Materials@LU Symposium 2014 – Program

09.00 Introduction
Håkan Hallberg, Coordinator of Materials@LU
Research Portals at Lund University
Annika Olsson, Vice Dean LTH
Materials Research and Innovation
Maryam Olsson, LU Innovation System
Materials Research Using Synchrotron Radiation
Axel Steuwer, Maxlab

10.00 Coffee

10.15 The Nanometer Consortium at Lund University
Heiner Linke, nmC
Materials Research at the Nanometer Consortium
Magnus Borgström, nmC

11.00 Presentation Session 1
- Filip Lenrick, Div. of Polymer & Materials Chemistry: *Materials analysis using Focused Ion Beam (FIB), Scanning Electron Microscope (SEM) and a micro-manipulator probe*
- Johan Hektor, Div. of Solid Mechanics: *X-ray diffraction measurement of strain around a tin whisker*
- Lorenzo Grassi, Div. of Biomedical Engineering: *Full-field strain measurement on cadaver human femora using Digital Image Correlation*
- Alva Thorell, Div. of Building Materials: *Water sorption thermodynamics of proton exchange membranes (PEMs) studied by calorimetry*

12.15 Lunch

12.45 Presentation Session 2
- Masha Saeidpour, Div. of Building Materials: *Effect of hysteresis and supplementary cementitious materials on transport properties of cement materials*
- Marianna Yanez, Div. of Physical Chemistry: *Nanostructured Surfaces Created by the Interactions of Dendrimers and Nucleolipids: Recognition of Biomolecules*
- Wureguli Reheman, Div. of Solid Mechanics: *Stress induced hydride formation and surface instability*
- Jemila Habainy, Div. of Materials Engineering: *Properties of Spallation Material exposed to a High Flux of High-Energy Proton and Spallation Neutron irradiation*
- Annika Weiber, Dept. of Chemistry/Centre for Analysis and Synthesis (CAS): *High-performance polymer membranes*

14.15 Coffee

14.30 Presentation Session 3
- Hossein Sina, Div. of Materials Engineering: *Studies on the formation of aluminides in heated Nb-Al powder mixtures*
- Matilda Larsson, Dept. of Chemistry/Centre for Analysis & Synthesis (CAS): *Bioplastics for the Next Generation of Packaging Materials*
- Yujing Li, Div. of Building Materials: *System dynamic study of mould growth on building materials*
- Abdallah Shokry, Div. of Solid Mechanics: *A Methodology for using Kalman Filter to Determine Material Parameters from Uncertain Measurements*
- Kenneth Frogner, Div. of Production & Materials Engineering: *Development and characterization of composite materials*

16.00 Closing

Time
June 5th 2014, 09.00-16.00

Place
Belfragesalen, floor 15 of house D at the Biomedical Center (BMC) on Klinikgatan 32 in Lund

For more information, please visit: **materials.lth.se/symposium2014**

Presentation Session 1

Materials analysis using Focused Ion Beam (FIB), Scanning Electron Microscope (SEM) and a micro-manipulator probe

Filip Lenrick, Div. of Polymer & Materials Chemistry

Focused Ion Beam (FIB) coupled with a Scanning Electron Microscope (SEM) and a micro-manipulator probe is a very versatile instrument set-up for material analysis, sample preparation and micro-device assembly. It has the possibility to sputter etch with a precision down to a few nanometer, deposit a variety of different materials with equal precision and image these processes in real time using SEM.

In this talk a short introduction to the FIB/SEM instrument and some of its uses will be given. Where sample preparation for Transmission Electron Microscopy (TEM), FIB tomography and 3D reconstruction, and single nanowire manipulation are a few examples.

X-ray diffraction measurement of strain around a tin whisker

Johan Hektor, Div. of Solid Mechanics

Metal whiskering is a microstrucural phenomenon where filiform grains grow spontaneously out of a surface. Whiskers have been found on a number of different metals, such as zink, silver and cadmium. A particularly interesting type of whiskering occurs on tin plated copper. Tin whiskers are a problem mainly in the electronics industry because of short circuits caused by whiskers bridging two contacts. Until recently, the solution to the problem was to alloy tin with lead, which prevents the formation of whisker. Due to environmental concerns, the use of lead in consumer products is now prohibited in the European Union. As a result there is a renewed interest to better understand the mechanisms causing whisker growth to identify alternatives to the tin-lead alloy.

