The Materials Science Graduate Days 2013

A symposium on Materials Science for Graduate Students

An initiative of the Departments of Applied Physics, Chemical and Biological Engineering and Materials and Manufacturing Technology

Supported by the Materials Science Area of Advance

17-18 April, 2013

Kollektorn, MC2 building, Kemivägen 9, Chalmers
The Materials Science Graduate Days

Dear fellow student,

This year, for the first time, Chalmers is organizing a symposium entirely focused on you, the graduate student. The event will take place in Kollektorn, MC2 building, Chalmers campus Johanneberg between 17th and 18th of April. This initiative is held jointly by the Materials Area of Advance and the Departments of Applied Physics, Chemical and Biological Engineering and Materials Manufacturing Technology. Our goal is to promote interdisciplinary interest and facilitate networking among fellow PhD students. This is a great opportunity to get in contact with your peers and learn about their areas of research in a friendly and casual environment. You will have the unique opportunity to present your work in a popular science manner in oral or poster sessions and learn about materials science research at Chalmers. Invited speakers from industry and academia will share their experiences and insights and hopefully, give you some new perspectives on life after graduate studies. A tour of the lab facilities at the Department of Applied Physics is also planned as a first step towards increasing awareness of the resources available at Chalmers.

We hope that this initiative will be an interesting and fruitful experience to you as a graduate student and that you will enjoy your time there.

Welcome!

The Organizing Committee:

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Program

Wednesday, April 17

09.00 - 09.15 Registration
09.15 – 09.30 Welcome
09.30 - 10.00 University-Business Co-operation
    Jan-Eric Sundgren, Volvo Group Global
10.00 - 11.10 COFFEE and mounting of posters, Poster session
11.10 – 11.30 “Atom probe tomography of corroded Zircaloy-2”
    Gustav Sundell, Division of Materials Microstructure, TF
11.35 - 11.55 “Transition metal ion-chelating ordered mesoporous carbons as noble metal-free fuel cell catalysts”
    Johanna K. Dombrovskis, Applied Surface Chemistry, KB
12.00 - 13.00 LUNCH
13.00 - 13.30 Scientist career at AkzoNobel Pulp and Performance Chemicals
    Nina Simić, Akzo Nobel Pulp and Performance Chemicals
13.35 - 13.55 “CP/MAS $^{13}$C-NMR study of pulp hornification using nanocrystalline cellulose as a model system”
    Alexander Idström, Applied Surface Chemistry, KB
14.00 - 14.20 “Piezoelectric polymer bicomponent fibres”
    Anja Lund, The Swedish School of Textiles, University of Borås
14.20 - 15.30 COFFEE and Poster session
15.30 - 15.50 “On the effect of hydrogen addition on lean NOx reduction with methanol over Ag-Al$_2$O$_3$”
    Marika Männikkö, Competence Centre for Catalysis
15.55 - 16.15 “Evaluation of a measuring system for extensional flow through numerical and experimental studies”
    Magda Nyström, SIK - The Swedish Institute for Food and Biotechnology
16.20 - 16.40 Presentation of Materials Science Area of Advance
    Anders Palmqvist, Area of Advance Co-Director
17.00 – 19:00 Lab tour, Applied Physics
    19:00 DINNER: buffé, mingle, social activities and poster viewing
Thursday, April 18

09.00 - 09.20 “3D polysaccharide structures with controlled micro architecture by bottom-up fabrication”
   Johan Sundberg, Biopolymer Technology, Wallenberg Wood Science Center, KB

09.25 - 09.45 “Effects of graphene preparation methods on the photocatalytic performance of TiO₂/graphene composites”
   Raja Sellappan, Division of Chemical Physics, TF

09.45 - 10.30 COFFEE and Poster session

10.30 - 10.50 “The Graphene Flagship”
   Tomas Löfwander, Microtechnology and Nanoscience

10.55 - 11.15 “New concepts for copper-free antifouling coatings for marine applications”
   Alireza Movahedi, Applied Chemistry, KB

