

**Poster Abstracts**  
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**Soft Materials**

**Poster S-1**

**Structure and Dynamics of Biological Macromolecules Investigated Through Molecular Dynamics Simulations**

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**Poster S-2**

**An approach to the encapsulation of membrane proteins in ordered mesoporous hosts**

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Water soluble proteins can be encapsulated in the mesopores of ordered mesoporous silicas and immobilized with their biomolecular activities preserved. The surface charge density in the unmodified mesoporous silica determines its affinity for water soluble proteins bound via electrostatic interactions. Thus, the retention of cytochrome c (horse heart) in the pores of a mesoporous silica (MCM-41) synthesized using a cationic surfactant template is found to be significantly higher than the retention in a structurally equivalent material (SBA-15) synthesized using a neutral triblock copolymer as template. This is attributed to the different chemical modes of assembly of the silica framework in the two cases. The surface of the mesoporous silica can also be modified with covalently bound groups to enhance the protein interaction. For example, aminoalkyl or mercaptoalkyl groups can be attached to the pore surfaces either by using a co-condensation technique during the synthesis of the mesoporous silica, or by using methods of post-synthesis modification. The binding of the protein to these surface-modified mesoporous silicas can be effected either by electrostatic interactions or by the use of covalent grafting techniques. Protein denaturation is avoided, but we have observed perturbations of the encapsulated cyt. c that vary with the nature of the mesopore surface.

Membrane proteins, on the other hand, cannot be directly immobilized in inorganic mesoporous hosts if their biomolecular activity is to be preserved. These proteins require that their native environments be very closely mimicked. Polymer-grafted lipid-based complex fluids, comprised of a phospholipid, an amphoteric surfactant, and a hydrophilic polymer (polyethylene oxide) dispersed in water, self-assemble into ordered mesophases that contain the desired contiguous hydrophilic and hydrophobic components. It has been previously demonstrated that membrane proteins (e.g., bacteriorhodopsin) can be confined in these fluids with their biochemical activity preserved. One conception of a robust host matrix for active membrane proteins involves integrating the mixed environment of the complex fluid into the rigid framework of a mesoporous silica to produce an ordered hybrid

nanostructure. We have demonstrated the synthesis of an ordered composite material using a lamellar complex fluid as the template for growth of a silica framework. This opens the possibility of creating robust biocomposite materials wherein the specialized natural function of the encapsulated membrane proteins can be exploited for energy transduction, e.g., in photoinduced electron transfer reactions.

This work was supported by the U.S. Department of Energy, Division of Chemical Sciences, Office of Basic Energy Sciences, under Contract W-31-109-Eng-38.

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### Poster S-3

#### NMR Investigations of SAMs on Liquid Metal Surfaces.

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Nuclear magnetic resonance (NMR) spectroscopy and imaging offer powerful methods for probing molecular structures and dynamics in oriented materials. We have recently developed a detector capable of recording proton NMR spectra of organic/inorganic hybrid structures one monolayer thick on mercury and gallium surfaces. We achieved sufficient sensitivity by probing a contiguous monolayer of several square centimeters, which was composed of approximately  $10^{15}$  proton spins. Our investigations of alcohols on gallium surfaces complement previous studies of dodecanethiol self-assembled monolayers on mercury surfaces. In addition, to simulate natural systems, we have studied a lipid monolayer, composed of phosphothioethanol (PPT). PPT has a pendant thiol head group that was employed for covalent attachment to the mercury surface of the NMR detector. Above the PPT monolayer we plan to place various phospholipids to form a bilayer. The most physiologically common phospholipids will be used, including phosphatidylcholine (PPC), phosphatidylserine (PPS), and phosphatidylethanolamine (PPE). Our aim is to characterize these self-assembled model bilayers and eventually work to integrate membrane proteins into them, setting the stage for structure determination, and kinetic studies of ion transport in trans-membrane proteins. This work was supported by the U.S. Department of Energy, Division of Chemical Sciences, Office of Basic Energy Sciences, under Contract W-31-109-Eng-38.

### Poster S-4

#### Scaling of the dynamics of “sweetened” sponges

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Surfactant sponges are complex isotropic labyrinths of membrane in solvent. Although sponges usually behave as Newtonian fluids, tuning the solvent viscosity by adding sugar to the brine solvent in the CPCI-hexanol sponge system allowed us to probe a shear regime in which both macroscopic (rheological) and microstructural (small angle neutron scattering) responses of this system follow a scaling suggested by Cates and Milner - varying with the cube of the membrane volume fraction and inversely as the solvent viscosity. Under Couette shear sufficiently viscous sponge samples are easily observed to shear-thin as they are progressively transformed to a shear-induced lamellar phase with membranes aligned in the flow-vorticity plane ("c"). Time-resolved SANS measurements were performed to study the decay of this shear-induced lamellar phase to the equilibrium sponge state. This relaxation time shows an Arrhenius relationship to the much shorter membrane diffusion times determined by dynamic light scattering. This is consistent with a thermally activated topological relaxation process predicted by Milner - with an energy barrier for the formation of membrane handles is proportional to the intrinsic Gaussian curvature of the membranes ( $\sim 10kT$  for our system). These dynamic studies also clearly demonstrate that the observed shear-induced sponge to lamellar transformation is not a first order transition.

