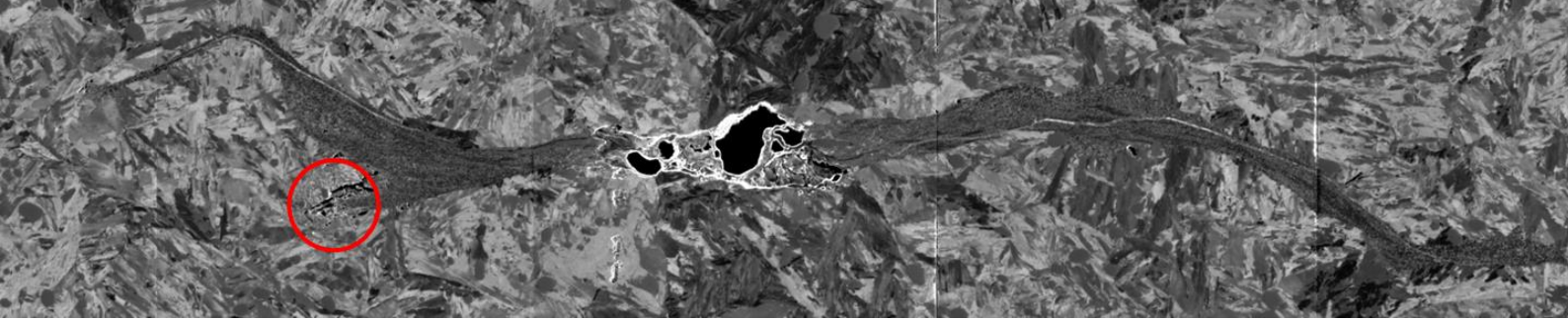
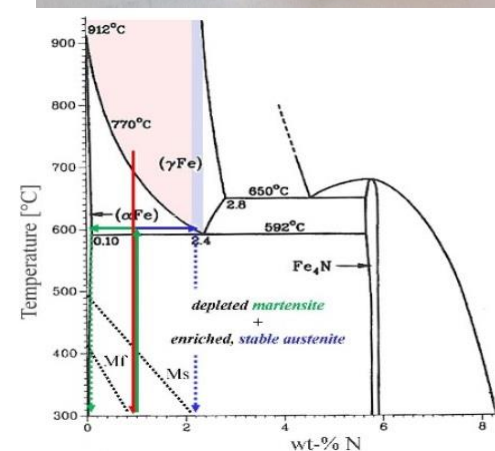
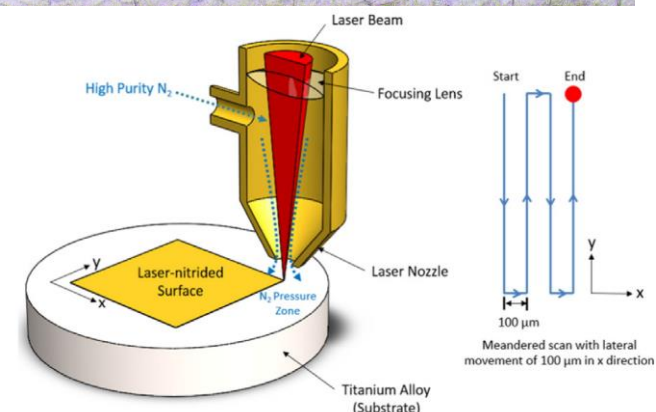
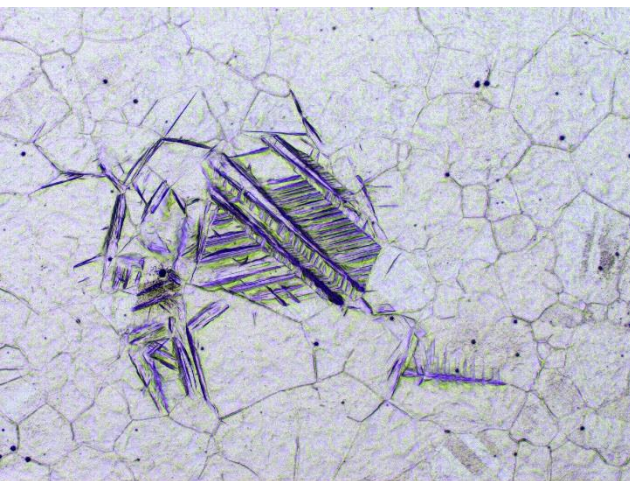




Materiale- og overfladeteknologi

Materials and Surface Engineering

Student projects Autumn 2017



The section for Materials and Surface Engineering has expertise in many aspects of materials science and engineering and covers disciplines as materials synthesis, materials solutions, materials characterisation, materials performance and materials modelling. The section consists of 8 faculty staff and teaches and researches in topics covering fundamental materials science as well as innovative materials engineering solutions. Within the section three broad titles cover the many activities.