11.15 Concluding remarks

11.30 LUNCH
POSTERS

P1. Heading towards eco-efficient surface protection: To explore aspects that govern release from microparticles

   Jonatan Borg, Applied Surface Chemistry, KB

P2. Delubrication of Powder Metallurgy steels

   Seshendra Karamchedu, Surface and Microstructure Engineering, MT

P3. Fate of Hydrogen upon Oxidation of Zirconium Alloys by Water - A First Principles Study of Chemical Reactions in the Solid State

   Mikaela Lindgren, Division of Energy and Material, KB

P4. Surface chemical analysis of soft magnetic composite powders

   Christos Oikonomou, Surface and Microstructure Engineering, MT

P5. Environmentally friendly plasticized PVC by means of nanotechnology

   Henrik Petersen, SP Technical Research Institute of Sweden

P6. Waste Electrical and Electronic Equipment (WEEE) Plastics Composition

   Erik Stenvall, Polymeric Materials and Composites, MT

P7. Real time in situ monitoring of catalyst sintering on different support materials

   Pooya Tabib Zadeh Adibi, Competence Centre for Catalysis

P8. Evolution of the microstructure during creep testing of WC-Co based cemented carbide

   Amine Yousfi, Division of Materials Microstructure, TF

P9. Desorption of water from nanostructured models of interstellar dust grains

   Anna Clemens, Division of Chemical Physics, TF
Abstracts – Oral Presentations

Wednesday, April 17

**Transition metal ion-chelating ordered mesoporous carbons as noble metal-free fuel cell catalysts**

Johanna K. Dombrovskis
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Co-authors: Hyung, K. Fossum, O. Terasaki, A.E.C. Palmqvist

Polymer electrolyte membrane fuel cells (PEMFCs) are highly efficient at converting the chemical energy in hydrogen to electrical energy. PEMFCs are more efficient than combustion engines, but the drawback is that their fabrication is expensive. This is to a large part due to the high cost of the noble metal catalysts used in PEMFCs. Development of highly active catalysts of low cost is necessary to make the PEMFC technology competitive. Research on noble metal-free catalysts has recently resulted in insignificant improvements in catalytic performance for transition metal-containing carbon materials. Despite these improvements further development is necessary in order to fully replace noble metal catalysts. In this PhD work the fascinating topic of noble metal-free PEMFC catalysts has been combined with ordered mesoporous materials research. The possibilities for tuning the structure and properties of ordered mesoporous materials are used to specifically tailor new highly active catalyst materials. The developed catalysts are transition metal ions in nitrogen-functionalized ordered mesoporous carbons (OMCs). Nitrogen was incorporated in an OMC matrix giving the material chelating properties for transition metal ions that allow for the formation of porphyrin-like active sites.

Hornification is a phenomenon that occurs in fiber cell walls when subjecting wet delignified paper pulp fibers to drying. Upon sequential rewetting of the paper pulp, the swelling capacity of the fiber walls decreases, the internal surface area is reduced, and the pulp fibers turn stiffer and less conformable. The pore collapse and the subsequent inability of regaining the original porous fiber structure is often explained by strong hydrogen bonds formed between the cellulose molecules in the fiber walls. It has however been debated if hydrogen bonds solely can be responsible for the hornification phenomenon. Based upon 13C solid-state NMR studies, co-crystallization between cellulose fibrils in pulp fibers has been proposed as a possible reason for hornification.