**Poster S-5****Measuring the properties of water clusters in supersonic expansions**Yoojeong Kim<sup>1</sup>, Barbara E. Wyslouzil<sup>1</sup>, Judith Wölk<sup>2</sup>, and Reinhard Strey<sup>2</sup><sup>1</sup>Department of Chemical Engineering, Worcester Polytechnic Institute, Worcester MA 01609-2280<sup>2</sup>Institut für Physikalische Chemie, Universität zu Köln, 50939 Köln, Germany

Particle formation in supersonic flows occurs in many natural and industrial situations, and water is the most important and frequently studied substance. We developed techniques to extract quantitative nucleation rates  $J$  from supersonic nozzle experiments. First, we used pressure trace measurements to define the characteristic time, temperature, and supersaturation that correspond to the maximum nucleation rate. The average radius of the droplets in the supersonic nozzle is  $\sim 10$  nm, when they are fully grown. Therefore, we used small angle neutron scattering experiments to determine the number concentration of the aerosol that formed during the nucleation burst. After correcting for the difference in density between the viewing volume and the nucleation zone, the experimental rate  $J$  is simply equal to the aerosol number density divided by the characteristic time. Using nozzles with different expansion rates, we measured isothermal nucleation rates ranging from  $4 \times 10^{16}$  to  $3 \times 10^{17}$  as a function of supersaturation. Finally, the number of molecules in the critical cluster and the excess internal energy were estimated by applying first and second nucleation theorems, respectively.

**Poster S-6****Organization of enthalpically compatibilized nanocrystals in block copolymer/nanocrystal blends**

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The preferential sequestration of enthalpically compatibilized nanocrystals within block copolymer microstructures hold the promise to engineer mesoscopically ordered hybrid materials that capitalize from the physical properties of the template material and the unique optical properties of nano-sized matter. In our contribution we demonstrate the hierarchical pattern formation in ternary nanocrystal/block copolymer blends. The free energy and equilibrium structural parameters of the hybrid materials are derived as a function of block copolymer and particle characteristics. The theoretical results are compared to experimental data.

**Poster S-7****Stabilizing amyloid fibril formation by metal ions**Jijun Dong<sup>1</sup>, Jaby Jacob<sup>1</sup>, P. Thiyagarajan<sup>2</sup>, and David G. Lynn<sup>2</sup><sup>1</sup>Departments of Chemistry and Biology, Emory University; <sup>2</sup>Intense Pulsed Neutron Source, Argonne National Laboratory

Under certain conditions, small peptides can self-assemble to form long ordered supra-molecular fibrils, among them is included the amyloid  $\beta$  peptide (A $\beta$ ) and its derivatives. Full-length amyloid  $\beta$  peptides (A $\beta$ ) with 42 amino acids self-assemble to form unbranched, twisted or flat fibrils 100Å wide and several micrometers long. By shortening the peptide length or changing the solution conditions, the fibril morphology shown by electron microscopy is variable and controllable. Based on solid state NMR and small angle neutron scattering experiments, we have proposed a molecular model of the fibrils formed by A  $\beta$ (10-35), which is identical in primary sequence and fibril morphology to full-length amyloid  $\beta$  peptide. Understanding of the contributions of various factors that stabilize the fibril structure, especially on the lamination dimension, is important to control peptide self-assembly into fibrils with expected structure and function. In the poster we discuss the role of Zn<sup>2+</sup> in controlling both fibril assembly and morphology, characterized by several techniques, such as CD, HPLC, electron microscopy and small angle neutron scattering (SANS).