Surface engineering and materials design

Surface engineering is the major materials synthesis activity in the section and for this purpose electrochemical, thermochemical and PVD techniques are applied and further developed. The research activities in surface engineering cover the entire chain from basic research of generic importance to applied research, engineering and innovation.

Microstructure evolution and phase transformations

Materials microstructure evolution during processing as well as during application lies at the very essence of understanding the relations between process parameters, properties and performance. Understanding the occurring microstructure evolution and phase transformations in materials forms the basis for materials modelling activities.

Materials performance and degradation

Research in materials performance and materials degradation is concentrated on corrosion, tribology and high temperature performance (creep and corrosion), i.e. the chemical, biological, mechanical and thermal interaction (or combinations thereof) of materials with the environment. The strategy is to investigate, identify and understand the degradation mechanisms that affect the performance of materials and to use this understanding to predict the life expectancy and design of improved materials solutions.

Currently, we focus on materials for (sustainable) energy applications and materials in medical applications. We have a broad international network of collaboration within academia and industry.

Faculty staff:

Marcel A.J. Somers- head of section

John Hald

Rajan Ambat

Seunghwan Lee

Karen Pantleon

Wolfgang Pantleon

Grethe Winther

Thomas Lundin Christiansen

List of projects waiting for students:

High temperature corrosion in materials for power plants
Biophysics, biomechanics, and biotribology of mucins and mucus gels
Improving tribological and biocompatibility of orthopaedic implants
Can Fe-C coatings replace hard chrome coatings?
Improving the quality of hard chrome by pulse plating
Hydrogen in superheater tubes and its influence on steam oxidation in thermal power plants
Optimization of a dual phase model alloy
Refinement of Si containing ferritic alloys for applications in biomass fired power plants
Surface optimization of steel via shot peening
TWIP steel for crash absorber for automotive industry
Quality of laser cladding for repair purposes
Material optimisation to optimise production of filter house
Nanoindentation to determine thermo-chemically induced changes in mechanical properties at the grain scale
Forming limits after strain path changes in ferritic and austenitic steels
Improved materials for fusion reactors
Monitoring fatigue in fcc metals by X-rays
Intrinsic versus enforced anisotropy in plastic deformation of single crystals
Hysteresis and relaxation of springs in auto injectors
Mapping the microstructural path
Grain refinement by combined martensitic transformation and plastic deformation in steels
Corrosion of materials in oil and gas applications
Climatic reliability of electronic devices
Material characterization of ECG electrodes
Materials and surfaces for electrical connectors
Surface modification of aluminium alloys
Sulphur corrosion of silver
Investigation of martensite formation in Fe-29%Ni model alloy
Martensite formation in AISI 301
Can advanced Fe-N steels be produced?
Effect of Cu precipitates on the microstructure of low C martensitic steels
Studying bainite formation on heating?
Investigation of reverse austenite formation in stainless steels
Application of cryo-treatment for a new generation of martensitic stainless steels
Synthesis and characterization of High Entropy Alloys with interstitials
Development of porous iron and metastable iron phases
Development of porosity in iron-nitrides
Surface hardening of titanium alloys
3D printed stainless steel impellers
Characterization of white etching cracks in bearings for wind turbines
Development of irregular white etching cracks in bearings for wind turbines
Long-term microstructure stability of 12%Cr steels in Danish power plants
High N martensitic steel for high temperature steam turbines
Kinetics analysis of the formation and retainment of δ -ferrite in cast low-carbon martensitic stainless steel
Microstructural evolution in centrifugally cast high temperature austenitic alloys

