The Materials Science Graduate Student Days 2017

Booklet

February 21-22, 2017
Virtual Development Laboratory
Chalmers Campus Johanneberg
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**Program 2017**

**Tuesday, February 21**

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<td>09.00 - 09.15</td>
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| 09.15 - 09.45 | Invited: Belgium, The Netherlands and Sweden ... well, there must be a difference  
|               | Vincent Nierstrasz, Department of Textile Technology, The Swedish School of Textiles, University of Borås |
| 09.45 - 10.30 | COFFEE and Poster session                                           |
| 10.30 - 10.50 | Surface integrity characterization of gears with respect to running-in  
|               | Dinesh, Mallipeddi, Materials and Manufacturing Technology           |
|               | **Chair:** Giulio Calcagno, Chemistry and Chemical Engineering       |
| 10.55 - 11.15 | Oxide evolution on X-750 Ni alloy in simulated BWR environment       
|               | Silvia Tuzi, Physics                                                 |
| 11.20 - 11.40 | Probing protein intercalation at organic–inorganic interfaces        
|               | Simon Isaksson, Chemistry and Chemical Engineering                   |
| 11.45 - 12.15 | Invited: Spread the word: popular presentation of your research      
|               | Cecilia Berlin, Product and Production development                   |
| 12.15 - 13.15 | LUNCH                                                                |
|               | **Chair:** Casey Jessop / Johanna Ekberg, Materials and Manufacturing Technology |
| 13.15 - 13.25 | Presentation of Lab tours                                            
|               | Casey Jessop / Johanna Ekberg, Materials and Manufacturing Technology |
| 13.25 - 15.00 | Lab tours: Materials and Manufacturing Technology                    |
| 15.00 - 15.30 | COFFEE and Poster session                                           |
|               | **Chair:** Hanna Härelind, Chemistry and Chemical Engineering        |
| 15.30 - 15.50 | Hydrophilic and hydrophobic functionalizations of aqueous silica sols for Pickering  
|               | Sanna Björkegren, Chemistry and Chemical Engineering                |
| 15.55 - 16.15 | Hysteresis-free nanoplasmonic Pd-Au alloy hydrogen sensors           
|               | Ferry Nugroho, Physics                                               |
| 16.20 - 16.40 | In-depth microstructural characterization of sustainable electrodeposited alloys  
|               | Antonio Mulone, Materials and Manufacturing Technology               |
| 16.40 - 18.00 | Scientific Speed Dating / Poster session                             |
| 18.00         | DINNER at Hyllan                                                     |
Wednesday, February 22

Chair: Lena Falk, Physics

09.00 - 09.30 Invited: How do you know which path you will adopt? - Research in industry or academia?
Shabira Abbas, SCA Hygiene Products AB

09.35 - 09.55 Ageing of RPV steel – an atom probe tomography study
Kristina Lindgren, Physics

09.55 - 10.30 COFFEE and Poster session

10.30 - 10.50 Cost effective carbon fibre composites in future aeroengines
Dennis Wilhelmsson, Applied Mechanics

Chair: Anand H S Iyer, Physics

10.55 - 11.15 High-entropy alloys (HEAs) -- Alloxed pleasures or multimetallic cocktails
Saad Sheikh, Materials and Manufacturing Technology

11.20 - 11.40 Modelling of cyclic and viscous behaviour of pearlitic steels
- Application to tread braked railway wheels
Ali Esmaeili, Applied Mechanics

11.45 - 12.00 The Area of Advance Materials Science
Anders Palmqvist, Area of Advance Co-director

12.00 - 13.00 LUNCH

Chair: Casey Jessop / Johanna Ekberg, Materials and Manufacturing Technology

13.00 - 13.30 Invited: The path from a narrow topic to a broad application of microscopy and microanalysis
Melina da Silva, Swerea IVF

13.35 - 13.55 Microstructure and defects in titanium alloy welds
Sakari Tolvanen, Materials and Manufacturing Technology

14.00 - 14.20 Characterisation of cation ordering, oxygen vacancy distribution and proton site in hexagonal and cubic BaTi$_{1-x}$Sc$_x$O$_{3-δ}$
Nico Torino, Chemistry and Chemical Engineering

14.25 - 14.55 Invited: An academic career - Pros and Cons
Fang Liu, Physics

14.55 - 15.30 Reflections on Scientific Speed Dating and COFFEE

15.30 Concluding remarks
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Surface Integrity Characterization of Gears with respect to Running-in

Dinesh Mallipeddi

Department of Materials and Manufacturing Technology
Chalmers University of Technology

Abstract

A gearbox with gears of different sizes is part of a vehicle transmission system that plays an important part in transmitting the engine power to the wheels. The efficient energy transmission highly relies on the performance of gears. Together, the mesh efficiency and durability determines the gear performance. It has been reported that the final smoothening of surface by running-in process has increased the mesh efficiency of ground gears. This is not the case for superfinished gears. However, in comparison to ground, superfinished gears produced a higher overall efficiency but only at higher speeds.

Micropitting is a surface contact fatigue failure which occurs in all type of gears and for all heat treatments. Depending on the initial surface micro-geometry and contact conditions used, the micropitting can initiate after a relatively short running time. Further progression of micropitting due to continuous operation leads to degradation of profile and thereby gears fail in the form of pitting, spalling or tooth breakage.

The hard finishing of gear surface by different manufacturing methods, for example grinding, honing and superfinishing etc., produces a unique characteristic surfaces in terms of roughness, surface lay and residual stresses. These topographical characteristics of tooth flank affects the efficiency and durability of gears. In addition, contribution of microstructure and surface chemistry should not be neglected. Running-in process is known to alter the aforementioned surface characteristics via smoothening the surface asperities. This creates an interest to understand the initial running period with the purpose to improve efficiency and longevity of gears. The aim of this study is to investigate the effect of running-in load on the surface characteristics of gears set by generating grinding. Secondly, to follow how these characteristics influence and further develop during the initial usage. To characterize the surface layers of gears a methodology is developed by combining analytical techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS).

The results showed that running-in process smoothened the surface asperities through plastic deformation and the severity of deformation increased with the magnitude of the load. On the other hand, higher load produced more micropits and this trend continued for in-line efficiency testing as well. It has been found that on one side of the as-ground teeth the stresses were rather uniform while there were stress gradients from tip to dedendum and in axial direction on the other side. The deformation created by running-in increased the compressive residual stresses but only at the surface confined to 5 µm. It has also been found that surface conditioned by higher running-in load gave subsurface cracks during efficiency testing. High atomic concentration of phosphorous-containing extreme pressure additive (EP) was also observed after efficiency test that ran at high load.