**Poster S-8****Dewetting of polyhedral oligomeric silsesquioxane (POSS) filled polymer films**

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Polymer thin films on substrates are used in a wide range of technological applications such as dielectric layers for semiconductor devices, fabrication of microelectronic and microelectromechanical devices, novel drug delivery systems, and biosensors. The performance of thin polymer films is dependent on their stability after being spread on a substrate. However, dewetting in polymer thin films pose a serious problem in nanoscale fabrication of polymeric coatings. An understanding of the mechanism of dewetting and its suppression is necessary to enhance the performance of polymer thin films. POSS, an organic-inorganic hybrid material has gained attention as nanofillers for high temperature and novel adhesive applications. Incorporation of POSS into polymer thin films alters the wetting properties of these films on surfaces. Langmuir-Blodgett films of trisilanolphenyl-POSS (TPP) filled poly (t-butyl acrylate) (PtBA) blends on silicon substrates have been used as a model system to investigate stability of filled polymer films on annealing at elevated temperatures. Optical and atomic force microscopy, and x-ray photoelectron spectroscopy studies of dewetting and phase separation as a function of film thickness were conducted for this study. The results indicate that the stability of the TPP filled films is highly dependent on film thickness, and nanofiller-polymer phase separation.

**Poster S-9****Interfacial properties of novel hybrid POSS-amphiphiles**

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With rigid inorganic cores and flexible organic substituents, polyhedral oligomeric silsesquioxane (POSS) molecules are ideal nanofillers for adhesive, high temperature, and aerospace applications. Open-caged POSS molecules, like trisilanol-POSS derivatives, exhibit surface activity at the air/water interface. Different substituents dramatically change their amphiphilic characteristics and lead to different monolayer and multilayer phase morphologies. Surface pressure-area per molecule isotherm and Brewster angle microscopy studies allow us to probe the phase diagrams of these materials. Furthermore recent results show that one of the POSS amphiphiles – trisilanolphenyl-POSS can form Langmuir-Blodgett (LB) films on solid substrates, which affords a feasible method to assemble well-defined POSS/polymer model films for studying interfacial interactions and properties of filler/polymer systems.

**Poster S-10****Self-assembly behavior of pullulan abietate**

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Wood is a complex biocomposite that exhibits a high work of fracture, making it an ideal model for multiphase man-made materials. Typically, man-made composites demonstrate interfacial fracture at failure due to abrupt transitions between neighboring phases. This phenomenon does not occur in wood because gradual phase transitions exist between regions of cellulose, hemicellulose, and lignin and therefore adhesion between adjacent phases is increased. The formation of interphases occurs as a consequence of the self-assembly process which governs the formation of wood. If this process was understood more thoroughly, perhaps tougher man-made, biobased composites could be prepared. To study self-assembly phenomena in wood, a system composed of a

model hemicellulose and a cellulose substrate has been prepared. The self-assembly of the polysaccharide pullulan abietate onto a regenerated cellulose surface prepared using the Langmuir-Blodgett technique can be studied via surface plasmon resonance (SPR). The adsorption behavior of pullulan abietate versus underivatized pullulan will be presented.

### **Poster S-11**

#### **Phase behavior of trisilanolisobutyl-POSS Langmuir-Blodgett films with polar PDMS derivatives at the air/water interface**

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Organic/inorganic hybrid nanocomposites are important materials for adhesive and high temperature applications. The key challenges are controlling dispersion and improving interfacial adhesion. By incorporating polar functional groups into both the nanofiller and matrix polymer, it is possible to modulate interactions between the two components. As a model system, polar poly(dimethylsiloxane) (PDMS) derivatives are studied as monolayer blends at the air/water interface with a polyhedral oligomeric silsesquioxane derivative, trisilanolisobutyl-POSS. Isotherm and Brewster angle microscopy studies allow direct imaging of morphological changes in the blend, which are correlated with both the type and number of functional groups on the PDMS.

### **Poster S-12**

#### **Polyelectrolyte-like behaviour of poly(ethylene-oxide) solutions with added monovalent salt**

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Solvent effects on the conformation of poly(ethylene-oxide) (PEO) and complexation of PEO by monovalent cations, have been examined by using small-angle neutron scattering. In methanol and acetonitrile, a big change in inter-chain interaction, osmotic compressibility and local chain conformation have been observed upon addition of small amounts of potassium iodide. The amplitude of the total intensity decreases significantly and a peak at a certain value of the wave vector  $q^*$  appears as signature of a polyelectrolyte-like behavior. With further addition of salt, the ionic strength of the solution increases and potassium binding becomes less favorable: the binding constant decreases with the ionic strength and PEO behaves as a neutral polymer with excluded volume. No association between PEO and potassium iodide was observed in aqueous solutions.