The hornification process of paper pulp was investigated using solid-state 13C-NMR spectroscopy. Nanocrystalline cellulose was used to serve as a model system of the crystalline parts of the fibrils in pulp fibers. Characterization of the nanocrystalline cellulose dimensions was carried out using scanning electron microscopy. The samples were treated by drying and wetting cycles prior to NMR analysis where the hornification phenomenon was recorded by spectral changes of the cellulose C-4 carbon signals. An increase of the crystalline signal and a decrease of the signals corresponding to the accessible amorphous domains were found for both paper pulp and nanocrystalline cellulose. These spectral changes grew stronger with repeating drying and wetting cycles. The results show that cellulose co-crystallization contribute to hornification. Another conclusion is that the surfaces of higher hydrophobicity in cellulose fibrils have an increased preference for aggregation.
Piezoelectric polymer bicomponent fibres

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A novel piezoelectric fibre has been manufactured and characterised. The fibre is of a bicomponent core/sheath structure (see Figure 1), with poly(vinylidene fluoride) (PVDF) as the piezoelectric sheath component and an electrically conductive compound of carbon black (CB) and high density polyethylene (HDPE) as core material. The conductive fibre core works as an inner electrode.

The piezoelectric properties of PVDF are already well-known and exploited in commercial products, usually in the form of films. The manufacturing of piezoelectric fibres is still a new area of research. Difficulties include formation of piezoelectric $\beta$-phase crystallites, application of electrodes, and poling. We have previously shown that melt-spinning of bicomponent $\beta$-phase fibers with an integrated inner electrode can be done in a spinning process equivalent to that used in the industry [1-3]. Further, different approaches to applying the outer electrode, and different poling techniques and their effect on the piezoelectric characteristics of our bicomponent yarns have been evaluated [4]. The fibers show a clear piezoelectric effect, see Figure 2.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Micrograph of the bicomponent fibres' cross-sections}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure2.png}
\caption{Relationship between applied force (black line) and piezoelectric signal (grey line) from [3]}
\end{figure}

References:
On the effect of hydrogen addition on lean NO\textsubscript{x} reduction with methanol over Ag-Al\textsubscript{2}O\textsubscript{3}

Marika Männikkö,
Competence Centre for Catalysis, Chalmers University of Technology

Owing to concerns for increasing carbon dioxide emissions and limited fossil fuel resources the interest in bio-fuels and fuel-efficient combustion engines, operated in excess oxygen, is growing. Since the conventional three-way catalyst is not active for NO\textsubscript{x} reduction in lean conditions other techniques have to be developed. One possibility is hydrocarbon assisted selective catalytic reduction (HC-SCR), where the fuel is used as reducing agent and injected in appropriate amounts into the exhaust system prior to the SCR catalyst. A major challenge, however, is high catalytic activity at the relatively low temperatures of the lean exhaust gases. Ag/γ-Al\textsubscript{2}O\textsubscript{3} is a promising system for HC-SCR applications, and the low-temperature activity of the catalyst can be improved by the addition of hydrogen [1]. Methanol, here investigated as reducing agent for NO\textsubscript{x}, is considered a promising renewable fuel for the transport sector. In this study the influence of hydrogen addition on the lean NO\textsubscript{x} reduction with methanol over Ag-Al\textsubscript{2}O\textsubscript{3} is investigated. Specifically, the influence of silver loading and hydrogen concentration, with focus on low-temperature activity and selectivity for reduction of NO\textsubscript{x} to N\textsubscript{2} are studied.

Figure 1 shows that the addition of H\textsubscript{2} enhances the activity for NO\textsubscript{x} reduction to N\textsubscript{2} at low temperatures for Ag-Al\textsubscript{2}O\textsubscript{3} samples [2], with 2 - 4 wt% Ag. The higher activity is accompanied by a higher degree of oxidation of both the reducing agent and NO, in agreement with ref. [3]. This results in a more extensive formation of N\textsubscript{2}O, which is a strong greenhouse gas. Increasing the inlet H\textsubscript{2} concentration further increases the N\textsubscript{2}O formation at low temperatures and lowers the NO\textsubscript{x} reduction to N\textsubscript{2} at high temperatures. The results provide important input to the search for the reasons behind the positive effect of H\textsubscript{2} on HC-SCR. However, the benefit of H\textsubscript{2} addition for methanol-SCR applications is questionable.