Keywords: Gears, running-in, micropitting, surface roughness, residual stresses, surface chemistry, surface asperities
O2

Oxide evolution on X-750 Ni alloy in simulated BWR environment

Silvia Tuzi\textsuperscript{1*}, Kenneth Göransson\textsuperscript{2}, Seikh M.H. Rahman\textsuperscript{1}, Sten G. Eriksson\textsuperscript{1}, Fang Liu\textsuperscript{1}, Mattias Thuvander\textsuperscript{1}, and Krystyna Stiller\textsuperscript{1}

\textsuperscript{1}Chalmers University of Technology, SE-412 96 Göteborg, Sweden
\textsuperscript{2}Westinghouse Electric Sweden AB, SE-721 63 Västerås, Sweden

Nickel alloy X-750 is used extensively in the nuclear industry, in particular as spacer grids in boiling water reactors (BWR). The X-750 is a precipitation hardened Ni-Cr-Fe superalloy with high strength at elevated temperature and good corrosion proprieties. Still, in BWR environment the alloy might suffer from significant corrosion. The material is typically hardened by a heat treatment at 700°C, resulting in formation of $\gamma'$-Ni$_3$(Ti, Al) precipitates. This treatment can also serve as pre-oxidation, which gives better resistance to corrosion in service. However, as the surface of spacer grids may be damaged, and hence the protection of the pre-oxide can be lost, it is also important to know the corrosion properties and the evolution of the oxide formed on samples that have not been pre-oxidized. In this study, samples that have not received the hardening heat treatment have been studied, mainly using electron microscopy.
Probing Protein Intercalation at Organic–Inorganic Interfaces


Div. of Applied Chemistry, Chalmers University of Technology, *Materials Physics and Application Div., Los Alamos National Laboratory, **Dept. of Pharmacy, Uppsala University, ***Dept. of Biomedical Sciences and Biofilm-the Research Center for Biointerfaces, Health and Society, Malmö University, ****Dept. of Chemistry and Molecular Biology, University of Gothenburg, *****Div. of Biological Physics, Chalmers University of Technology

Proteins are key components in a multitude of biological processes, of which the functions carried out by transmembrane (membrane spanning) proteins are especially demanding to investigate. This is because this class of protein needs to be incorporated into a lipid bilayer representing its native environment, and in addition many experimental conditions also require a solid support for stabilization and analytical purposes. The solid support substrate may, however, limit the protein functionality due to protein–material interactions and lack of physical space.

We have tailored the pore size and pore ordering of a mesoporous silica thin film to match the native cell-membrane arrangement of the transmembrane protein human aquaporin 4 (hAQP4). Using neutron reflectivity, we provide evidence of how substrate pores host the bulky water-soluble domain of hAQP4, which is shown to extend 7.2 nm into the pores of the substrate (Figure 1). Complementary surface analytical tools, including quartz crystal microbalance with dissipation monitoring and fluorescence microscopy, revealed successful protein containing supported lipid bilayer (pSLB) formation on mesoporous silica substrates, while pSLB formation was hampered on nonporous silica. TEM, SEM, DLS, stopped-flow light scattering, and SAXS were employed to provide comprehensive characterization of this novel hybrid organic-inorganic interface, the tailoring of which is likely to be generally applicable to improve function and stability of a broad range of membrane proteins containing water soluble domains. Our long-term goal is to make use of these kinds of design for water treatment purposes.

Figure 1: Illustration depicting the spontaneous formation of a pSLB from proteoliposomes. Protein (blue) domains protruding the lipid bilayer (brown) are shown intercalated into the pores of mesoporous silica (grey).
Hydrophilic and hydrophobic functionalizations of aqueous silica sols for Pickering emulsions

Sanna Björkegren, Lars Nordstierna, Anders Törncrona*, Michael Persson*, Anders Palmqvist

Chemistry and Chemical Engineering, Applied Surface Chemistry, Chalmers University of Technology, Gothenburg, Sweden, *AkzoNobel Pulp and Performance Chemicals, Bohus, Sweden

In this work, water-based silica sols have been functionalized with hydrophilic and hydrophobic groups with the aim to be used as emulsifiers for solid-stabilized emulsions, so called Pickering emulsions (see Figure 1). Since properly designed colloidal particles can accumulate and adsorb, in principle irreversible, at the interface of two immiscible liquids, such as oil and water, these have great potential for the mentioned emulsion systems.

The hydrophilic functionalization (poly(ethylene glycol) or mPEG silane) of the silica particle provides an efficient reduction of surface charge density, achieves a pH dependent and controllable flocculation behaviour and provides surface active particles. These properties are all beneficial for emulsion formulation. However, the hydrophobic functionalization of the silica sols, which is accomplished by attaching organosilanes containing propyl or methyl groups, was required to achieve efficient emulsifiers. It was found that the combination of both hydrophilic and hydrophobic groups was most efficient in formation of small emulsion droplets, compared to unmodified or only hydrophobically modified silica. The pH and the salt concentration are examples of important parameters for efficient emulsion droplet formation. A pH below 6 to 4, depending on the particle used, is beneficial and in some cases required for emulsion formation. The Pickering emulsions obtained display a high stability towards coalescence over a long period of time (more than 1.5 years).

Hysteresis-Free Nanoplasmonic Pd-Au Alloy Hydrogen Sensors

Ferry A. A. Nugroho*, Beniamino Iandolo**, Carl Wadell*, Emil Lidström*, Jakob B. Wagner** and Christoph Langhammer*

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The imminent prospect of a hydrogen economy and recent market introduction of hydrogen fuel cell cars demand a development of safe and reliable hydrogen sensors. Hydrogen sensors are also becoming increasingly important in many industrial processes, food industry and in health monitoring. Specifically for fuel cell cars, stringent performance targets have been set by the automotive industries to ensure a safe operation [1].

In this regards optical sensors are beneficial because they provide means for remote readout and are considered safer than electrical sensors as they pose no risk of spark generation. A growing avenue within the field of optical hydrogen sensors is the use nanostructured metal-hydride systems as signal transducer in plasmonic hydrogen sensors [2]. For this application, palladium (Pd) has been the main transducer material since it readily absorbs and releases hydrogen at RT. However, pure Pd exhibits wide hysteresis upon sorption which creates ambiguous readout depending on the sensor’s history. To this end, alloying the hydride-forming metal with another metal (i.e. Pd–Au) has been known to reduce the hysteresis.

In this work we present a novel plasmonic optical hydrogen sensor using Pd–Au alloy nanoparticles [3] and its generic fabrication strategy [4]. As the key features of our study, by increasing the Au content in the alloy nanoparticles up to 25 at.%, we find an eight-fold sensitivity increase in the sub-10 mbar pressure range (i.e. the critical pressure range before hydrogen reaches its flammability limit at 40 mbar) compared to a pure Pd, sensor accuracy < 5% throughout the studied 1 mbar–1 bar hydrogen pressure range and, by engineering the sensor nanoparticle size, sub-second sensor response times. Thus we meet or exceed the performance targets set by the automotive industry. Finally we also report a distinct wavelength-independence of the qualitative sensor response to hydrogen pressure. This opens up the possibility to use single-wavelength plasmonic hydrogen sensing, which promises the use of low-cost optical components such as cheap LED light sources and a simple photodiode detector for implementation in real devices.