### **Poster S-13**

#### **Biomaterials high pressure system**

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We constructed a temperature controlled sample cell suitable for the study of biomimetic materials [e.g. lipid bilayers] using neutron diffraction, and capable of exerting hydrostatic pressures of up to 370 MPa. The advantage of this sample cell, compared to previous cells of its type, is that it allows for the use of samples aligned on a solid support [Si substrate] which, compared to 'powder' or liposomal preparations, require only small amounts of sample, and allows for the clear differentiation between in-plane and out-of-plane structures. The sample cell is machined from aluminium alloy 7075-T6. This alloy was chosen because aluminium is practically transparent to neutrons, and it has a very high yield strength [525 MPa]. Despite its high yield strength, 7075-T6 Al alloy is not suitably corrosion resistant, especially at elevated temperatures. To overcome this inherent deficiency, the sample cell was hard anodized to a total  $Al_2O_3$  plating of 50  $\mu$ m. Moreover, a 'sacrificial' zinc anode [cathodic protection] was installed to further minimize damage to the cell. Finally the cell was fitted with helicoil inserts in all pressure fittings, as there was measurable thread stretching with repeated use without them.

**Poster S-14****Dynamics of supercooled water in mesoporous silica matrix MCM-48-S**

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Using three different quasielastic neutron spectrometers with widely different resolutions, we have been able to study the microscopic translational and rotational dynamics of water, in a mesoporous silica matrix MCM-48-S, from T=300K to 220K, with a single consistent model. We formulated our fitting routine using the relaxing cage model. Thus, from the fit of the experimental data, we extracted the fraction of water bound to the surface of the pore, the characteristic relaxation times of the long-time translational and rotational decays, the stretch exponent describing the shape of the relaxation processes, and the power exponent determining the Q-dependence of the translational relaxation time. A tremendous slowing down of the rotational relaxation time, as compared to the translational one, has been observed.

**Poster S-15****Structure and dynamics of monolayer films of oligomer molecules adsorbed onto graphite.**

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The structure and dynamics of monolayer films of the aromatic molecules o-terphenyl (o-3P), p-terphenyl (p-3P) and p-quaterphenyl (p-4P) adsorbed onto the surface of graphite has been carried out using elastic and quasielastic neutron scattering. Structural results indicate an orientation consistent with the long molecular axis parallel to the graphite surface. Initial dynamic results indicate a phase change within both p-3P and p-4P films at approximately 300-325 K. At temperatures below 325 K, the scattering is consistent with reorientation of the phenyl rings around the long molecular axis. At temperatures above 300 K, the scattering is much stronger and appears at lower momentum transfers, indicating some form of translational motion in combination with reorientation of the phenyl rings. The o-3P films exhibit motions consistent with both reorientations of the phenyl rings as well as translations at temperatures as low as 190 K. There is no abrupt change in the character of the dynamics for the o-3P films.

**Poster S-16****Effects of molecular weight and temperature on the structure and interaction of triblock copolymer micelles in electrolyte solutions**

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We have investigated the phase behavior of 5 wt% aqueous solutions of triblock copolymers of PEO and PPO, Pluronic F38, F68, F88, F98 and F108, at different temperatures and sodium carbonate concentrations by using small angle neutron scattering (SANS). We determined the thermodynamic parameters of micellization by measuring the critical micellization temperature (CMT) as a function of F108 concentration in the presence of sodium carbonate. From SANS we obtained information on the size of the core and corona, inter-micelle distance, aggregation number and the volume fraction of the micelles. These studies show that temperature, polymer molecular weight and carbonate ion concentration have strong influence on the phase behavior of the triblock copolymers. The micelle aggregation number and the radii of the core and corona increase with increasing polymer

molecular weight. As expected, an increase in polymer molecular weight and/or the presence of carbonate ions decrease both the CMT and the temperature at which spherical to cylindrical micelle transformation occurs. The mechanism for micellization and spherical to cylindrical micelle transformation is the gradual dehydration of the copolymer chains with increasing temperature and/or salt concentration. Progressive dehydration occurs in the core at lower temperatures and progressive insertion of PEO units into the core occurs at higher temperatures.

## Instruments

### Poster I-1

#### Fast Exchange Refrigerator for Neutron Science (FERNS)

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Modern neutron science can exploit techniques that locate samples in a neutron beam while controlling experimental variables such as temperature, pressure, and magnetic field [1]. The Fast Exchange Refrigerator for Neutron Science (FERNS) instrument is being designed to enable efficient use of beamline resources, achieve a high throughput of samples, and meet needs expressed by the user community for an instrument that combines: (i) reliable cryogenic temperature control, (ii) automated sample exchange and storage, (iii) rapid thermal response, (iv) ease-of-use with various sample configurations. FERNS is designed to exploit the fast measurement capabilities of high flux beamlines by integrating automated sample handling and cryogenic temperature control *via* a computerized operating and sample management system. The instrument integrates computer controlled sample handling with a cryostat to enable automated sample exchange. The design also provides local and remote control and monitoring via a network computer connection. The use of a “pick-and-place” sample exchange system minimizes the thermal mass of cooled components. Work in progress includes: (i) consulting with users to identify requirements, (ii) constructing a prototype sample exchange instrument, (iii) demonstrating the system in T-range 4K-300 K, (iv) demonstrating exchange of 6 samples under computer control, (v) investigating requirements for thermal baffles, and (vi) developing draft advanced design concept for Phase II and performing preliminary design review (PDR). Longer term work is expected to include: implementation of an advanced design concept, investigation of using a “thermal switch” [2] to enable wider temperature range for samples, and pre-cooling of sample to reduce exchange time.