Figure 1. Gas phase products formed: a) during cooling ramps over Ag-Al\textsubscript{2}O\textsubscript{3} with 1 - 4 wt % Ag as indicated in figure 1a, in the presence of 1000 ppm H\textsubscript{2} (solid lines) and without H\textsubscript{2} (dotted lines), b) at constant temperature over Ag-Al\textsubscript{2}O\textsubscript{3} with 3 wt% Ag, in the presence of 5000 ppm H\textsubscript{2}, 1000 ppm H\textsubscript{2}, and without H\textsubscript{2}, as indicated in figure 1b. Gas composition: 1700 ppm CH\textsubscript{3}OH, 500 ppm NO, 10% O\textsubscript{2}, Ar (bal.) and with/without H\textsubscript{2}. GHSV = 33400 h\textsuperscript{-1}.

References
Evaluation of a measuring system for extensional flow through numerical and experimental studies

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Knowledge of the behaviour of fluids in flow is necessary for appropriate quality control, product development and process design. Since food products often are non-Newtonian, viscoelastic liquids, the characterisation becomes more complicated. Effective and reliable characterisations of fluids are therefore needed. There are various techniques available for characterising viscoelastic liquids in shear but extensional properties are at least as important and presently there are not many techniques available commercially.

In this work a measuring technique based on hyperbolic contraction (see Fig. 1) is evaluated both by numerical and experimental studies. The method measures the pressure drop over a hyperbolic contraction caused by fluid extension and fluid shear, where the extensional component is assumed to dominate. Pressure drop predictions for different constitutive models, of varying shear and extensional response, through the hyperbolic contraction were studied to especially address the shear and first normal stress difference impact on the measured pressure drop. The predictions were performed through a hybrid finite element/finite volume scheme\textsuperscript{1,2} and compared to experimental measurements of the system.

The results showed the advantages of a hyperbolically shaped nozzle in achieving a constant strain rate and minimizing shear contribution to the measured pressure drop (see Fig. 2). Both numerical and experimental results demonstrate increasing pressure drops with increasing deformation rates. However, the rise in pressure drop is more pronounced for the experimental results. Numerical studies also shows the negative effect of shear on measured pressure drop and a strong correlation between first normal stress difference and pressure drop.

Fig. 2 Contour plot of the strain rate through a hyperbolic contraction. A constant extension rate is achieved at the symmetry line, enabling a broader measuring range.

References:
New concepts for copper-free antifouling coatings for marine applications

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Bio-fouling is a big problem in many marine applications. Authorities have banned many of the substances that were discovered toxic to marine organisms; however, these regulations have also led to the use of high concentrations of less effective biocides such as copper.

Here we suggest a new approach based on scavenging natural abundant copper from the sea. The scavenged copper (Cu²⁺) is strongly adsorbed into the coating material and following reduction into Cu⁺, it is released to the coating-water interface to act as biocide.

Our focus is to make a multifunctional host material which is able to act as a support for the copper ions within the coating. The host material has the ability to bind (Cu²⁺) and stabilize (Cu⁺) copper ions and to provide control of copper ion flux at the coating-water interface.

So far, we have functionalized commercial polymers with copper ion ligands. These new materials have been characterized by standard polymer characterization techniques and their copper uptake ability have been quantified. The next step is biological antifouling tests and subsequent fine tuning of material properties to optimize anti-fouling efficiency.
Wood derived biopolymers offer an environmentally friendly substitute to petroleum based synthetic polymers. Cellulose is the most abundant biomacromolecule found in nature and it constitutes a renewable and biodegradable resource with great potential. The β (1-4)-glucan linked cellulose chains form strong inter chain and intra chain hydrogen bonds making them insoluble in water. The strong interaction between the chains also provides great mechanical performance.

Ionic liquids are powerful solvents that can be used to dissolve cellulose. They are composed entirely of molten salts that are liquid at ambient temperatures. Ionic liquids are often referred to as “green” solvents due to their low vapor pressure and chemical stability. Dissolution of cellulose is possible in the ionic liquid 1-Ethyl-3-methylimidazolium acetate (EmimAc) and the process doesn’t result in any significant degradation of the cellulose polymer chain.

