Neutron Scattering Focus Topic
Room: East Exhibit Hall - Session NT-TuP

Neutron Scattering Poster Session

NT-TuP1 High Pressure and High Temperature Neutron Reflectometer Cell for Solid-Fluid Interface Research, ***P. Wang***, *D. Hickmott, A. Lerner, J. Majewski*, Los Alamos National Laboratory

To understand the interfacial behaviors of materials under high pressure (P) and temperature (T) are of great importance and interest since lots of natural phenomena and practical applications involve those conditions. For instance, mineral surface/fluid interactions control success or failure of many attempts to engineer Earth's subsurface for energy and/or environmental applications. The corrosion of metals and alloys in high subcritical aqueous systems, and especially in supercritical environments is an important safety issue in nuclear power plants.

However, due to the fact that most interface characterization techniques are difficult to implement at elevated P-T, little experimental attention has focused on solid/fluid interfaces at high P-T.

Neutron reflectometry (NR) is increasingly being used as a characterization tool to study the surface/interface of planar substrates. SPEAR at Lujan center is a Time-of-Flight (ToF) NR facility which is specifically designed to study solid-liquid interface and is able to in-situ monitor the surface/interface behavior with a space resolution of a few angstroms. The obtained real space model from reflectivity curve fitting can provide a lot of physical and chemical information about the interface.

One key gap to study the interfacial behaviors of materials under high P-T conditions is the lack of well designed pressure cell capable of handling P-T conditions close to or above supercritical conditions. To build up the capability of studying high P-T surface/interface, Lujan Center developed a special designed pressure cell which allows us to reach 200 MPa and 250 °C (in the future such cell will be equipped with with *in-situ* spectroscopic Raman and IR capabilities). Neutron is highly penetrating, which is able to "see through" high P-T aluminum cell walls and examining the surface/interface properties. Besides the pressure cell itself, the high P-T cell system includes three other subsystems: temperature, pressure and sample chamber environment control systems.

NT-TuP2 Polarized Neutron Reflectivity of Exchange Inversion Layers, ***H. Lee***, *J. Yu, N. Pachauri, S. Keshavarz, P. LeClair, G.J. Mankey*, University of Alabama, *H. Ambaye, V. Lauter*, Oak Ridge National Laboratory

Bulk FeRh undergoes an antiferromagnetic to ferromagnetic phase transition as it is heated above room temperature. The addition of Pd lowers the phase transition temperature so that, in thin film form, the details of the phase transition can be studied while maintaining the same structural and morphological properties of the as-deposited film. The FeRhPd thin film was prepared by DC magnetron sputtering in an ultraclean sputtering system. A FeRhPd/Pt/FeRhPd trilayer was grown at 600ºC on an a-axis sapphire substrate with a Rh seed layer and a Pt buffer layer. The epitaxial orientation of this 111-oriented thin film was confirmed by X-ray diffraction methods including standard high-angle diffraction, rocking curve analysis and pole figure analysis. The first-order metamagnetic phase transition and thermal hysteresis of the magnetic moment were examined by vibrating sample magnetometery. To study the detailed magnetic structure of a trilayer with a Pt spacer between two epitaxial films we applied polarized neutron reflectivity (PNR). PNR is used to detect the magnetic moment distribution in layered structures. Temperature-dependent PNR showed the dependence of ferromagnetic spin-splitting for the neutron reflectivity of the two spin polarization channels. Fitting of the PNR data shows a change of the spin splitting that is consistent with vibrating sample magnetometry data. PNR measurements at two different applied magnetic fields of 1 T and 0.5 mT revealed the dependence of magnetic splitting on applied magnetic field and a modification of the thermal hysteresis. This data confirms the strong field dependence of the magnetically stable state. Analysis of the off-specular neutron reflectivity data will show how the magnetic domains change with experimental conditions. The authors gratefully acknowledge financial support from DOE award DE-FG02-08ER46499. Research at Oak Ridge National Laboratory's Spallation Neutron Source was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

Neutron Scattering Focus Topic
Room: 207 - Session NT+AS+MI-WeM

Applications of Neutron Scattering I

Moderator: V. Lauter, Oak Ridge National Laboratory

8:00am **NT+AS+MI-WeM1 A Deeper Look into Spintronic Material Systems with Neutrons and Synchrotron Radiation**, ***T. Brueckel***, Forschungszentrum Jülich, Germany **INVITED**

The discovery of the Giant Magnetoresistance GMR effect triggered the evolution of Spintronics, i.e. information storage, information processing and information transport using the spin of the electron. While the first Spintronic devices were merely transition metal multilayers, the interest has shifted to include transition metal oxide systems, laterally structured films and magnetic nanoparticles. Scattering techniques applied to model systems are ideal to provide fundamental microscopic information on the spin and domain structure.