Refs:

1. I.F. Bailey, “A Review of Sample Environments in Neutron Scattering,” *Z. Kristallog.*, **218**, 84-95 (2003).
2. K.J. Volin and D.E. Bohringer, “A “Hot Stage” Displex: 20-800K in a Neutron Scattering Environment,” IPNS Equipment information.

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### Poster I-2

#### Design of a high-resolution macromolecular neutron diffractometer (MaNDi)

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Neutron macromolecular crystallography (NMC) can play a critical role in structural biology and functional genomics by providing accurate information on the positions of the protons and water molecules at active sites of enzymes that will enable the elucidation of the mechanistic details involved in their function. In order to exploit the high neutron flux that will become available by 2006 at the Spallation Neutron Source (SNS), and to leverage the enormous interest shown by the macromolecular crystallography community, it is proposed to develop a dedicated best-in-class high throughput and high resolution time-of-flight single crystal macromolecular neutron

diffractometer (MaNDi) at the SNS high power target station. Design calculations show that the data rates at the MaNDi instrument will be over 50 times greater than those for the best existing facilities. Furthermore, it will enable studies of crystals with larger lattice constants than is possible at the current facilities for NMC. It is expected that the unprecedented high data rates and resolution with MaNDi for the high resolution neutron macromolecular crystallography will greatly advance the fields of structural biology and enzymology.

### **Poster I-3**

#### **IPNS sample environment overview present and future**

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Development of sample environment laboratory equipment for neutron spectroscopy at the IPNS has steadily progressed over the past 22 years; in capabilities and in number. IPNS as well as outside scientific, engineering and technical staff have constructed a suite of furnaces, pressure cells/generators, cryogenic apparatuses, magnets, specialized equipment, etc. Many of which are standards among the neutron scattering facilities, directly contributing to the success of the IPNS scattering program. The IPNS is undergoing an enhancement plan which will include facility and instrument enhancements resulting in new areas of science and position IPNS to play a key role in increasing the US scattering community; readying them to effectively use next-generation capabilities at the SNS. This will put new scientific and operation demands on the IPNS sample environment group. Requests for more extreme physical parameters, quicker system equilibrium, and device monitor/remote control capabilities, etc. will be met by the SE group with a comprehensive management plan to maintain present equipment readiness and reliability and an engineering plan to address new technical challenges for proposed equipment. This will assist in insuring IPNS' success and preparedness for any new science coming over the horizon.

### **Poster I-4**

#### **The Spallation Neutron Source Backscattering Spectrometer**

Kenneth W Herwig and S. Keener, Spallation Neutron Source, Oak Ridge National Laboratory

When the Spallation Neutron Source is operational in 2006 at Oak Ridge National Laboratory, it will provide the most intense pulsed neutron beams in the world for research. This poster describes the expected performance of the near backscattering, crystal-analyzer spectrometer designed to provide extremely high energy resolution at the elastic peak ( $\delta\omega = 2.2 - 2.7 \mu\text{eV}$ , varying with sample dimension). The design requires a long initial guide section of 84 m from moderator to sample in order to achieve the requisite timing resolution. The design is optimized for quasielastic scattering but will provide 0.1 % resolution in energy transfer,  $\omega$ , up to  $\omega = 18 \text{ meV}$ . This spectrometer will provide an unprecedented dynamic range near the elastic peak of  $\pm 258 \mu\text{eV}$ . For experiments that require the full dynamic range available at comparable reactor based instruments (or greater), this spectrometer is expected to have an effective count rate  $\sim 64$  times that of the current best spectrometers, although at lower energy resolution.

### **Poster I-5**

#### **Time-Resolved Reflectometry at the Spallation Neutron Source**

John F. Ankner and Ch. Rehm, Spallation Neutron Source, Oak Ridge National Laboratory

The unprecedented peak neutron flux expected at the Spallation Neutron Source (SNS) promises to open up new fields of investigation. One of the more exciting of these is the study of time-resolved phenomena. Two reflectometers are planned for the initial suite of SNS instruments available in 2006, a vertical-surface polarized-beam reflectometer and a horizontal-surface reflectometer suitable for liquid studies. We have carried out extensive simulations of the expected performance of these instruments that indicate significant enhancements in data collection times relative to existing instruments (? 50 $\times$  faster in favorable cases with SNS running at 2 MW). We will quantify the implications of this faster data rate for conventional specular reflectivity measurements of varying spatial resolution and evaluate the feasibility and implementation of per-pulse data collection. We calculate outgassing from an Fe/Nb multilayer to evaluate the time-resolving capabilities of the liquids reflectometer.