References
In-depth microstructural characterization of sustainable electrodeposited alloys

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The objective of this research is to study the structural characterization of sustainable and innovative electrodeposited materials, in order to correlate the observed microstructure with the electrodeposits properties. This presentation focuses on the characterization of two different electrodeposits: pure Sn and FeW. The innovation and the sustainability aspect of such coatings involve the use of “green” and environmental friendly baths for the materials deposition. Pure Sn electrodeposits were produced from two different electrolytes, a stannous chloride and a methane sulfonic acid (MSA)-based bath. The aim of the investigation is to understand the effect of the applied plating conditions (i.e. different electrolytes, and different additive concentrations) on the deposited Sn structure (i.e. surface morphology, grain structure and grain size, texture). The results from structural characterization techniques (i.e. surface topography imaging by Scanning Electron Microscopy, and crystallographic orientation by X-ray analysis) and electrochemical characterization (i.e. polarization and Tafel measurements) show that the applied plating conditions influence differently the electrodeposited Sn structure (i.e. topography, grain size and crystallographic orientation). In particular the investigated additive shows prominent effects in the stannous chloride electrolyte: i.e. exchange current density decrease, suppression on H₂ and slight grain refinement. FeW coatings with different W content are deposited from an ammonia-free citrate electrolyte. X-ray analysis of these electrodeposits show an amorphous or nanocrystalline structure, depending on the different W content. Amorphous iron-based alloys are superior to crystalline in some application (i.e. higher corrosion resistance and mechanical strength). In this work the thermal stability of such amorphous coating and its variation depending on the chemical composition is studied by means of X-ray analysis after annealing the FeW samples at different temperatures.
Ageing of RPV steel – an atom probe tomography study

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Reactor pressure vessel (RPV) steels are a life-limiting component of a nuclear reactor. Neutron radiation during operation makes the steel embrittleled, the higher dose the larger effect. In Ringhals R4 RPV welds the combination of low Cu (0.04 at%) and high Ni (1.6 at%) is influencing the formation of clusters, forming Ni, Mn and Si rich clusters rather than the Cu rich clusters formed in RPV welds with higher Cu content. In this research material irradiated in the Halden test reactor under high neutron flux to fluences of 2.0 and 6.4 \(10^{19}\) n/cm\(^2\) was investigated. The clusters are in the nanometre range (see Figure) and thus atom probe tomography (APT) is a suitable technique for characterisation.

Here, the evolution of the microstructure is studied in order to understand the formation and growth mechanisms of the clusters. Cluster number densities, composition of clusters and cluster size distributions are compared for different fluences and to the mechanical properties. It is found that the nanometre clusters contain mainly Ni (55 %), Mn (29 %) and Si (13 %). The clusters also contain small amounts of Cu (3 %). A comparison of flux effects is also carried out, with the help of a surveillance material, supporting the relevance of using accelerated irradiation.

Figure: Clusters shown by NiMnSi isoconcentration surfaces and Cu atoms (dots) in the RPV steel.
Cost effective carbon fibre composites in future aeroengines

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With ever increasing traffic levels, the civil aircraft industry is in constant need of new technologies to make air travel environmentally sustainable. One such technology is light materials where the energy consumption can be reduced. Currently, up to 53 weight percent is made from carbon fibre reinforced polymer (CFRP) material in the Airbus A350XWB aircraft, mainly in the fuselage and wings. There is now interest to increase the use of CFRP in cold and moderately high temperature parts of the engines, where the outlet guide vanes is an example of such a part. The current research is carried out in response to an industrial interest of cost-effective CFRP components in load carrying parts of the engine. One problem for use of NCF composites in these applications is the lower compressive strength due to higher waviness of the carbon fibres, compared to the “standard” high performance composites.

The current study investigates the influence of intrinsic material variations on the performance of a unidirectional (UD), non crimp fabric (NCF), carbon fibre composite loaded in compression. These intrinsic variations to the material have been identified as potentially likely to occur in future aero-engine composite structures. Fibre misalignment angles from measurements were used to study how the out-of-plane waviness influence the longitudinal stiffness and strength of the composite.

As expected, fibre waviness is found to have a strong adverse effect on the compressive mechanical properties of the composite, where we have been able to demonstrate a correlation between the out-of-plane waviness and stiffness reduction.

Figure 1. a) Micrograph of fibre waviness out-of-plane for a section with length 20 mm and thickness 2mm. b) Measurements of fibre misalignment angles in the above micrograph with respect to a horizontal reference. Angles are defined positive clockwise and are presented in degrees.
High-Entropy Alloys (HEAs)--Alloyed pleasures or Multimetallic cocktails

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Abstract:

This project is devoted to studying the alloy development and mechanical property of high-entropy alloys. High-entropy alloy (HEAs), or multi-component alloys with equiatomic or close-to-equaiatomic compositions, emerge as novel metallic materials with great potential to be used as high temperature materials, or coating materials requiring high hardness and high wear resistance. Although being highly concentrated multi-component alloy systems, simple solid solutions tend to form in HEAs, with the absence of intermetallic compounds. This seemingly unexpected phenomenon is partly due to the significant entropic contribution, which lowers the Gibbs energy of the solid solution phases at elevated temperatures. Normally, the formed solid solutions in HEAs are of fcc and/or bcc structures. The mechanical behaviour of HEAs are greatly affected by the phase constitutions. How to simultaneously achieve both high strength and high tensile ductility is a great challenge in the field. It mainly involves alloy development and thermomechanical treatments, to tune the phase constitution and the microstructure, aiming at obtaining novel HEAs with balanced strength and tensile ductility.
Modelling of cyclic and viscous behaviour of pearlitic steels
Application to tread braked railway wheels

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Abstract
Wheel tread damage is one of the elusive phenomena that still limits the capacity of wheels from reaching desired mileage performance in the order of a million km. The tread material, in the block (tread) braked wheels, is subjected to high contact stresses induced by rolling and sliding of the wheel and also elevated temperatures due to frictional heat generation (between brake block–wheel and wheel–rail). The thermal loading results in the stresses induced by constrained thermal expansion. Moreover, the lowered strength of the material at the elevated temperatures has an impact on the resulting stress strain field in the tread.

In the presented study, thermomechanical cracking of railway wheel treads is investigated using a combined experimental and numerical approach. Experimental results are presented from controlled brake rig tests of repeated stop braking cycles of a railway wheel that is in rolling contact with a so-called railwheel. The test conditions are numerically analysed using finite element (FE) simulations that account for the thermomechanical loading of the wheel tread.

In this context, a methodology for FE simulation of full scale brake rig tests is developed. It includes three types of finite element analyses: an axisymmetric thermal analysis, a 3D mechanical wheel-rail contact analysis and a 3D thermomechanical analysis. The studied railway wheel steel is a medium carbon steel ER7T (0.55 wt. % C), heat-treated to a near pearlitic microstructure. The wheel material behaviour is modelled by a viscoplastic material model calibrated against results from cyclic strain controlled experiments at several temperatures, from room temperature up to 625 C. Resulting variations of stresses and strains in the material near-to the wheel tread are studied in detail for the assessed braking load cases. Fatigue life predictions are estimated from the evaluated ratcheting response using a simplified accumulation rule. The life time predictions are found to be in reasonable agreement with experimental results. The presented work constitutes one step towards experimental validation of simulation models for studying RCF damage at tread braking. It is found that it is instrumental to perform combined experimental and numerical studies in order to reveal the key phenomena that ultimately will be part of controlling the service life of tread braked wheels.