In this contribution, we will give an overview highlighting the capabilities of modern neutron and synchrotron x-ray techniques. We will show that neutron scattering under grazing incidence is able to provide unique depth resolved information on magnetization, magnetic correlations and magnetization dynamics relevant for basic and applied research on nanostructured magnetic materials and how synchrotron x-ray scattering can provide complementary element specific information. Examples for current research on patterned metallic multilayers, thin transition metal oxide films and magnetic nanoparticles will be given.

8:40am **NT+AS+MI-WeM3 Magnetic Properties of FePtRh Films and Multilayers Studied by Neutron Scattering**, ***D. Lott***, *J. Fenske*, Helmholtz-Zentrum Geesthacht, Germany, *G.J. Mankey*, Univ. of Alabama, *W. Schmidt*, *K. Schmalzl*, Forschungszentrum Juelich, Germany, *E. Tartakowskaya*, National Academy of Science, Ukraine, *H. Amabye*, ORNL, *F. Klose*, *A. Mulders*, ANSTO, Menai, Australia, *A. Schreyer*, Helmholtz-Zentrum Geesthacht, Germany, *V. Lauter*, ORNL

Ordered FePt alloys with $L1_0$ structure are known as materials with FM order and a high magnetic moment of Fe providing a large magnetization. The large atomic number of Pt on the other hand results in a high magnetic anisotropy. If grown in thin films, the high anisotropy often results in perpendicular magnetization which is the preferred orientation for current magnetic recording media. One way to control the magnetic properties in these materials is through the introduction of a third element into the crystal matrix e.g. Rh. When Rh is added to replace Pt in the equiatomic alloy, new magnetic phases emerge. Here neutron diffraction studies on the magnetic properties of different thick $Fe_{50}Pt_{50-x}Rh_x$ films in dependence on temperature and external magnetic fields allowed us to investigate the rich phase diagram of the system for thin films, e.g. the transition from the FM to AF state in the system with increasing Rh concentration. In particular films with a Rh concentration of about 10% show a temperature dependent AF-FM transition. From the neutron data it was moreover possible to determine the magnetic configurations in dependence on concentration, temperature and magnetic field on a microscopic scale. Based on the observed results a theoretical model considering the changes in the anisotropies could be developed. In a next step magnetic multilayer consisting of $Fe_{50}Pt_{50-x}Rh_x$ bilayers with different Rh concentrations were grown and studied by polarized neutron reflectivity to investigate the magnetic interactions along the lattice matched interfaces. First results will be presented here.

9:00am **NT+AS+MI-WeM4 Spectroscopic and Magnetic Characterization of the Spin-Crossover Transition in Thin Films of $Fe(C_{12}H_8N_2)_2(NCS)_2$.**, ***E.C. Ellingsworth***, *G.J. Szulczewski*, The University of Alabama, Tuscaloosa, *V. Lauter*, Oak Ridge National Laboratory

The octahedral complex bis(1,10-phenanthroline)dithiocyanate iron(II), $Fe(phen)_2(NCS)_2$, is known

to exhibit an abrupt transition between a high and low magnetic spin state from 170 – 180 K in the

bulk phase. As a result, $Fe(phen)_2(NCS)_2$ is an interesting organic semiconductor to study charge and

spin transport in thin films. We synthesized and characterized $Fe(phen)_2(NCS)_2$ according to literature

procedures. Thin films of $Fe(phen)_2(NCS)_2$ were made by vapor deposition onto a variety of substrates

including Si, KBr, Au and Al and characterized by infrared and photoelectron spectroscopy, SQUID

magnetometry, optical microscopy, and polarized neutron reflectometry. The films were found to be

very sensitive to water vapor under ambient conditions, which complicates the structural, chemical and

magnetic analysis of the films. However, appropriate capping layers can be deposited onto the

$Fe(phen)_2(NCS)_2$ thin films to protect them from water vapor before removal from the vacuum system. The

differences in the magnetic behavior of the thin films will be compared to the bulk phase.