High temperature corrosion in materials for power plants

Supervisors: Melanie Montgomery, Karen Pantleon, Yohanes Malede

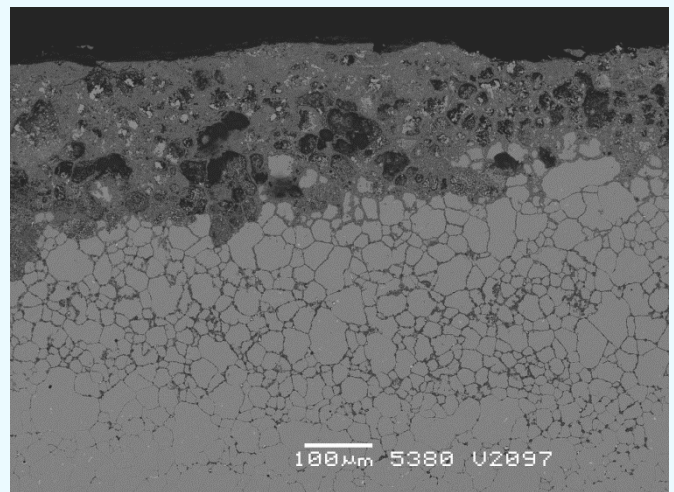
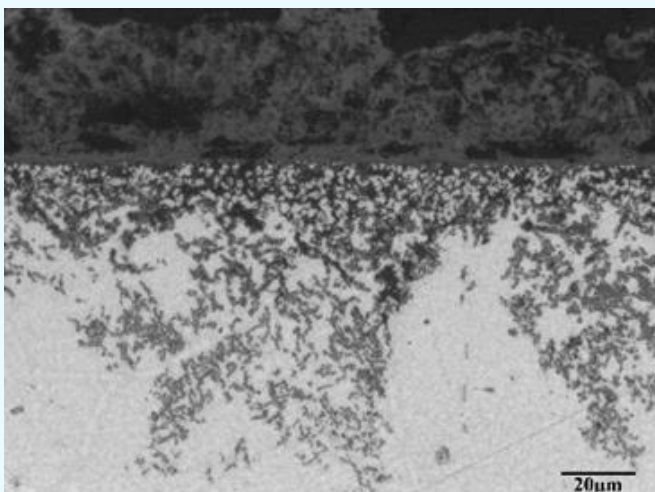
The lifetime of superheater tubes in biomass firing thermal plants depends on the materials response to high temperature exposure together with a complex aggressive chemical environment with variations in flue gas, deposition and temperature. To understand the mechanisms of high temperature corrosion, studies under well-controlled laboratory conditions are required mimicking specific parameters in the power plant. BSc/MSc projects are offered where a specific parameter is investigated as suggested in the examples below:

Experimental

An austenitic steel (aged or as received or heat-treated) will be exposed to KCl. Different parameters can be varied to investigate specific mechanisms, for example:

- Gas atmosphere: air, 15% H_2O +5% O_2 (standard exposure conditions), 50% H_2O + 5% O_2 ,
- Temperature: different temperatures 560°C (temperature of actual boilers), 600°C, 640°C, and possible temperature cycling.
- Various deposition of salts e.g. KCl, K_2CO_3 : a) immersion, b) the salt dissolved as slurry, c) the specimens are placed over the salt d) salt as powder.