Keywords: Railway wheels, tread braking, rolling contact fatigue, full-scale brake rig testing, thermomechanical finite element analysis.
Titanium alloys are widely used for components in aeroengines owing to their high specific strength and creep properties at moderate temperatures. Aeroengine parts are often manufactured from large single piece castings that are machined into final shape. A significant amount of the initial casting weight is machined away in order to achieve the finished geometry. This is neither environmentally friendly nor economically viable. Fabricating large static compressor components through welding them out of smaller subcomponents instead of machining them from large single piece castings provides several potential benefits. There will be less scrap material and the structure can be designed to be more efficient owing to the possibility of selecting high strength titanium alloys in specific parts of the structure enabling increased functionality and improved performance, in the end resulting in reduced environmental impact. Fabrication technologies however require joining of subcomponents which is why welding and the mechanical properties of welds have been investigated here.

Fusion welding involves localized melting of materials which produces changes and defects in the material, which affect the mechanical properties of weld zones and make the design of fatigue resistant joints a complicated process. These reasons also result in a large statistical scatter in the fatigue test data. Of interest in this study are the microstructures and welding defects produced by different welding processes. The microstructure of the welds affects the static properties, such as tensile strength and ductility, and the crack initiation and crack growth under cyclic loading. Defect size and location are two important parameters for damage-tolerant design. Based on the initial defect size, the fatigue life can be predicted and this concept is also used as baseline for setting up periodic inspection of components. Several factors are believed to contribute to fatigue crack initiation from a pore, such as stress concentration around a defect, its size and location, whether it is located near the surface, presence of other pores nearby, and residual stresses from the welding process. It is also believed that a critical size exists and that the presence of defects below this size does not affect the fatigue strength.

Welds produced with electron beam welding (EBW), laser beam welding (LBW), tungsten inert gas welding (TIG) and plasma arc welding (PAW) have different microstructures and different populations of defects in terms of their size and distribution. The effect of these aspects on mechanical properties under different testing conditions have been investigated in the present work.
In the field of protonics, acceptor doped perovskites represent a valid choice for the development of electrolyte materials for fuel cells, steam electrolyzers, and hydrogen sensors and separation membranes. Still, the technical challenges call for a deeper understanding of the proton environment and its dynamics, in order to bring those technologies to maturity. The solid solution $\text{BaTi}_{1-x}\text{Sc}_x\text{O}_{3-\delta}$ ($0.1 \leq x \leq 0.8$) [1, 2] is characterised by a structural transition from a 6H perovskite to a cubic one, with increasing Sc content. We found that below 600°C, in humid atmosphere, protons are the main charge carriers all through the series. The proton conductivity, on the other hand, dramatically increases when the composition favours a cubic crystal structure over the hexagonal one. Rietveld analysis of X-ray diffraction data suggests different oxygen vacancy ordering within the 6H perovskite structure, whereas the vacancies are randomly distributed in the cubic one. However, the driving force for the compositionally driven structural transition, and the poorer conductivity of the 6H structure, continued to elude our understanding. New insights will be presented, where neutron powder diffraction (NPD) complements the previous structural studies, and is coupled to density functional theory (DFT) calculations. A long range ordering of the Ti and Sc ions on the B-site of the 6H perovskites of the series was revealed, and a preferred proton site individuated. Further, in situ studies, combining NPD and thermogravimetric analysis, shed a light on the migration pathways within the material.


Mechanism for Solid-State Ion-Exchange of Cu\textsuperscript{1} into Zeolites

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Abstract

Density functional theory calculations are used to investigate solid-state ion exchange of copper into zeolites. In particular, the energetic conditions for functionalization of chabazite (CHA) with copper ions from Cu\textsubscript{2}O(111) via formation of Cu(NH\textsubscript{3})\textsubscript{2}\textsuperscript{+} are explored. It is found that the diamine complexes form easily on Cu\textsubscript{2}O(111) and diffuse with low barriers over the surface and in the CHA framework. The charge neutrality of the systems is maintained via counterdiffusion of H\textsuperscript{+} in the form of NH\textsubscript{4}\textsuperscript{+} from the zeolite to the Cu\textsubscript{2}O surface where water can be formed. The efficient solvation of Cu\textsuperscript{+} and H\textsuperscript{+} by ammonia renders the ion-exchange process exothermic. The present results highlight the dynamic character of the Cu ion sites and provide means to understand zeolite functionalization.
Characterization of Thermal Barrier Coatings obtained by Axial Injection Suspension Plasma Spray Process

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Thermal barrier coatings (TBCs) are used in the hot section of gas turbines to protect the underlying components from high temperature loads. They extend the lifetime of the turbine but also allow for a higher combustion temperature which enhances the engine efficiency and results in lower emissions. Axial suspension plasma spraying (ASPS) is a relatively new, innovative spray technique using a feedstock consisting of fine powder particles suspended in a liquid. With ASPS, TBC topcoats with columnar microstructures have been produced that containing nm- and µm-sized pores. The columnar structure is preferable because it can better tolerate high strains that occur during thermal cycling. However, so far the formation of the microstructure is not yet well understood. Characterization of the TBC topcoat microstructure and the first deposited plasma sprayed layer can provide important information on the phenomena controlling the coating formation process.

The wide pore size range (including nano-sized pores) in TBC topcoats produced by ASPS makes it a challenge to find a technique to measure porosity and pore size distribution in these TBCs. However, it is important to investigate the porosity as it affects the thermal conductivity. Hence, here two new characterization technique for determining porosity in topcoats is introduced.
The oral admission of drugs can occur in different ways by creating different formulations [1], such as tablets, pellets or capsules. To control the drug release from different oral drug formulations, polymers can be added to cover the drug as a thin film [2]. The controlling aspect of using polymer coatings arise from utilising their differences in for example water solubility [3]. This study concerns three free standing phase-separated polymer films. Consisting of two immiscible polymers, ethyl cellulose (EC) and hydroxypropyl cellulose (HPC) where the HPC is water soluble. The films are produced with different volume fractions of HPC as reflected in the film name, i.e. HPC22, HPC30 and HPC45. Previous results have shown a percolation onset for pore connectivity at HPC22 [4]. Below 22 volume fractions of HPC, HPC is encapsulated in the EC phase, thus results in a low permeability in leached films. While above, HPC forms a continuous phase which results in a porous microstructure upon contact with water as HPC leaches out, leaving a porous EC matrix. The release properties are determined by the pore size and the pore connectivity.

The aim of this work was to reconstruct the porous microstructure of soft materials, in this specific case, free standing phase-separated polymer films used for the purpose for controlling the drug release. A spatial resolution that ranges from micrometres to nanometres and a high precision are required for this work and therefore a combined focused ion beam and scanning electron microscope (FIB-SEM) has been used.