9:20am **NT+AS+MI-WeM5 Nanoscopic Magnetic Phase Separation at the $SrTiO_3(001)/La_{1-x}Sr_xCoO_3$ Interface**, *M. Sharma*, *M.A. Torija*, Univ. of Minnesota, *J. Gazquez*, *M. Varela*, ORNL, *J. Schmitt*, *C. He*, Univ. of Minnesota, *J.A. Borchers*, *M. Laver*, NIST, *S. El-Khatib*, American University of Sharjah, *V. Lauter*, *H. Ambaye*, *R. Goyette*, ORNL, ***C. Leighton***, Univ. of Minnesota **INVITED**

The remarkable functionality of complex oxides, when combined with the favorable lattice matching that is possible at their interfaces, provides many opportunities for new physics and applications. The perovskite manganites and cobaltites are excellent examples, being of interest in gas sensing, catalysis, and as electrodes in ferroelectric memory and solid oxide fuel cells. From the magnetism perspective they have potential for high conduction electron spin polarization, and a variety of functional ground states. However, the same delicate balance between phases that provides such impressive functionality also leads to a serious problem; it can be difficult to maintain desired properties (e.g. high spin polarization and conductivity) close to the interface with a dissimilar oxide. This is exemplified by magnetic tunnel junctions for example, where the interface spin polarization is suppressed and drops rapidly with temperature. In this work, using SrTiO3(001)/La1-xSrxCoO3 [1] as a model system, we have combined epitaxial growth by high pressure oxygen sputtering with atomic-level stuctural characterization (including STEM/EELS imaging [2]), conventional magnetometry, electronic transport, small-angle neutron scattering, and polarized neutron reflectometry. We observe the usual degredation in magnetization and conductivity in the very thin film limit. We demonstrate that this is due to nanoscopic magnetoelectronic phase separation in the interface region [3]. Essentially, nanoscopic ferromagnetic (FM) clusters form in an insulating non-FM matrix near the interface, resulting in reduced magnetization and conductivity, even at compositions that display no such phase separation in bulk. STEM/EELS depth profiling of the chemical composition reveals that this effect has a chemical origin, being due to subtle depth-wise variations in Sr and O content, resulting in reduced hole doping near the interface. Simple thermodynamic and structural arguments for the origin of these variations are provided, based on Sr dissolution energies and the critical link between srain state and O vacancy concentration provided by O vacancy ordering [2,3].

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[1] Torija, Sharma, Fitzsimmons, Varela, Wu and Leighton, *J. Appl. Phys.* **104** 023901 (2008).

[2] Gazquez, Luo, Oxley, Prange, Torija, Sharma, Leighton, Pantiledes, Pennycook and Varela, Nano. Lett. **11** 973 (2011).

[3] Torija, Sharma, Gazquez, Varela, He, Schmitt, Borchers, Laver, El-Khatib, Maranville and Leighton, published online, Adv. Mater. (2011).

10:40am **NT+AS+MI-WeM9 Study of $L1_0$ Ordering in $^{57}Fe/Pt$ Multilayers**, ***K. Srikanti***, Ugc-Dae, Csr, India

Ordered $L1_0$ FePt, FePd and CoPt alloy thin films have large magnetic anisotropy constants suitable for high-density recording media. The $L1_0$ ordered phase is obtained with post growth annealing at high temperatures. However, the high temperature annealing leads to grain growth. The present work is an attempt to lower the transition temperature starting with multilayer precursors. The evolution of the structural and magnetic properties of Si(sub) [^{57}Fe(19Å

)/Pt (25Å)]$_{x10}$ multilayers as a function of vacuum annealing at different temperatures is studied. The film thickness is selected to have equi-atomic stoichiometry. The multilayers are prepared by ion beam sputtering. X-ray

reflectivity (XRR), X-ray diffraction (XRD), and magneto optical Kerr effect (MOKE) and conversion electron Mössbauer spectroscopy (CEMS) are used to characterise the as-deposited and annealed multilayers. Using XRR it is observed that due to intermixing FePt alloy formation takes place with annealing. The XRD indicated the presence of superstructure peaks at 350^0C and above.Mössbauer measurements indicated a clear evidence for the strong exchange coupling between the soft fcc FePt and hard fct FePt phase. The results indicate that the multilayer structure does not transform directly to the ordered fct FePt, rather first an fcc FePt phase is formed and subsequently it gets converted in to the fct FePt phase as a function of annealing. A detailed study on stoichiometric FePt system embedded in Carbon matrix prepared by ion beam sputtering technique will also be discussed.