The use of ionic liquids has opened up for a wide range of applications, making it possible to dissolve cellulose and reproduce it by coagulation. For cellulose to be successful as an alternative to synthetic polymers it is important to develop methods that control the structure and properties of the materials made from cellulose. The use of 3D printing as a bottom-up fabrication method is increasing and is becoming more user friendly and affordable. To develop a method of printing cellulose could be an efficient way of controlling the structure of regenerated cellulose.

Via bottom up fabrication this project aims to control the structure and properties of polysaccharides by printing 3D structures of cellulose dissolved in ionic liquids followed by regeneration by coagulation.

Figure: Left; Solutions of dissolved cellulose in EmimAc. Middle; Regenerated cellulose film. Inset; Chemical structure of EmimAc. Right; Printing with cellulose solubilized in EmimAc.
Effects of graphene preparation methods on the photocatalytic performance of TiO$_2$/graphene composites

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In this work, we aim at elucidating the effect of graphene synthesized by different techniques on the photocatalytic performance of TiO$_2$/graphene composite systems. Graphene was prepared by widely used techniques such as catalyst-assisted chemical vapor deposition (CVD), catalyst-free CVD and spin coating from commercially available graphene oxide solution. Of special importance is given to assess electrical conductivity and surface morphology of graphene. The prepared graphene was characterized by AFM, four-probe, and PL techniques. It was observed from AFM that the prepared graphene by different methods exhibit different morphologies. The sheet resistivity of catalyst-assisted graphene was lowest and followed by catalyst-free and solution processing as determined from four-probe techniques. The prepared photocatalysts showed enhanced photoactivity compared to bare TiO$_2$ for the evaluated methanol photooxidation test in the UV region. However, the enhanced photocatalyst was different for different preparation techniques. The enhanced activity was attributed to improved charge carrier separation as confirmed by PL measurements. It was also revealed from PL studies that charge carrier lifetime of catalyst-assisted graphene is lower compared to catalyst-free graphene implying the former has lesser defects than latter. This trend was also observed in photocatalytic activity as well as in sheet resistivity measurement. From these results, it is confirmed that graphene prepared by different techniques have great impact on photocatalytic performance because of difference in electrical conductivity and interfacial properties of graphene. Finally, the composites were compared with graphitic carbon and Au thin films based composites to emphasize the importance of interface and electrical conductivity on the performance of photocatalysts.
Atom probe tomography of corroded Zircaloy-2

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Due to their low thermal neutron capture cross-section, zirconium alloys are widely used in the nuclear industry for fuel cladding and structural components. The lifetime of the fuel assemblies in the reactors are largely dictated by the ability of the fuel cladding to withstand corrosion and mechanical damage. The waterside corrosion mechanism of zirconium alloys is closely related to another material degradation process, namely hydrogen pick-up. In order to study the hydrogenation of zirconium on the atomic level, atom probe tomography (APT) is utilized. This technique offers some unique virtues for nanometer scale material analysis, such as equal sensitivity to all elements.

The metal-oxide interface in corroded Zircaloy-2 was studied using APT. High resolution chemical analysis of region of interest for oxidation and hydrogen pickup kinetics are examined, such as grain boundaries and secondary phase particles.
Fate of Hydrogen upon Oxidation of Zirconium Alloys by Water
A First Principles Study of Chemical Reactions in the Solid State

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Department of Chemical and Biological Engineering Division of Energy and Material

Design of corrosion resistant zirconium alloys in air-free water relies on a compromise between two opposing strategies, minimize the oxidation rate and minimize the hydrogen pick-up by favoring hydrogen evolution. Understanding these processes is of major importance to the nuclear industry where zirconium alloys are used as fuel claddings. In this study, the oxidation of zirconium by water is analyzed in terms of an anode reaction and a cathode reaction by means of first principle calculations. The anode reaction involves Zr oxidation by oxide ions resulting in release of electrons. Details in the complementary cathode reaction is of particular importance owing to the dramatic influence that transition metal additives have on the fraction of hydrogen picked up in the alloy.