**Poster I-6****SEQUOIA: A Fine Resolution High Energy Transfer Chopper Spectrometer for the SNS**  
Garrett E. Granroth, Spallation Neutron Source, Oak Ridge National Laboratory

A direct geometry fine resolution high energy transfer spectrometer (SEQUOIA) is being designed for installation at the SNS. The incident energy range of the spectrometer is  $E_i = 10 - 1000$  meV. Its primary monochromating device is a Fermi chopper that, along with the 5.5 m final flight path, will provide a minimum energy resolution of ( $DE/E < 1.5\%$ ). The long final flight path also provides fine Q resolution. The current details of the SEQUOIA design will be presented along with a comparison to other direct geometry spectrometers in design and under construction at the SNS.

**Poster I-7****Neutron Scattering Facility for Continuous High Magnetic Fields up to 40 Tesla at Hahn-Meitner Institut Berlin**

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The Hahn-Meitner-Institut has made great efforts to offer the users of its neutron scattering facilities at BENSC outstanding experimental conditions especially in sample environment: our possibilities range from experiments at high temperatures up to 1600 K to low temperatures down to 30 mK and high magnetic fields up to 15 T (for temperatures down to 1.5 K even 17.5 T). To further extend the range of continuous magnetic fields to be used for neutron scattering and because of limitations with superconducting technique, we have proposed a new facility with normal conducting magnets up to 40 T. The technical concept (40 MW power station, magnets, cooling circuit) will be described as well as results and perspectives of neutron scattering experiments at high magnetic fields (e.g. correlations in HTSC's or BEC of triplet states in  $TiCuCl_3$ ).

**Poster I-8****VISION – A neutron vibrational spectrometer for the Spallation neutron Source**

John Z. Larese, University of Tennessee

**Hard Materials****Poster H-1****Weld station**

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A multi-axis translation system accurately positions a 1018 carbon steel pipe sample in the incident neutron beam while a TIG torch lays down a continuous bead on the sample surface. The sample pipe can be positioned such that the weld pool, the heat affected zone, or the base metal may be probed by the neutron beam. Experiments have been conducted to examine both the changes in crystal structure as a function of proximity to the weld pool, as well stresses and strains created in the vicinity of the weld bead. Repeatable results relied on the stability of the weld arc. It was found several variables must be addressed in order to create an arc that is stable for the 1 ½ hour duration that an experiment required. Experiments were conducted to determine the best electrode composition and electrode tip geometry. The pipe samples were not concentric so a tracking torch mount was designed that maintained a constant electrode to work gap regardless of variations in the pipe dimensions. To prevent the sample pipe from heating excessively during an experiment a pre-chiller was installed on the backside of the pipe, it consisted of a shroud that directed a stream of cold helium gas over the sample thus cooling it to near room temperature before the material rotated into the weld bead and neutron beam.

## Poster H-2

### High temperature vacuum furnace

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The high temperature furnace is a compact lightweight instrument capable of continuous duty from 30C to 2000C. Its small volume and open geometry allows for rapid pump downs following sample changes. A number of specialized inserts have been incorporated into the furnace to provide unique sample environments. These inserts include; a sintering cell that pressurizes the sample to 900 torr with the selected gas and allows heating to 2000C. A similar insert, the purge cell, continuously flows gas around the sample during a heat cycle. The quench insert heats a sample to temperatures up to 1400C, and then on command quenches the sample at a rate of 50C / sec. For texture measurements a specimen rotation insert has been developed. An external stepper motor, via a ferrofluid feed through, drives a gear head in the furnace. The gear head in turn rotates a sample spindle allowing for continuously spinning of the sample in the neutron beam. Extensive testing and development has evolved a furnace that has high reliability and repeatability. Temperature gradients are minimal due to the combinations of heat shielding, reflectors and the heater design. Heater lifetime is several hundred hours, allowing for uninterrupted experiments lasting days.