Few preliminary results of polarised neutron reflectivity measurements on FePt/FeNi exchange spring magnets will be discussed.

11:00am **NT+AS+MI-WeM10 Influence of Capping Layer Rigidity on Properties of Supporting Temperature Sensitive Hydrogel Polymers Using Neutron Reflectivity**, ***M. Dubey***, Los Alamos National Laboratory, *M.S. Jablin*, Carnegie Mellon University, *M. Zhernenkov*, Los Alamos National Laboratory, *R. Toomey*, University of South Florida, *J. Majewski*, Los Alamos National Laboratory

Temperature sensitive hydrogel polymers are utilized as responsive layers in various applications. While the polymer's native characteristics have been studied extensively, details concerning its properties during interaction with bio-related structures are lacking. This work investigates the interaction between a thermoresponsive polymer cushion and different lipid membrane capping layers probed by neutron reflectometry. N-isopropylacrylamide copolymerized with methacroylbenzophenone first supported a lipid bilayer composed of 1,2-Dipalmitoyl-*sn*-Glycero-3-Phosphoethanolamine (DPPE) and subsequently 1,2-Dipalmitoyl-*sn*-Glycero-3-Phosphocholine (DPPC). The polymer-membrane systems were investigated above and below the polymer lower critical solution temperature (37 and 25 °C). While the same cushion supported each lipid membrane, the polymer hydration profile and thickness were markedly different for DPPE and DPPC systems. Since DPPE and DPPC have different bending rigidities, these results establish that the polymer-membrane interaction is critically mediated by the mechanics of the membrane, providing better insight into cell-hydrogel interactions. There has been increased interest in the effect of matrix elasticity on cell lineage specification. Polymeric matrices with known stiffness are utilized as supports to understand the physical effects of *in vivo* tissue microenvironment for therapeutic uses of stem cells. This work focuses on the infleunce of a capping layer on the mechanical properties of the underlying support.

Neutron Scattering Focus Topic
Room: 207 - Session NT+AS-WeA

Applications of Neutron Scattering II

Moderator: J. Majewski, Los Alamos National Laboratory

3:00pm **NT+AS-WeA4 Probing Fractals by the Combined Ultra-Small- and Small-Angle Neutron Scattering (USANS/SANS) Technique**, ***M. Agamalian***, Oak Ridge National Laboratory

Many natural and man-made materials exhibiting multi-level morphology (atoms – molecules – aggregates – agglomerates), in other words, existence of intermediate structural units between atomic/molecular and macroscopic levels, usually call hierarchical structures. The combined USANS/SANS is one of the best techniques using at present time for characterization of the hierarchical structures, which in many cases shows fractal behavior. The current presentation is focused at the mass and surface fractals discovered experimentally in the sedimentary rocks, attractive colloidal glasses and aggregates of soot particles in MCT-30 engine oil. Some of the fractal structures, particularly the surface fractals in rocks, are extended over three orders of magnitude in the length scale; therefore, application of the combine USANS/SANS technique, which covers the Q-range extended over five orders of magnitude in the reciprocal space ($2x10^{-5}$ $Å^{-1}$ < Q < 1 $Å^{-1}$), is required to obtain complete structural information for complicated hierarchical structures with fractals.