Efficient utilization of the exothermic Zr oxidation reaction by water is found to drive proton-electron recombination by forming reactive hydride intermediates. Once formed, the hydride can undergo hydride-proton recombination forming H₂. The magnitude of the exothermicity of this recombination is found to be dependent on transition metal and correlates to the transition metal dependence of hydrogen pick-up fraction: Larger exothermicity implies smaller hydrogen pick-up fraction.

ZrO₂ is unstable in contact with Zr metal resulting in oxygen dissolution in the metal matrix. The impact of the high solubility of oxygen in Zr in the effective anode potential is addressed. In particular, the ability of the anode reaction to sustain the cathode reaction is demonstrated for relevant suboxide concentrations. Moreover, the influence of oxygen dissolution on the hydrogen pick-up is addressed and found to be minor also for concentrations well above the suboxide saturation limit. Support for both relevance and validity of the analysis is provided by semi-quantitatively reproducing the experimentally observed thermodynamic oxygen solubility limit in Zr.
Environmentally friendly plasticized PVC by means of nanotechnology

Henrik Petersen,
SP Technical Research Institute of Sweden Materials and Manufacturing Technology

Polymer composites and especially nanocomposites attract a lot of attention not only from a scientific point of view but also within a wide range of industrial applications. With the incorporation of nanofillers in a polymer matrix it is possible to improve several important properties such as mechanical strength, barrier properties and flame resistance. Despite extensive research efforts a rather limited number of polymeric nanocomposite products have made their way into the market.

Polyvinyl chloride (PVC) is one of the world’s most widely produced polymers used in areas such as building, transport, packaging, electrical/electronic and healthcare. Manufacturing of PVC is dependent of several additives, e.g. plasticizers, heat stabilizers and flame retardants. The leakage of particularly plasticizers from PVC reduces the products usable lifetime and can cause health and environmental effects.

In this project the focus lies mainly on reducing the migration of additives in plasticized PVC. The idea is to exfoliate our choice of nanofiller, Montmorillonite (MMT) clay, in the polymer matrix. Below is briefly explained the mode of procedures we intend to carry out.

- Organically modify a nanoclay suitable for PVC
  An organic molecule is intercalated into the Montmorillonite clay galleries with the purpose to expand interlayer distance and to make the clay platelets more compatible with the polymer matrix (fig. 1).

- Optimize processing conditions for PVC nanocomposites
  By shear and diffusion processes polymer chains penetrate the clay galleries and then eventually clay platelets are peeled apart to generate an exfoliated nanocomposite (fig. 2).

- Manufacture PVC nanocomposite prototypes
  In the final stage prototypes will be manufactured using industrial production methods and evaluated with regards to their performance, emissions and durability.
Heading towards eco-efficient surface protection: To explore aspects that govern release from microparticles

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To protect a painted house facade from e.g. mold or algae, anti-growth agents are mixed in the paint. Most of the applications, used in the past and based on heavy metals, have today been banned due to their negative impact on the environment. Alternative treatments are therefore required to improve growth and decay resistance. A major problem with the biocides that are used today is that their protective ability is only sufficient during a short period of time. Biocides are small molecules and diffuse rapidly inside the paint matrix. By fast leaching and rinsing with the rain water the protection disappears. A promising improvement of anti-growth protection can be achieved by the use of encapsulated biocides that slows down the diffusion in the coating. The biocide is placed into microparticles, from where it is slowly distributed into the surrounding coating matrix. A microparticle can basically be either a microcapsule (core-shell structure) or a microsphere (homogenous polymer matrix). The preparatory step involves the solvent evaporation technique. The specific research objective of this PhD project is to explore and understand the fundamental mechanisms that govern controlled release of actives from microparticles. As the project focuses on a variety of parameters that influence the release, several experiments methods will be considered. In a long-term perspective, the ambition is to develop surface protection systems that are both economically and ecologically sustainable.