A concept to reconstruct the three-dimensional microstructure of soft porous materials will be presented. The parameters for FIB-SEM of soft porous insulating materials have been optimised where the effects of the interaction between both the electron beam and the ion beam with the soft materials can be challenging. The separation of surface from sub-surface information has also been addressed. The porous microstructure is visualised by a three-dimensional reconstruction from sequential cross section secondary electron images.

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Catalytic oxidation of methane and ethane under lean conditions

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As the need for diverse energy sources keeps growing, more attention is directed towards the world’s vast amount of natural gas which in turn motivates further development of gas powered engines. By replacing fossil fuels with natural gas, which typically consists of 90-95 % methane, a reduction of carbon dioxide emissions could be achieved due to the lower C/H ratio [1]. However, the methane slip from natural gas fuelled engines is typically around 1000 ppm which has to be oxidised to make the fuel a strong competitor. This is still a challenge at lower temperatures because of methane's stable molecular structure.

This study aims to elucidate the mechanism of methane oxidation and effects of total pressure on reaction mechanism and overall methane conversion. Using diffusive reflectance infrared Fourier transform spectroscopy (DRIFTS) together with flow reactor experiments and modelling, the knowledge about methane oxidation and the preferred reaction pathway will be enhanced. A model is developed to combine the well-known mass and heat transport relations from classical chemical engineering and first principle calculation in terms of density functional theory (DFT).

Figure 3. TOF of methane oxidation over PdO(101) against temperature and total pressure.

As shown in Figure 1, first principle calculations predict an interesting effect on intrinsic reaction rate when changing total pressure. At lower temperatures the turn over frequency (TOF) is hampered by an increased total pressure while at higher temperatures the opposite effect is observed, probably due to lower coverage of poisonous species. One question of interest is whether transport limitations are affecting the overall reaction rate. A model in MATLAB has been developed which combines concepts of classical chemical engineering and first principle calculations for more details on kinetics and transport limitations of methane oxidation.

References

Enzyme immobilization in tailored siliceous mesostructured cellular foams for the conversion of CO$_2$ to methanol

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The conversion of CO$_2$ to methanol is a highly desired reaction and it can be performed using three enzymes: formate-, formaldehyde-, and alcohol dehydrogenase. Enzyme immobilization in mesoporous materials can be advantageous for the improvement of the catalytic and cost efficiency of this bioprocess. Siliceous mesostructured cellular foams (MCF) can be specifically designed with tailored surface properties, pore size and volume to increase the interactions MCF-enzymes.

In this work, MCF with suitable characteristics was synthesized (pore size: 26.8 nm; window size: 10.5 nm, pore volume: 1.81 cm$^3$/g; surface area: 541 m$^2$/g) and its surface was modified with aminopropyl, mercaptopropyl, octyl and chloromethyl groups (MCF-AP, MCF-MP, MCF-OC and MCF-CM, respectively). The immobilizations were made separately for each enzyme at pH 5.6 and 7.0. The pH 5.6 was found to be most favorable for the immobilizations. The highest loading of formate dehydrogenase was observed in MCF-MP and MCF-CM. For formaldehyde dehydrogenase, the MCF-CM was also the best material, whereas for alcohol dehydrogenase the highest loading was in the non-modified MCF. The leakage of the enzymes was analyzed in 2 cycles of 24 h each at pH 6.5 and 37 ºC. The best results were obtained for formate- and formaldehyde dehydrogenase in MCF-CM, while for alcohol dehydrogenase less leakage was observed in MCF-MP.

In the follow-up phase of the studies, the activity of the immobilized enzymes are being investigated and the initial results suggest that the enzymes retain their activity after immobilization.

These findings demonstrate that the enzyme loading and adsorption stability is strongly dependent on the MFC modifications, allowing for the selection of the best materials to support these enzymes for the conversion of CO$_2$ to methanol.

Additive manufacturing (AM) is an inherently materials efficient manufacturing technique compared to conventional methods such as casting or machining. One of the key aspect here is the reusability of the excess powder that has been exposed to the AM process. Hence, it is important to understand the possible scenarios of change in the powder properties during exposure to the conditions in the build chamber under multiple process cycles and their effect on the final properties of the built components. In this study, the influence of powder reuse in case of Inconel 718 during Electron Beam Melting (EBM) was investigated. This was done by means of X-ray photoelectron spectroscopy (XPS) and high-resolution scanning electron microscopy (HR SEM) of the virgin and used powder for different number of cycles to detect alterations in powder surface chemistry and its correlation to the bulk chemistry as well as particle size distribution, shape and morphology. It was found that the composition and concentration of the surface oxides on the used powder increased significantly already after a few building cycles, especially on the powder particles sampled close to the built component. Correlations between powder degradation and component microstructure are made and finally the implications of these factors on quality of built components are discussed.
Measuring fracture properties of thermally grown sub-micron oxide scales

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Abstract

Superalloys form an important class of materials that are mostly used in the aviation industry in which high temperature strength is critical. These components are subjected to thermomechanical fatigue which is the primary reason for their failure. High temperature fatigue has been investigated for a long period of time. It has been found that a period of constant load at high temperature (dwell fatigue) causes rapid crack growth compared to cycling of loads. One of the mechanisms proposed for such a behaviour is Stress Assisted Grain Boundary Oxidation (SAGBO). According to this mechanism, the rapid crack growth occurs due to the cracking of oxides formed ahead of the crack tip. Therefore, an understanding of the properties of oxides in superalloys at operating temperatures is essential.

This work focuses on measuring fracture properties of oxides formed in superalloys at operating temperatures through micromechanical testing. Alloy 718plus is used in aero engines and they enable operation up to a temperature of 704 °C. In the current work, Alloy 718plus is exposed to oxygen at 700 °C to develop oxide scales. These samples are used to manufacture bi-layered microcantilevers with oxide on top and the metal beneath. They are bent in a SEM using a Kleindiek micromanipulator setup. The setup enables recording of the force-displacement data which is used to study the fracture properties of oxides.

Fig: Test setup for micromechanical testing of cantilevers inside the SEM.
Enabling Textile Recycling: On/Off Dyeing

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With a world increasing population, the demand of textile is expected to grow in the future. At the same time more than 60 % of consumed textile is today disposed, putting natural fibers in an unbalanced situation between offer and demand as cotton production is reaching a peak. This has driven the research for the development of new technologies to recycle textile over the last decades.

Despite large efforts, the upcycling of textile is still difficult. One bottleneck during recycling is the removal of the color without damaging the fiber. The approach of this research project is to develop a new strategy for dyeing that includes an on-demand de-dyeing functionality. The key to achieve this functionality is to control the colloidal behavior of the pigment and its adsorption at the textile surfaces.