The mechanism for whisker formation is not yet fully understood, but it is thought that the stress state of the Sn layer is playing a key role, and that the whisker growth is a stress relief phenomenon. To measure the strain around a whisker X-ray Laue microdiffraction has been used. This technique allow for good spatial resolution in all three spatial dimensions.

Full-field strain measurement on cadaver human femora using Digital Image Correlation

Lorenzo Grassi, Div. of Biomedical Engineering

Strain gauges (SG) are the gold standard for strain measurement in bones, but the number of measurements is limited. Digital image correlation (DIC) can provide thousands of measurements using a non-contact method. The aim of the study was to investigate the strain response of a cadaver human femur using DIC.

A proximal cadaver femur was obtained, and prepared for DIC recording by painting a white background over the anterior surface, with a random black speckle pattern. The bone was loaded in a quasi-axial configuration at 15mm/s. DIC images were recorded at 3000 fps, 1MPx, and correlated using Vic 3D 2010. Simulated SGs were identified to compare our data with literature.

DIC provided ~50000 points. The force-displacement curve was linear (R2=0.99), and the principal strains from simulated SGs were also correlated with the force (R2>0.94).

Based on our results, the set-up provides extensive information about the strain evolution under fast loads, and a benchmark for FE model validation.

Water sorption thermodynamics of proton exchange membranes (PEMs) studied by calorimetry

Alva Thorell, Div. of Building Materials

Ionomeric membranes such as Nafion are used as electrolytes in PEM fuel cells. Their proton conductivity depend on the degree of hydration, as their internal structure changes with the hydration number, λ (mol water /acidic group).

The water-membrane interactions can be studied by using an isothermal sorption calorimeter developed at Lund University. In the sorption ampoule, the evaporation chamber with water is connected to a sorption chamber containing dried sample. The thermal effects from the evaporation (Pvap) and the sorption (Psorp) are measured, and from these the thermal effect of mixing (Psorp-Pvap), λ and the water activity as a function of time can be calculated.

The described technique allows for identification of changes in sorption energy as a function of λ. Measurements on Nafion dried at room temperature show a continuous change in the sorption energy, while drying at 120°C results in more complex mixing enthalpies. The peaks/plateaus may be associated with different binding energies of the absorbed water, but could also be non-equilibrium effects due to moisture-induced structural changes. The origin of these effects will need further investigation.

Presentation Session 2

Effect of hysteresis and supplementary cementitious materials on transport properties of cement materials

Masha Saeidpour, Div. of Building Materials

Portland cement production is responsible for 5% of global man made CO2 emissions. The CO2 emissions mostly come from burning fuel during clinker production. The solution to this problem is replacing Portland cement with supplementary cementitious materials (SCMs) such as slag, silica fume, fly ash and natural pozzolans.

Most of the important deterioration mechanisms of concrete structures involve water vapour transport. Diffusion coefficient of water vapor in cement based materials is a function of moisture content. Presences of SCMs change the nanostructure and also transport properties of material. In this project we quantify the moisture dependency of water vapor diffusion coefficient in presence of SCMs in different absorption/desorption cycles.

Nanostructured Surfaces Created by the Interactions of Dendrimers and Nucleolipids: Recognition of Biomolecules

Marianna Yanez, Div. of Physical Chemistry

Poly(amidoamine) PAMAM dendrimers have been studied extensively due to their well-defined hyperbranched structure and their cationic charge that make them potential vehicles for gene therapy. Novel nucleolipids, dilauroylphospholiponucleosides (DLPN) derivatives, have generated interest due to their capacity for base pairing with DNA and RNA, which ordinary PAMAM do not have. We investigated the interactions between PAMAM generation 4 and DLPNs based in uridine (DLPU) and adenosine (DLPA) at the solid-liquid interface and the ability of the formed layers of recognizing biomolecules. The data show that the layers formed depend on the type of nucleotide and the adsorption protocol. The layers containing DLPA interacted selectively with the biomolecules while DLPU layers did not show any sign of interactions under the conditions employed. The present work represents an alternative pathway for the development of gene therapy vehicles by combination of PAMAM dendrimer with nucleolipids that will open up the possibility to tune the structure of the formed complexes as well as to provide means for molecular recognition without extensive synthesis procedures.