Figure 1: Light microscopy photo of microcapsules.
Delubrication of Powder Metallurgy steels

Seshendra Karamchedu

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Powder metallurgy (PM) is a manufacturing route wherein powders are used as the starting material and are converted into useful components through processing. The processing steps in a typical process for PM steels consist of compaction at room temperature, where the final shape of the component is achieved, followed by a temperature assisted sintering process which imparts sufficient strength to the compact through the formation of metallurgical bonds between the particles. The powders are mixed with lubricants prior to the compaction stage since they reduce the friction between the powder particles and between the particles and the die-wall, thereby promoting compaction homogeneity and decreasing die-wear. However, these lubricants have to be removed after the compaction stage since they hinder the sintering process.

Lubricants commonly used for PM steels are based on ethylene bis-steramide and lubricant removal is achieved through thermolysis in the initial zone of the sintering furnaces. Improper delubrication can lead to defects in parts such as micro-cracking and sooting and can also lead to deposition of delubrication products on the furnace components and compacts. This results in a decrease in productivity and increase in maintenance costs. Hence an industrially feasible method for monitoring delubrication process would be valuable. In the present work, a methodology for monitoring the delubrication process has been proposed based on analyzing the processing atmosphere through sensors (dew-point, CO₂ and O₂) commonly used in the industry (see Figure 1-a). Through the experiments performed using the laboratory set-up, it has been shown that the O₂ sensor is useful in monitoring the initial stages of delubrication and the CO₂ sensor can be used to monitor the later stages (see Figure 1-b). Using the proposed methodology a systematic study of the effect of various process parameters- heating rate, temperature, effect of graphite addition, composition and purity of the process atmosphere on delubrication has been performed. Based on the results it has been suggested that delubrication has to be performed at 450°C in dry N₂ atmosphere at a slow heating rate with flow sufficient enough to provide dynamic conditions around the component surfaces.

Figure 1 a: Set-up for process atmosphere monitoring, b: A monitoring profile generated using the set-up
Evolution of the microstructure during creep testing of WC-Co based cemented carbide


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Abstract

The evolution of WC grain size during high temperature creep deformation of a cemented carbide has been studied. The material had a Co content of 8.37 wt.% and smaller additions of Cr (0.84 wt.%). A hot compressive creep test was performed at 1000 °C under an applied load of 900 MPa for 700 seconds. The WC grain size was determined before and after deformation by two different methods, i.e. linear intercept measurements from scanning electron microscope images and electron backscatter diffraction techniques. The results indicate that WC grain growth, preferentially perpendicular to the load axis, took place during creep deformation. It is suggested that plastic deformation of WC grains leads to dislocations emerging at WC grain surfaces, and that these sites facilitate nucleation of atomic layers of W and C, and thus grain growth, at this comparatively low temperature. In addition, formation of binder phase lamellae at some WC / WC grain boundaries was observed, suggesting that also grain boundary sliding took place during deformation.

Keywords

Cemented carbide, EBSD, Grain growth, Linear intercept, Plastic deformation.
Waste Electrical and Electronic Equipment (WEEE) Plastics Composition

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The lifetime becomes shorter and shorter in society for electronic equipment, influenced by new technology, fashion and competition. This results in an increasing waste stream of what is called waste electrical and electronic equipment (WEEE). Traditionally metal recycling has been profitable and straightforward from WEEE, but at least 1/3 of the weight of WEEE consist plastics. Plastics recycling have struggled with separation problems of different plastics from each other due to similar properties and many different types of plastics used, making separation costly and complicated. Of this reason this study focuses on the composition that can be expected from a WEEE plastics stream of recyclable plastics. The composition analysis of the plastics was based mainly on 14 samples collected from a real waste stream, by direct sampling from a falling stream. The waste material, consisting of plastic flakes, was identified by infrared spectroscopy. The results showed that the major constituents were high impact polystyrene (42 wt%), acrylonitrile-butadiene-styrene copolymer (38 wt%) and polypropylene (10 wt%). Their respective standard deviations were 21.4%, 16.5% and 60.7%, indicating a considerable variation even during a short sampling time of 3 hours. Trends of the three major constituents from the 14 samples extracted in chronological order were also studied. A hypothesis test of the slope of the trend-line (with a significance level of 0.05) did however not indicate any trend-like behaviour for the three material groups during the sampling time.
Real time *in situ* monitoring of catalyst sintering on different support materials