The On/Off technology will focus on cellulose material since it is the most used natural textile fiber. The cellulosic material will be cationized in order to promote the main driving force for the dyeing, i.e. the electrostatic interactions between the anionic pigment dispersion and cationized cellulose surface. The de-dyeing mechanism will be based on reversing the charge of the colloid. In order to achieve this functionality, this project focuses on the development on switchable surfactants and polymers.
Ni-based superalloys are a unique class of materials with both excellent strength and corrosion resistance at high temperatures. Therefore they find extensive application in the hot sections of aircraft engines, where they account for up to 50% of the total weight. Its excellent high temperature properties however make the fabrication of components by means of conventional metal cutting very challenging. This is mainly attributed to the generation of high temperatures and stresses in the cutting zone which in turn lead to rapid degradation of the cutting tools.

Conventionally, coolant is supplied by means of uncontrolled flooding of the cutting zone. In this way, zones of highest temperature are not penetrated and therefore no effective temperature reduction is achieved.

High Pressure Jet Assisted Machining (HPJAM) is a high performance cooling strategy which involves directing high pressure coolant jets precisely into the cutting zone. Numerous publications are reporting reduced cutting temperatures along with longer tool life which is the basis for application of higher cutting parameters. Hence higher metal removal rates can be achieved which is highly beneficial in terms of overall productivity.

Despite the presence of studies regarding the positive influence of the high pressure coolant jet on the wear rate, there is a lack of understanding the basic mechanisms behind the tool wear mechanisms being involved. This work aims at closing this knowledge gap by characterizing flank wear of uncoated cemented carbide tools which were used in HPJAM tests.
SiO$_2$ nanoparticle grafted surfaces as a model material for studying protein adsorption using acoustic sensing and circular dichroism

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The surface chemistry and topography influences what happens when a foreign material enters the body and meets blood and tissues. Nanotechnology and the development of nanomaterials have resulted in the opportunity to design nanostructured implants with novel topographical and chemical surface properties. However, knowledge of how the topography at the nanoscale, e.g., curvature, affects protein adsorption and further the fate and success of tissue integration is still limited.

The aim of this study was to investigate how the size and curvature of nanofeatures affect the adsorption of blood proteins. By developing a model material, constructed using APTES-functionalization and SiO$_2$ nanoparticles of different sizes, protein adsorption onto a large spectrum of curvature with homologous chemistry can be systematically studied using the acoustic sensor technique Quartz Crystal Microbalance with Dissipation Monitoring (QCM-D). Circular Dichroism (CD) will also be employed to study conformational changes of the protein secondary structure using a holder with stacked functionalized substrates.

Nanoparticle deposition using 0.1 mM PBS as dispersant is demonstrated in Fig. 1 and some preliminary results of albumin adsorption using QCM-D.

![Fig. 1: Left: Scanning electron microscopy images of SiO$_2$ nanoparticles deposited onto APTES functionalized silica substrates using 0.1 mM PBS as dispersant. 9 nm (a), 25 nm (b), 40 nm (c) and 80 nm (d). Scalebar: 100 nm. Right: Observed frequency and dissipation shifts upon 1 mg/ml albumin adsorption onto SiO$_2$ np grafted surfaces. 9 nm (●), 25 nm (■), 40 nm (▲).](image)

Preliminary results of albumin adsorption using QCM-D indicate that adsorption is affected by the size of the nanoparticles. More results will follow later.

This study was supported by grants from the Centre for Environment and Sustainability in Gothenburg
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Synthesis of Transition Metal Ion-chelating Ordered Mesoporous Carbons for Noble Metal-free Fuel Cell Catalysts

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Fuel cells are of great interest as energy conversion devices with high potential to efficiently convert chemical energy to electricity. Nevertheless, the main reason to the fuel cells’ slow commercial development and limited market introduction over the last decades is mainly due to the current use of expensive noble metal-based catalysts. In order to make fuel cells economically competitive to other energy conversion devices the cost of the catalyst has to be reduced. Therefore, it is of interest to develop new noble metal-free catalysts with similar properties in terms of catalyst conversion efficiency and operation stability as platinum. Another important requirement in developing such catalysts is to achieve a low degree of complexity of the catalyst preparation in order to further reduce cost. This project aims to design and synthesize materials with the purpose to find a simple, inexpensive way to prepare efficient noble metal-free catalysts.
A major concern for the railway industry is the problem of rolling contact fatigue (RCF) damage in wheels and rails, with increased traffic, accelerations/decelerations, and loading conditions. The wheel/rail contact which occurs in railway applications is complex, and the understanding of the associated damage from combined mechanical and thermal loadings is a main concern for predicting fatigue life and developing appropriate maintenance systems. With respect to mechanical damage, different methods have been used to geometrically describe squat crack networks, and it was concluded that the different methods are complementary, and observations made using one method can sometimes explain deficiencies of other methods [1]. The effect of initial thermal damage, in the form of a small martensite spot (or WEL) on test bars, on fatigue life was examined. Investigation of crack initiation from notches was done by strain-controlled experiments using a MTS809 servo-hydraulic test machine. Comparisons between WEL observed in field samples and WEL artificially produced on test bars were made regarding microstructure and residual stresses. The presence of initial thermal damage was found to reduce the fatigue life.
Making foam from natural polymers

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The industrial demand for cheap, lightweight, single-use packaging material has led to an increase in popularity of fossil based polymeric foams like expanded polystyrene. This is mainly due to their low weight and relatively high mechanical properties. However, there are two major drawbacks of these foamed materials. The first one is that the raw material is coming from a non-renewable resource and the second one is that the recycling process of these foams can be problematic.

Cellulose is a promising non-fossil based alternative material for foaming applications, as it is lightweight, abundant and renewable. The properties of cellulose, which consists of long polysaccharide chains (up to 15000 monomer units long), can be tailored to suit different applications through various chemical modifications.

Here, hydroxypropyl methylcellulose (HPMC) was used together with water, acting as the blowing agent, in order to produce foams with a hot-mold foaming technique and by continuous extrusion. The rheological and mechanical properties of HPMC were evaluated by shear rheometry and dynamical mechanical testing (DMTA) and the produced HPMC-foams were characterized with regard to their apparent density and by scanning electron microscopy (SEM) imaging.

The aim of the project is to provide knowledge about alternative and renewable materials and understand the mechanism of foam formation of those, which hopefully in the future will be able to replace or reduce the use of fossil-based products.

Figure 1: From left to right: HPMC:H2O-mixtures, schematic image of an extruder, SEM images of the internal pore structure of extruded samples.
Characterization of virgin and recycled 316L powder used in Electron Beam Melting

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Electron Beam Melting (EBM) is an additive manufacturing (AM) process for fabricating high performance metallic parts. It is a powder bed method that uses a metallic powder as raw material. The powder is pre-heated in the build chamber and locally melted using a high-energy electron beam to build the part layer by layer. The loosely sintered powder after the build is blasted away and recovered. That powder is then sieved and mixed with virgin powder. This recycled blend will then be used in the next build. It is essential to understand the influence of this recycled blend in terms of powder characteristics and compositions.

In this study, a set of different techniques have been employed to characterize both virgin and recycled powder in order to further understand the influence of recycling on the powder properties. Powder microstructure was investigated by means of light optical microscopy (LOM) and the phase composition was determined by means of XRD. The powder surface characterization and chemical analysis were done by using XPS and SEM equipped with EDX.