## Poster H-3

### Neutron and x-ray diffraction studies of vitreous germania at high pressure

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Vitreous germania is an archetypal network glass. The structure comprises of a three-dimensional framework of corner-sharing tetrahedra which form small cages (or rings). At relatively low pressures below ~6 GPa dramatic changes in intermediate range order occur as the cages collapse. At higher pressures between ~6 and ~9 GPa a gradual transformation is observed from tetrahedral to six-fold coordinated Germanium. Since this transition is reversible, it can only be studied in-situ at high pressure. We present data from recent neutron and high energy x-ray diffraction experiments performed using the Paris-Edinburgh cell on GLAD at the IPNS and a Moissanite cell on BESSRC-CAT at the APS. The neutron data show a dramatic decrease in the first sharp diffraction peak with increasing pressure up to 5 GPa, indicating a breakdown of intermediate range order in the glass. The high energy x-ray data follow the polyamorphic transformation to completion, up to pressures of 15GPa. The two techniques provide complimentary structural information as neutrons are sensitive to the Oxygen correlations and the x-ray spectra are dominated by the Germanium interactions. Both of these high pressure techniques are likely to become important in future diffraction studies of oxide and non-oxide glassy materials under extreme pressures.

## Poster H-4

### Mn<sub>14</sub>Al<sub>56+x</sub>Ge<sub>3-x</sub> (x = 0 – 0.6): A New Intermetallic Phase Containing Unprecedented “Half-Broken” Mackay Icosahedra as Building Units.

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The new Mn<sub>14</sub>Al<sub>56+x</sub>Ge<sub>3-x</sub> (x = 0 – 0.6) compounds of a new structure type have been synthesized and characterized by physical property measurements and electronic structure calculations. In contrast to their well-known silicon analogues, their unique structure (P $\bar{3}$ ) exhibits unprecedented partially-destroyed Mackay icosahedra that retain the icosahedral symmetry only in one-half part of the individual polyhedra. The electronic band-structure analysis indicates that the chemical bonding in the structure is still optimized despite the destruction of the Mackay icosahedra, and that a further valence electron concentration (VEC) optimization is achieved by the partial occupation of aluminum on a germanium site. The weakly conducting property predicted from the electronic band-structure calculations is in agreement with the poor metallicity observed for the compounds. While the Mn<sub>14</sub>Al<sub>56</sub>Ge<sub>3</sub>

is metallic, the resistivity of  $\text{Mn}_{14}\text{Al}_{56.6}\text{Ge}_{2.4}$  undergoes more drastic changes as the temperature increases, showing a minimum around 20 K and a maximum around 100 K. Both of the compounds are Pauli-paramagnetic.

### Poster H-5

#### **Nanostructure and magnetic properties of the metallic glassy alloy $\text{Nd}_{60}\text{Fe}_{30}\text{Al}_{10}$**

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$\text{Nd}_{60}\text{Fe}_{30}\text{Al}_{10}$  metallic glasses are well-known for their ferromagnetic properties and remarkable thermal stability. Magnetic measurements reveal two ferromagnetic-to-paramagnetic transition, at ~ 50 and ~ 550 K indicating biphasic Nd-rich and Fe-rich regions. These results are corroborated by small-angle neutron scattering measurements, which reveal mass-fractal Nd-rich aggregates of size 15 nm and a magnetic correlation length of 34 nm in the ferromagnetic phase. The demagnetizing coercive field,  $H_c$ , is found to strongly depend on the quenching rate of the glass, and evidently by the extent of the two phases. The results are understood using the domain-wall pinning and random anisotropy models.

### Poster H-6

#### **Sample environment at the new neutron source FRM II**

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At the New Neutron Source FRM II first neutrons are expected in early autumn 2003. The poster presents recent development of sample environment at FRM II. Low temperature sample environment at FRM II basically operates with cryogen free systems. A Sample-Tube Cryostat was developed in close cooperation with the coldhead manufacturer VeriCold. Low temperatures are achieved by a Pulse Tube coldhead providing 0.4 W @ 4 K. The diameter of the sample tube is 50 mm. Specific features are easy handling, no use of cryoliquids and rapid sample exchange. A  $^3\text{He}$ -cryostat designed for operation in the Sample-Tube Cryostat was developed and constructed at FRM II as well as the appropriate TACO controlled handling system. In cooperation with the Walther Meissner Institute, Garching,  $^3\text{He}/^4\text{He}$  dilution inserts are under construction. A cryogen free Superconducting Split Coil Magnet is under construction at ACCEL GmbH, Germany. The magnet provides a vertical symmetric magnetic flux of 10 T, a split of 30 mm and a room temperature bore (RTB) of 100 mm. The large diameter of the RTB allows for the operation of other environment variables like high pressure, low temperatures (the sample tube refrigerator fits to the RTB) or chemical reaction cells. In cooperation with attocube, Germany, a small, high precision modular goniometer is under development. The piezoelectric, inertial motor stages allow precise alignment in the temperature range of 0.3 K to 400 K and magnetic fields up to 15 T. For high temperature applications, a furnace with appropriate system control was developed. Based on resistive heating, the Nb heating element combined with 5 Nb radiation shields yields in a high temperature limit of 2250 K. System Control is accessible via TACO.