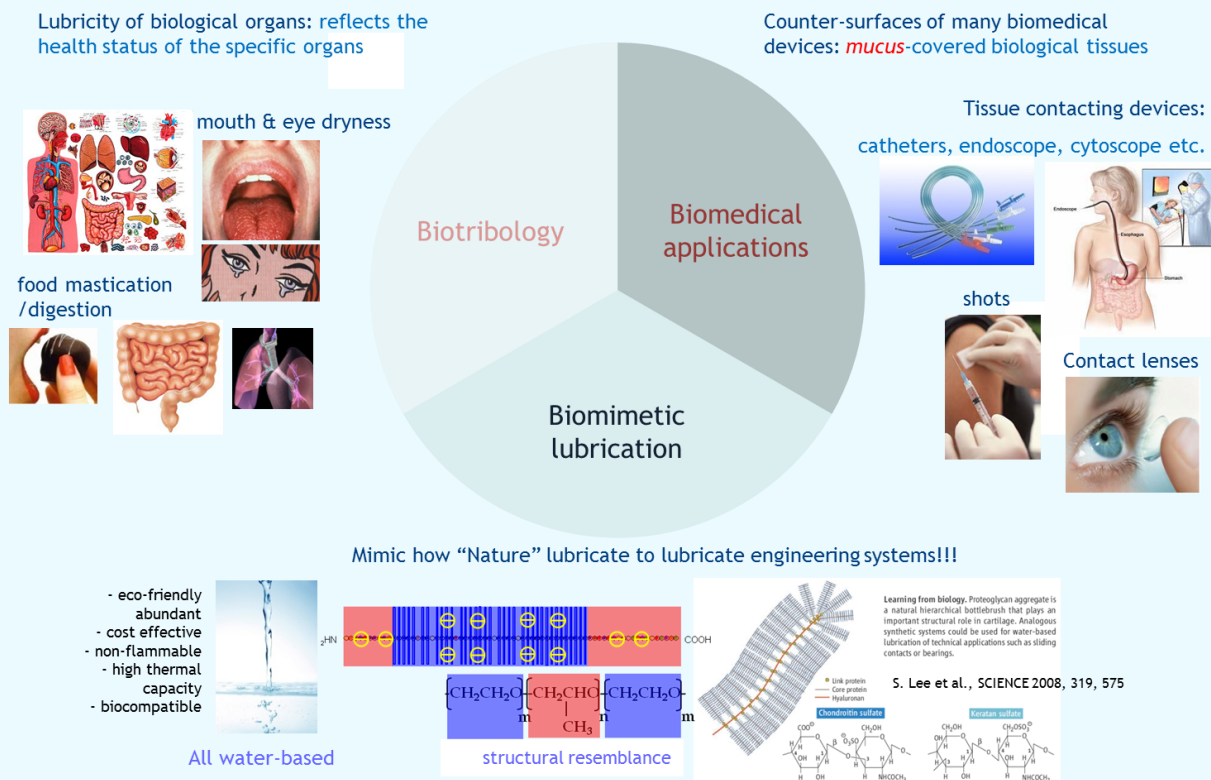
All projects require literature study and a lot of experimental work including high temperature corrosion experiments, and materials characterisation (microscopy, diffraction, spectroscopy).



Biophysics, Biomechanics, and Biotribology of Mucins and Mucus Gels

Supervisor: Seunghwan Lee (seele@mek.dtu.dk, Tel: 4525 2193)

The lubricity of mucus gels/mucins are often considered granted. However, systematic scientific researches and understanding of this topic is surprisingly scarce to date. The significance of lubricity of mucin/mucus gels is mainly three-folded; (1) **Biotribology**: “good lubricity” by mucus layers often indicates the state of health in respective organs; for instance, impaired lubricity of mucus gels leads to discomfort in eye blinking, dryness in oral cavity and disturbed mastication, several airway diseases (asthma), digestion problems in stomach, etc. (2) **Biomedical applications**: in the application of various biomedical devices and personal care products, in particular, tubular tissue-contacting devices such as catheters, endoscopes, and cytosopes, and contact lenses, counter surfaces that interact with them are mucus-gel-covered, soft biological tissues. Thus, it is of prime significance to understand the fundamental lubrication properties of mucus layers for optimum operation of such devices. In turn, it can provide many useful hints on how to tailor the surface properties of the medical devices for most favorable interaction with the biological tissues. (3) **Biomimetic lubrication**: efficacy of the lubrication by mucus gel layer is remarkable when it is considered that the base lubricant employed in biological systems is water; while many previous studies have shown that surface-adsorbing, brush-forming polymers behave as good aqueous lubricant additives, mucus gels can provide even more powerful approaches to utilize water as lubricant.



Multiple bachelor and master projects are currently available with diverse focuses on biophysics, biomechanics, and biotribological properties of mucins and mucus gels, depending on the specific interests of students.

Improving tribological and biocompatibility of orthopaedic implants

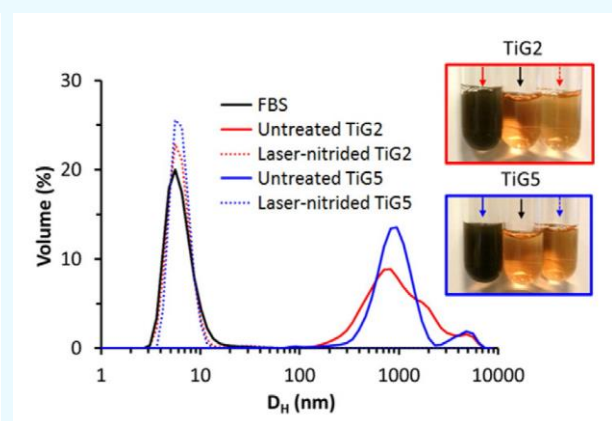
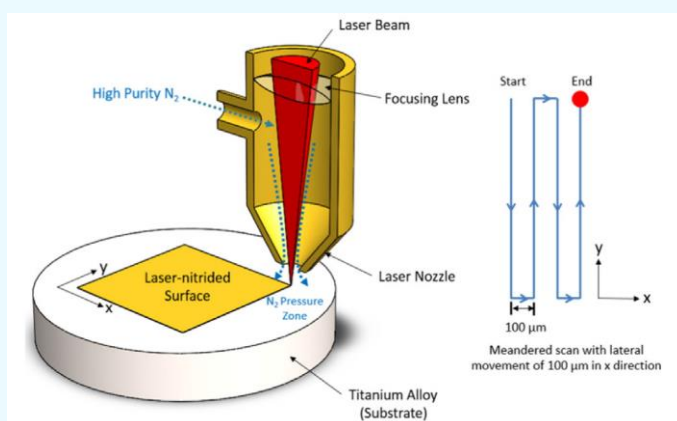
Seunghwan Lee (seele@mek.dtu.dk, tel 4525 2193)