Results showed that evenly distributed oxides particles in the submicron range can be observed on the surface of both virgin (~50nm) and recycled (~200nm) powder, see Fig. 1. The EDX analyses indicated enrichment of Cr, Si and Mn in oxides. According to XPS, both kinds of powder are covered by homogenous Fe-oxide layer with particulate oxide features rich in Cr and Mn. Significantly higher amount of Cr oxide was detected on the surface of recycled powder compared to the surface of the virgin powder. The recycled powder also had lower Mn content on the surface due to the sublimation of Mn, which drives the decomposition of Mn-oxide. The powder cross-section showed that the microstructure of the recycled powder had a tendency to change towards cellular structure, while the microstructure of virgin powder is fully dendritic.

Figure 4: SEM images of virgin (top) and recycled (bottom) powder showing particulates features.
High energy-density hybrid supercapacitors: Combining high-voltage window ionic liquids with MnO₂-nanomaterials

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Supercapacitors have attracted much attention the last years as the need of fast charging and reliable energy storage is of ever-increasing interest in today’s society and supercapacitors will fit this need perfectly. However there are still some issues that prevent supercapacitors from reaching bigger markets and more application. Perhaps the most important issue is the low energy density (per mass and volume) compared to batteries.

Two strategies have been employed to increase the low energy density:

1) Adding electrochemically active materials such as MnO₂ or RuO₂ to the carhode. This increases the capacitance of the electrode material and thereby the energy density.

2) Using electrolytes with larger voltage windows compared to traditional aqueous electrolytes. This enables higher operating voltages, which is especially important since the energy density of a capacitor has a quadratic dependence of the operating voltage.

These two approaches have mostly been explored separately, thereby missing the possibility to benefit from both. In our work we aim at merging both strategies, increasing both the capacitance and voltage window. We have investigated and compared different ionic liquids, how they interact with MnO₂-nanomaterials in a matrix of activated carbon. For instance we have compared the performance of the protic ionic liquids 1-ethylimidazolium bis(trifluoromethanesulfonyl) and 1-(2-Hydroxyethyl)-3-methylimidazolium bis(trifluoromethanesulfonyl) and the aprotic ionic liquid 1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl). In addition to this we have also investigated how different additives such as solvents and salts influence the performance of the MnO₂-nanomaterial.

Our results show that the availability of protons affects the performance of the MnO₂ however the dependence is quite complex. Our study provides an insight that can help to achieve a better understanding of the requirements on the electrolytes for hybrid supercapacitors.
Austenitic stainless steels (ASS) are among the most used engineering materials, particularly suitable for applications where high corrosion and oxidation resistance is required: e.g. chemical, pharmaceutical and food industry but also kitchenware. ASS have a face centered cubic crystalline structure, with substitutional alloying elements being chromium and nickel, are responsible for the anticorrosion properties: chromium forms a protective passive layer of Cr2O3 and nickel is austenite stabilizer allowing higher working temperatures. However, its outstanding chemical stability is not matched by other properties such as surface hardness and wear resistance, which can greatly hinder the lifetime of components where surface stresses are present. ASS are not hardenable by conventional heat treatments, on contrary, at high temperatures (>500°C) sensitization might occur due to precipitation of Cr at the grain boundaries, creating Cr depleted areas which can easily become source of corrosion. One possible improvement is given by low temperature carburizing/nitriding techniques (e.g. plasma), which triggers the formation a supersaturated interstitial solid solution case structure called S-phase, or expanded austenite. S-phase is characterized by the same corrosion resistance as the base material, but surface hardness up to five times higher and substantially higher wear properties. However, mechanisms of formation and stability, along with internal residual stresses and other structural characteristics are not fully understood. This topic is of great industrial interest due to the possible implications for improving materials properties both on a performance and economical point of view.
3D printed wood tissue

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The plant cell wall is the basis for the building up of all plants on earth, where the major component of the cell wall is cellulose. Trees are a major source of biopolymers, such as cellulose, and the forest is therefore valuable as a sustainable resource for future materials. In living wood plant cells, cellulose nanofibrils (CNF) are extruded and oriented in a specific pattern which provides optimal properties of the plant structure. The cell wall assembly is a complicated process where non cellulosic polysaccharides (hemicelluloses) adsorb onto cellulose nanofibrils which are then further crosslinked with lignin. We found that the 3D printing technology is an excellent tool for the conversion of CNF dispersions into composites with advanced functionality [1]. We are now investigating what effect the 3D printing process can have on the orientation of the printed nanofibrils since control of the orientation allows us to mimic the orientation of CNFs in the wood cell wall. This in turn, results in anisotropic materials with directed strength. One of the challenges when printing is the gelation of the printed material to avoid collapse upon printing 3D constructs. An all wood based solution is to use hemicellulose as a crosslinker to bind together the cellulose, as in the plant cell wall. However, native hemicellulose extracted from spruce needs to be modified in order to crosslink. We achieved crosslinking properties by functionalizing hemicellulose with tyramine, a molecule similar to lignin monomers [2, 3]. Apart from preventing collapse of 3D printed cellulose constructs, the crosslinked hemicellulose also adds strength to 3D printed cellulose films. Combining the technology of 3D printing with the inherent properties of wood biopolymers and the knowledge of the plant cell wall will lead to new fascinating materials that are strong, smart and renewable!

Thermomechanical Fatigue Life Prediction of High Specific Power IC Engine Cylinder Heads

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The constant drive towards higher specific power and lower displacement engines in recent years place increasingly higher thermal loads on the internal combustion engine materials. Further, the advent of hybrid power trains placing higher demands on quick starts and a rapid approach to maximum power necessitates the automotive industry to move towards a more robust computational thermo-mechanical fatigue life prediction methodology to develop reliable engines and reduce developmental costs. The study aims to develop constitutive and lifetime prediction models with just the necessary and sufficient parameters to predict the thermo-mechanical fatigue life of the highly loaded engine cylinder head. Uniaxial isothermal and non-isothermal tests are performed and the model parameters of a suitable constitutive model are calibrated to predict the non-linear stress-strain response under thermal and mechanical load cycling. The A356 T7 being the alloy of interest shows plastic and highly visco-plastic behaviour at room temperature and elevated temperatures respectively. With a suitable ageing model added, the study aims to successfully predict the lifetime pending fatigue crack initiation in the cylinder head.
TANTALUM AND NIOBIUM BASED Z-PHASE IN A Z-PHASE STRENGTHENED 12% CR STEEL

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Higher steam temperature in steam power plants increases their thermal efficiency. Thus there is a strong demand for new materials with better creep and corrosion resistance at higher temperatures, while retaining the thermal flexibility of martensitic steels. Z-phase strengthened 12% Cr steels have been developed to meet the 923 K (650°C) challenge in these power plants. Ta, Nb, or V forms Z-phase together with Cr and N. A new trial steel was produced based on combining Ta and Nb to form Z-phase. It was shown that Z-phase was formed with a composition corresponding to \( \text{Cr}_{1+x}(\text{Nb,Ta})_{1-x}\text{N} \). The Nb/Ta ratio in Z-phase precipitates was higher than that in MX precipitates. Z-phase precipitates based on Ta and Nb were coarser than precipitates in a similar trial steel based on Ta alone.