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*Materials Science Graduate School, enrolled in August 2011

Heterogeneous catalysts are often realized as nanometer sized metal particles dispersed on high surface area oxides, such as Al$_2$O$_3$. However, due to large surface to volume ratio of small metal particles they are prone to sinter (i.e. coalesce into large particles) under operating conditions. This is a major cause of catalyst deactivation and leads to additional costs with either metal overloading or regeneration procedures. In this project, indirect nanoplasmonic sensing (INPS) [2] is used to monitor the sintering kinetics of Pt nanoparticles in an oxidizing environment on different support materials in real time, *in situ* and under operating conditions. Recently, INPS in combination with transmission electron microscopy (TEM) was used to explore sintering of Pt nanoparticles on SiO$_2$ [3]. The figure shows our results from TEM and INPS experiments.

![Figure 1](image_url)

**Figure 1.** (Left) Normalized INPS signal related to sintering of Pt nanoparticles at 592°C in 4% O$_2$ in Ar on different support materials. The result for a sample without Pt (blank) exhibits no significant plasmonic shift. (Right) TEM images illustrating the sintering of Pt nanoparticles supported on sputtered silica at 597°C. (a) Fresh sample, (b) after 10 minutes, (c) after 1 hours and (d) after 6 hours. The scale bar represents 20 nm.

*In situ* Monitoring of sintering under operating conditions with high temporal resolutions is important to understand the sintering mechanisms. Scrutinizing the role of support materials is critical because (i) it can shed light on the importance of support-particle interactions and (ii) as it could pave the way to minimize sintering effects.

**References**

Surface Chemical Analysis of Soft Magnetic Composite Powders

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Soft Magnetic Composite (SMC) components produced based on traditional Powder Metallurgical (PM) techniques are strong candidate materials for electromagnetic applications. Their advantages are based on profitable and energy efficient production methods, shape complexity realization and uniquely uniform and isotropic 3D magnetic properties. SMC powder grades consist of encapsulated iron powder particles with an ultra-fine, electrically insulating surface coating. Component manufacturing procedure involves compaction of the admixed SMC powder with a lubricant to a final shape, as well as a subsequent heat-treatment that aims on the relaxation of stresses induced during the compaction. The concept of SMC is based on taking advantage of the dielectric properties of the surface coating and creating a laminated stack in a powder form. In this manner, products with superior magnetic performances can be produced as opposed to the more traditional laminated steels and ferrites, due to the higher reduction of core losses especially at high frequency applications.

It is obvious that the insulating coating is the paramount feature of the SMC technology. Its morphology, thickness, cohesion to the powder particles and durability during compaction and heat-treatment, are critical for the properties of a finished component. Hence, a methodology based on surface sensitive analytical techniques was developed in order to address these matters on a micro-level for such insulating coatings. Standard methods previously used for the powder surface characterization have been significantly modified due to the non-conductive nature of the surface coating, which led to charging during analysis. For this reason charge compensation was confronted by adding slow electrons and software correction. Under these conditions two commonly used sample preparation techniques were implemented and compared. Additionally, high resolution imaging combined with spectroscopic techniques and depth profiling (HR-SEM, EDX and XPS) provided complementary results for the coating characterization. Finally, the thickness of the insulating coating was evaluated based on a model that takes into account the specific geometry of the spherical particles and the experimental setup. The results indicate its thickness to be in the range of few tenths of nanometer.
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