Stress induced hydride formation and surface instability

Wureguli Reheman, Div. of Solid Mechanics

When the structural materials exposed to long term hydrogen environment they may interact with hydrogen, and cause various kind of structural damages due to the metal degradation. One of well-known hydrogen damage is hydride embrittlement. When the hydrogen concentration in the matrix exceeds a terminal solid solubility, it starts to form a hydride, e.g. zirconium, titanium and vanadium. In the metals the precipitation of small and local amounts of hydrides strongly affect the local mechanical properties and the overall integrity of the entire structure. Studies of this phenomenon follow a complicated chain of dependencies, e.g. diffusion, temperature and stress. In the present study the stress induced formation of hydride at the crack tip is investigated. A 2D linear elastic model developed to predict the hydride formation zone near crack tip. A finite element calculation is performed to compute the near crack tip stress field. The result shows that the shape of hydride growth in a self-similar manner, also the interface is not stable.

Properties of Spallation Material exposed to a High Flux of High-Energy Proton and Spallation Neutron irradiation

Jemila Habainy, Div. of Materials Engineering

Tungsten has been chosen as the spallation material for the European Spallation Source target. The target will be cooled by helium and operate under harsh thermo-mechanical conditions with proton irradiation corresponding to 5MW beam power and a time-averaged maximum operating temperature ranging between 400° and 500°C.

The aim of this project is the characterization of the irradiated tungsten exposed to a high flux of high-energy protons and spallation neutrons. A study of the defects and their impact on mechanical properties will be carried out.

Currently the fatigue and oxidation behavior of unirradiated tungsten reference material is being investigated. So far the results show that helium with an oxygen impurity of 5 ppm is not protective enough at these temperatures, and that the fatigue limit for this material is highly dependent on the manufacturing method. Tests also show that the electro polished samples have a much higher ultimate tensile strength than the unpolished ones – the mean values of UTS for samples in the rolling direction are 705 MPa and 397 MPa, respectively.

High-performance polymer membranes

Annika Weiber, Dept. of Chemistry/Centre for Analysis and Synthesis (CAS)

There is a growing need for new robust ion exchange membranes in order to advance energy related technologies such as fuel cells and batteries. These polymeric materials must possess high thermal and mechanical stability in combination with efficient ion transport properties. Our research group has since many years designed, synthesised and characterised high-performance polymers and membranes functionalised with various anionic and cationic groups. We study structure-property relationships using a wide range of techniques such as calorimetry, X-ray scattering and impedance spectroscopy. Recently, we have developed strategies to synthesise aromatic polymers with controlled distributions of sulfonic acid and quaternized ammonium groups along the chains, and studied how this influences membrane properties. Thus, water uptake and ion conductivity were measured and correlated with molecular structure and morphology to show significant advantages of high local ionic concentrations.

Presentation Session 3

Studies on the formation of aluminides in heated Nb-Al powder mixtures

Hossin Sina, Div. of Materials Engineering

The formation of aluminides during the heating of Nb-Al powder mixtures with different initial compositions (25, 33.33 and 75at.% Al) has been studied using a differential scanning calorimeter. The effect of parameters like particle size, compaction and the heating rate on the onset temperature of the reaction has been determined. The results show that an increase in heating rate leads to an increase in the onset temperature for compacted as well as loose powder samples in the particle size range considered. For Al-rich mixtures, compaction increases the onset temperature irrespective of particle size. For all samples, finer aluminium particles and slower heating rates resulted in a decrease in onset temperature while higher aluminium contents in the mixture led to a higher reaction temperature. In Nb-rich samples, compaction led to a decrease in the onset temperatures.