Keywords: martensitic steels, Z-phase, SEM, TEM, APT.
The unique and tuneable properties displayed by cellulose and cellulose derivatives have led to a wide variety of applications. Nanocelluloses are a relatively new member of the cellulose family and have created a lot of research interest during the past decades. There are two main types of nanocellulose; the very high aspect ratio cellulose nanofibrils and the highly crystalline cellulose nanocrystals with a lower aspect ratio. In recent years nanocelluloses have generated a lot of interest as reinforcing elements in polymer composites due to the large specific surface area and high Young’s modulus. However, as a result of the hydrophilic and often charged surface of nanocellulose, compatibility with the typically hydrophobic polymer matrix is usually poor. To obtain a material with good mechanical properties there is a delicate balance to tune the properties on a molecular as well as macro level. In an effort to improve matrix compatibility we have developed a novel modification technique for hydrophobization of the surface of nanocellulose. By reacting azetidinium reagents with the surface sulphate groups, a hydrophobic nanocellulose can be achieved, see figure below. The functionality of the azetidinium reagents can be varied for improved compatibility with a specific polymer matrix.

A key factor for manufacturing is that the reinforcing element in biocomposites can be produced in sufficiently large scale, something which has often been an obstacle for nanocellulose. We have developed an experimental method with which larger volumes of cellulose nanocrystals, CNCs, can be produced and chemically modified. Scaling up is however not the only aspect to consider; the thermal stability as well as the processability of the reinforcing element are important features to obtain composites with advanced performance.

The poster will present the synthesis and the rheological behaviour of CNC functionalised with azetidinium reagents containing different functional groups studied in water solution.
DETERMINING THE ORIENTATION DISTRIBUTION OF REGENERATED CELLULOSE MOLECULES WITH NMR SPECTROSCOPY

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An integral part in the material properties of regenerated cellulose fibers is the orientation distribution of the cellulose molecules relative to the direction of the fiber. This property will be investigated by Magic Angle Spinning (MAS) NMR spectroscopy and correlated to production of cellulose fibers. Due the semi-crystalline nature of regenerated cellulose, it is can be difficult to interpret results from X-Ray Diffraction, since amorphous cellulose exhibits broad, potentially overlapping peaks. NMR spectroscopy, in contrast, is not dependent on a crystalline structure, thereby making it possible to study the entire sample. The orientation distribution properties in NMR derived from the anisotropic spin interactions yield valuable information structure of cellulose. Specific experiments that are considered are ROSMAS, ORDER and DECODER.
To reduce the environmental impact of future transports, a considerable share of the fossil fuels needs to be replaced by renewable alternatives. Interesting candidate fuels are biogas and natural gas, which during combustion delivers more power per released carbon dioxide as well as low NOx, SOx and particulate emissions, compared to diesel and petrol.

A well-known challenge with catalytic oxidation of methane is to facilitate the dissociative adsorption of methane where the stable tetrahedral methane molecule is transformed into adsorbed hydrogen and methoxy (CHx) species. Previous studies state that the presence of water in the exhaust gas significantly lowers the catalytic activity over traditionally used catalysts. Here, a bottom-up research approach is used to thoroughly study single catalyst components and model catalysts.

Pd-based catalysts with different alumina supports have been studied by operando diffusive reflectance FTIR experiments during both dry and wet methane oxidation. Surface-bound molecular species have been identified and related to catalytic activity.
Cellulose fibres are used as reinforcing material component in composites due to its stiffening effect, sustainable origin, abundance, and low cost. When these cellulose pulps are further fibrillated a highly fibrillated cellulose pulps, called cellulose nanofibrils (CNF) or microfibrillated cellulose (MFC), are obtained. These fibers are small cellulose fibrils with diameter on the nanoscale and lengths on the micrometer scale. The advantage of CNF compared to pulp fibers is the high length to diameter aspect ratio, which is an important factor that influences the reinforcing effect in thermoplastic composites. In this work CNFs were used as reinforcement in a thermoplastic polymer matrix by water assisted mixing. CNF and water dispersible copolymer poly(ethylene-co-acrylic acid) (EAA) were mixed in the wet state to avoid fibril agglomeration and increase the reinforcing effect of CNF. The CNF content improved the mechanical properties of the resulting fiber reinforced composite material. The stiffness could be improved by a factor of 25 and the strength by a factor of 4. This reinforcing effect was visible up to CNF contents of 70 vol. %.
Direct conversion of methane to methanol (DCMM) is an interesting alternative process for efficient production of methanol. Inspired by the chemical functionality of methane monoxygenase (MMO), which is an enzyme that can selectively oxidize methane to methanol at ambient conditions, various metal-zeolites have been synthesized and tested for DCMM under mild conditions. Among these materials, Cu-exchanged zeolites have shown activity for DCMM when using molecular oxygen as the oxidant. A previous study has identified the active sites for DCMM in Cu-ZSM-5 as a bent mono-(µ-oxo)dicupric site [1], which has been related to an adsorption band at 22,700 cm$^{-1}$ in the ultraviolet-Visible (UV-Vis) spectrum [1, 2]. Comparable activities, however, have been observed also for Cu-mordenite [3, 4] and Cu-chabazite [5] suggesting that the bent mono-(µ-oxo)dicupric site may not be the only type of Cu site that is active for DCMM. On the contrary, zeolites exchanged with other metals, such as Fe and Co have also been reported to show activity. Most studies focus on identifying the active site and operating catalytic mechanism. There are, however, also other challenges such as regeneration of the active sites at low temperatures and extraction of the formed product from the zeolites that need be tackled.

In this study, a series of Cu-exchanged zeolites were prepared by aqueous ion-exchange. [6] The powder samples were characterized by UV-Vis spectroscopy and reflectance infrared Fourier transform spectroscopy (DRIFTS). Monolith samples were characterized by methanol temperature programmed desorption (TPD) using a continuous gas-flow reactor with outlet gas analysed by Fourier transform infrared spectroscopy (FTIR).

Metal injection molding (MIM) is an attractive alternative for net-shape manufacturing of components with high demands on shape complexity. Spherical powders with small particle sizes and a wide distribution are generally preferred for MIM parts. In this study, the sintering behavior of a standard and a low carbon and low oxygen spherical carbonyl iron powder grade and a sieved (-45 µm) water-atomized iron powder was investigated. Injection molded compacts made from the respective powders were sintered in a dilatometer in a hydrogen atmosphere to study the shrinkage behavior. The shrinkage of the powder during heating stage was related to the morphology and surface chemistry of the powders. Surface composition of the powders was studied by X-ray photoelectron spectroscopy (XPS) and high-resolution scanning electron microscopy (HR SEM) combined with EDX analysis (EDX). Results show strong correlation between the sintering activity, surface chemical composition, surface area, and current phase of the material.