### Poster H-7

#### **Latest developments in variable temperature cryostats for use in neutron scattering and diffraction experiments.**

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Driven by recent demands for more versatile cryostats that can be used to cool samples to cryogenic temperatures and, warm them up to room temperature and beyond, Janis Research Company had developed several models that are suitable for use in a neutron beam environment. In general, these can be divided into cryogen free units that make use of a variety of mechanical coolers, and units that require the use of liquid helium for cooling the samples to the desired temperature. The cryogen free units can either offer a vacuum environment for cooling the sample, or a more versatile exchange gas environment that allows quicker sample exchange, covering the temperature range 800 K to approximately 4 K. The liquid helium cooled units can be of the smaller continuous transfer type, that offer quick cool down, and the larger reservoir type units that can operate down to pumped helium

temperatures (~2 K), or He-3 temperatures (~0.3 K). The designs for each category will be discussed in some detail, with specific examples shown from each category, to illustrate the various configurations that are now available to the research community.

## Poster H-8

### High-Spin ( $S = 10$ ) organic molecule

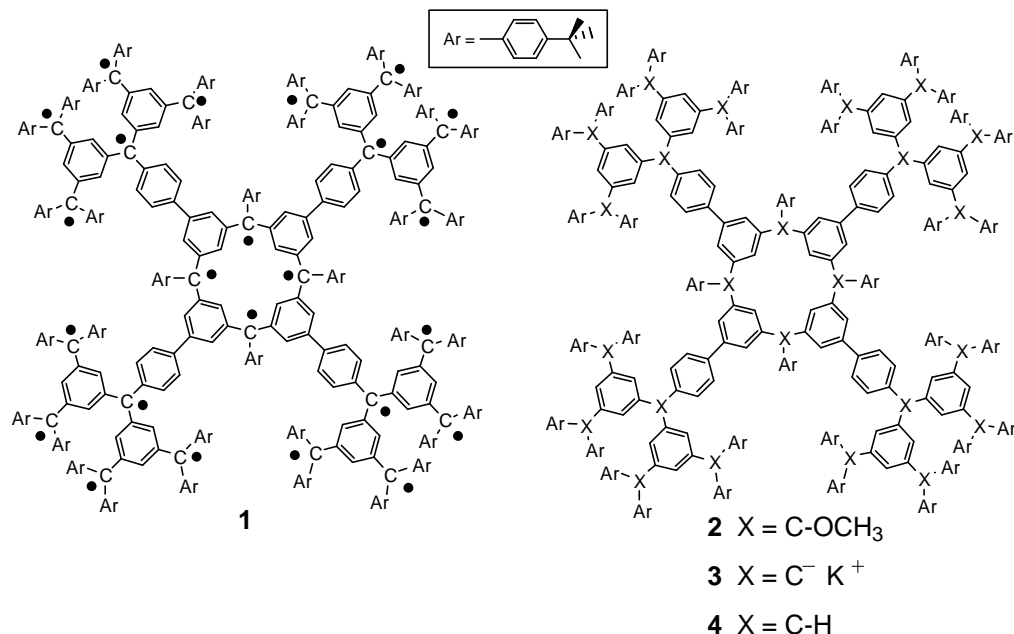
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The progress in the area of purely organic magnetic materials, based upon through-bond exchange coupling, has been measured by synthesis of high-spin molecules and polymers with the increasing values of spin quantum number  $S$ . The value of  $S = 10$  in molecule **1** is currently the highest among organic molecules.<sup>1</sup> Also, molecule **1** may be viewed as a model compound for recently reported first conjugated polymers with magnetic ordering.<sup>2</sup> Magnetic and SANS studies of **1** and its derivatives **2** – **4** in tetrahydrofuran- $d_8$  will be reported. Numerical fittings of the SANS data suggest similar, dumbbell-like shapes, for **1** – **4**, in qualitative agreement with conformational searches for **1** and **2**. Because **1** can only be handled in solution at or below 170 K and in the strict absence of oxygen, special procedures for sample handling on the beam line had to be devised. We acknowledge the National Science Foundation (CHE-9806954 and CHE-0107241) and the National Center for Neutron Research (beam time on NG3, grants No. 1781 and No. 2471) for the support of this research.



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<sup>2</sup>A. Rajca, J. Wongsriratanakul, S. Rajca, *Science* **2001**, *294*, 1503 – 1505.