After a heating cycle to 1000°C, EDS and XRD analyses confirmed the formation of only NbAl3 in Al-rich samples and a mixture of NbAl3 and Nb2Al along with unreacted niobium particles in Nb-rich samples. A subsequent heat treatment was necessary to obtain a single aluminide corresponding to the initial composition. These observations can be explained on the basis of niobium dissolution in molten aluminium and subsequent precipitation of NbAl3 in Al-rich samples and solid state diffusion through Nb3Al and Nb2Al in Nb-rich samples.

Bioplastics for the Next Generation of Packaging Materials

Matilda Larsson, Dept. of Chemistry/Centre for Analysis & Synthesis (CAS)

Plastic packaging materials are designed to enhance our quality of life by preserving and protecting food and medicals. However, a negative outcome of the durability and widespread use of conventional plastic is the worldwide pollution by plastic garbage. This motivates research for alternative renewable packaging materials. Polyhydroxyalkanoates (PHAs) are both biobased and biodegradable and are an alternative to plastics made from petrochemicals. PHAs are polyesters that can be accumulated as intracellular granules by many naturally occurring bacteria. The purified PHA may exhibit material properties similar to, e.g., polypropylene and polyethylene. In the current project, we aim to improve the processability and physical properties of PHA by different modifications. Blends of different biopolymers are prepared via melt extrusion to tune the material properties. In addition, composites of PHA with various organic and inorganic fillers are prepared to further improve mechanical, thermal and barrier properties, which is important for the applicability of the materials.

System dynamic study of mould growth on building materials

Yujing Li, Div. of Building Materials

During the recent decades indoor mould is one of the major concerns for the indoor environment and the sustainability of the material use since it is closely connected to the health issues for the building occupants. This study presents a system dynamic study of mould growth on building materials by taking into account of the physical properties of the building materials and the physiological properties of the mould. This model can be used to predict the mould growth in the buildings or even to prevent the mould growth from happening in practice. This is an inter-discipline study of the material science and the biological science.

Mould, building materials and the surrounding air are considered as a system. All the influencing factors within the systems and their interactive impacts are analyzed by the system analysis. Based on the analysis a system model is built by STELLA®. The model consists of the simulations of the moisture properties of the materials: moisture exchanged between the building material and the surrounding environment via desorption and adsorption processes,

which are influenced by the hysteresis properties of the material and the temperature. This part is connected to the activities of the mould, as mould consumes the moisture. The physiological properties of mould are also influenced by the moisture states of the material and the surrounding environment, such as temperature, oxygen etc. By providing the climate data, the model can predict the mould growth over the time.

A Methodology for using Kalman Filter to Determine Material Parameters from Uncertain Measurements

Abdallah Shokry, Div. of Solid Mechanics

A Kalman filter can be used to determine material parameters using uncertain experimental data. However, starting with inappropriate initial values for material parameters might include false local attractors or even divergence. Also, inappropriate choices of covariance errors of initial state, present state, and measurements might affect the stability of the prediction. In addition, the covariance of the error of both state and measurement was so far always assumed to be constant. In reality it is often the opposite as the process change over time. The present method includes a continuous adjustment of the covariance of the errors and known parameters are used to generate the data used as "measurement data". The method consists of two steps. First, a small and appropriate range of parameter values is chosen based on a graphical representation of the standard deviation. Second, the Kalman filter is used on the selected range. The modification of the filter significantly reduces the iteration time as compared with the standard Kalman filter. When the methodology is applied to real data, very good results are obtained in terms of convergence.

Development and characterization of composite materials

Kenneth Frogner, Div. of Production & Materials Engineering

I would like to give a presentation about the composite materials that have been developed at the division of Production and Materials Engineering, LTH, as well as the characterization of the materials. The materials feature soft magnetic properties and aim to improve the magnetic coupling and thereby the efficiency in electromagnetic components, for instance in induction heating processes. The presentation will also include a short part about processing and heat treatment of materials to locally reach certain mechanical properties using induction heating.