4:20pm **NT+AS-WeA8 Interaction of Alzheimer's Disease Tau Protein with Model Lipid Membranes**, *E.M. Jones,* Univ. of New Mexico, *M. Dubey,* Los Alamos National Lab, *P.J. Camp, B.C. Givler,* Univ. of New Mexico, *J. Biernat, E. Mandelkow,* Max Planck Unit for Structural Biology, Germany, *J. Majewski,* Los Alamos National Lab, ***E.Y. Chi***, Univ. of New Mexico **INVITED**

In addition to amyloid plaques, tau neurofibrillary tangles comprise another pathological hallmark of Alzheimer's disease (AD). The mechanism of tau's misfolding and aggregation is unknown, but evidence suggests that tau in AD brains may abnormally interact with the neuronal cell membrane. Using lipid monolayers at the air/water interface and supported lipid bilayers as model membrane systems, we characterized the interaction between 4 tau constructs with membranes of different lipid compositions and elucidated the structure of the protein-membrane films using a combination of biophysical techniques, including pressure-area isotherms, fluorescence microscopy, and x-ray and neutron scattering. Our data show that the full length human tau (hTau40) and its constructs are highly surface active and exhibited strong association with negative DMPG lipids and induced morphological changes observed with fluorescence microscopy, while exhibiting weaker and no interactions with positive DMTAP and neutral DMPC lipids. To elucidate molecular-scale structural details, we used X-ray scattering techniques to study tau and lipid monolayer association. X-ray reflectivity modeling indicated hTau40's presence under a DMPG monolayer and partial insertion into the lipid headgroup region, while grazing incidence X-ray diffraction data showed hTau40 insertion disrupted lipid packing. We also used neutron reflectivity assays to investigate hTau40's ability to disrupt lipid bilayers. The protein completely disrupted a DMPG bilayer while not affecting a neutral DPPC bilayer. These results indicate hTau40 has a propensity to interact with a negatively charged membrane surface and disrupt lipid packing, suggesting a possible protein-aggregate induced mechanism for aggregation and toxicity.

5:00pm **NT+AS-WeA10 Stabilization of a Lipid Multilayer System by Polysaccharides**, *M. Kreuzer, M. Strobl,* University of Heidelberg, Germany, *M. Reinhardt, R. Steitz,* Helmholtz-Zentrum Berlin für Materialien und Energie, Germany, ***R. Dahint***, University of Heidelberg, Germany

Hyaluronic acid (HA) is a high molecular weight polysaccharide. It is involved in a wide range of processes in the human body, such as wound healing, tumor progression and joint lubrication. Here we show that HA also stabilizes a lipid multilayer system at physiological conditions. The observed effect may be an important contribution to joint lubrication as lipid films covering the cartilage of natural joints are assumed to reduce internal friction. Neutron reflectometry investigations were carried out at V6 and the new BioRef neutron reflectometer at Helmholtz-Zentrum Berlin. Measurements against excess D_2O verified, that an oligolamellar DMPC lipid bilayer coating remains stable on a silicon substrate at 21 °C in its ordered state (L_{β}', P_{β}') with a d-spacing of 66 Å, but detaches almost completely from the solid support at 38 °C in its chain-disordered state (L_{α}). By contrast, oligolamellar lipid bilayers remain stable on a substrate at 38 °C when incubated with a solution of HA in D_2O. Lamella transformations occur over time, resulting in a new lamella phase with a d-spacing of 233 Å. This effect has to our knowledge not been reported before on solid-supported oligolamellar systems. We will discuss potential consequences of the "new" lamella phase with respect to further insight into joint lubrication.

5:20pm **NT+AS-WeA11 Neutron Reflectometry, QCM-D, and TIRF Study of the Interaction of Endoglucanases with Films of Amorphous Cellulose**, ***M. Kent***, Sandia National Laboratories **INVITED**

Cellulase enzyme cocktails include exoglucanases that digest cellulose chain ends and endoglucanases that cleave randomly at interior points along the chains. While it is known that these enzymes work synergistically, the details are not fully understood. In addition, cellulose binding domains (CBDs) are known to play an important role in the digestion of crystalline cellulose but much less is known about the benefit of CBDs in the digestion of amorphous cellulose. Amorphous cellulose is of interest as pretreatment of biomass with ionic liquids, a promising next generation technology, results in a combination of amorphous cellulose and cellulose II. Determining the full effects of endoglucanase activity is challenging because these enzymes can alter the structure of insoluble cellulose in addition to releasing soluble oligomers. To unravel the actions of endoglucanases and the role of cellulose binding domains in enhancing activity on amorphous cellulose, we have combined studies of the profile of water through cellulose films during digestion by neutron reflectivity, measurements of changes in mass and film stiffness using a quartz crystal microbalance (QCM), and visualization of the motion of individual enzymes by total internal reflection fluorescence (TIRF) microscopy.

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