementary aggregates, of very small size produced during tempering at 700°C. Evidence of this phenomenon is provided by previous work carried out on MANET both by SANS and by other microstructural techniques (1, 2).

The present investigation shows that, increasing the Cr content for an identical thermal treatment (20 h at 700°C after fast quenching from 1075°C) gives a higher SANS intensity for the AISI 410 samples which is interpreted as a higher volume fraction of C-Cr aggregates. No significant variation in average radius (~ 10 Å) is observed from one material to another. The compared analysis of nuclear and magnetic SANS components shows that there is also a SANS contribution of inhomogeneities having different composition from the one of those aggregates and much larger sizes (~100 Å): this is attributed to the presence of M<sub>23</sub>C<sub>6</sub> carbide precipitates.

#### REFERENCES

- (1) G. Albertini, M. Ceretti, R. Coppola, F. Fiori, P. Gondi, R. Montanari, *Physica B* 213-214 (1995) 812.
- (2) G. Albertini, F. Carsughi, R. Coppola, F. Fiori, M. Stefanon, in print on *J. N. Mat.*

**PS14.02.14 STRUCTURAL DYNAMICS OF CREATINE KINASE.** Michael Forstner<sup>1</sup>, M. Kriechbaum<sup>2</sup>, P. Lagner<sup>2</sup> and T. Wallimann<sup>1</sup>. <sup>1</sup>Institute for Cell Biology, ETH Zuerich, Switzerland, and <sup>2</sup>Institute of Biophysics and X-Ray Structure Research, Austrian Acad. of Sciences, Graz, Austria

Small-angle X-ray (SAXS) and neutron scattering were used to investigate structural changes upon binding of individual substrates or a transition state analogue complex (TSAC), consisting of Mg-ADP, creatine and KNO<sub>3</sub> to creatine kinase isoenzymes (dimeric M-CK and octameric Mi-CK) and monomeric arginine kinase (AK). Considerable changes in the shape and the size of the molecules occurred upon binding of Mg-ATP and TSAC, whereas neither creatine nor free nucleotide led to significant changes in CK structure. In Mi-CK, the radius of gyration was reduced from 55.6 Å (free enzyme) to 48.9 Å (enzyme + Mg-ATP) and to 48.2 Å (enzyme + TSAC). The experiments performed with M-CK showed similar changes from 28.0 Å (free enzyme) to 25.6 Å (enzyme+Mg-ATP, enzyme +TSAC). AK showed the same behaviour. Based on the X-ray structure coordinates of Mi-CK we have tried to model the nature of the structural transition found. The X-ray structure of Mi-CK in the presence of ATP is that of the "open" form as seen by comparison of the calculated and experimentally derived scattering curves. Homology modeling allowed for the derivation of models for M-CK and AK. The primary change in structure as seen with monomeric AK seems to be a magnesium-nucleotide induced domain movement relative to each other, whereas the effect of substrate may be of local order only. One highly mobile loop structure was identified, that might work as a lid to close the active site during catalysis. In CK, however, further movements must be involved in the large conformational change. In Mi-CK the appearance of compactness is concurrent with a change of the channel penetrating the octamer to a more cavity-like structure. Furthermore investigations on the time course of the structural transitions have been performed by time-resolved SAXS.