in collaboration with Dr. Chi-Wai Chan (Queens University of Belfast, UK)

Wear of articular joint implants, such as those used in total hip arthroplasty (THA) or total knee arthroplasty (TKA), has long been recognized as the principal cause of inflammatory bone loss and late revision of hip replacements. The particulate wear debris can initiate a cascade of adverse tissue responses, leading to osteolysis (bone resorption), consequent aseptic loosening of the components, and ultimately to failure of the implants. In this sense, biocompatibility and long-term stability are two faces of the same problem for articular joint implants; currently prevailing materials employed for orthopaedic implants, including ultrahigh molecular weight polyethylene (UHMWPE), metal alloys (CoCrMo), and ceramics (alumina or zirconia-toughened alumina (ZTA)), do not display any harmful biological problems in bulk form, but many of them do in particulate form, which evolves under tribological contact stress. It is therefore critical for the materials employed for articular joint implants to display excellent tribological properties (i.e. low friction, low wear), not only for smooth sliding and mechanical stability, but also for improving biocompatibility of the implants in the human body. The long-term stability of articular joint implants is a more significant issue for younger patients who receive joint replacement surgeries, due to the anticipated necessity for revision surgeries.

A number of Bachelor, Master, or special projects are available depending on materials of particular interest, ranging from metallic (e.g. CoCrMo alloys, Ti alloys, and stainless steel etc), ceramic (e.g. alumina) to polymeric materials (e.g. UHMWPE). Typically, laser nitriding (in collaboration with the Queens University of Belfast, UK) is employed to enhance hardness of metallic materials whereas fluidic polymer lubricants are used to suppress the production of wear particles for ceramic and polymeric materials.

[1]



References

- [1] C.-W. Chan, S. Lee, G. Smith, G. Sarri, C.-H. Ng, A. Sharba, and H.-G. Man, "Enhancement of wear and corrosion resistance of beta titanium alloy by laser gas alloying with nitrogen", *Applied Surface Science*, 2016, 367, 80-90.
- [2] T. Røn and S. Lee, "Aqueous lubrication of alumina surfaces as assisted with surface-adsorbing polymers" *Wear*, 2016, 368-369, 296-303.

Can Fe-C coatings replace hard chrome coatings?

Supervisors: **Jacob Obitsø Nielsen (jacobon@mek.dtu.dk)**

Karen Pantleon (kapa@mek.dtu.dk)

Improving the mechanical properties and performance of components for applications under extreme mechanical loading, like for example for bearings, is one of the key issues within surface engineering. Electrochemical deposition as a rather efficient, industrially feasible and widely applied process for numerous engineering applications. Although not being one of the most traditional electrochemically deposited materials, the formation of Fe-C coatings is a promising alternative to bulk steel components requiring extensive surface treatments for strengthening. Fe-C coatings will allow the changeover to more environmental friendly, hard and wear resistant coatings compared to the presently predominant hard chrome coatings, assuming that they meet the demands on mechanical and corrosion properties.

The interplay between different process parameters (e.g. plating additives, pH and temperature of the electrolyte, as well as current density, type of current, current efficiency) and the experimental set-up (e.g. pulse plating) affects the internal structure (e.g. grain size and shape, preferred crystallographic orientations, stresses and strains) and associated properties (e.g. hardness, wear and corrosion resistance) of the Fe-C coatings. Systematic studies are required for understanding the individual and synergetic influences for finally optimizing the quality of the deposited coatings.

Various BSc or MSc projects are offered, where each of the projects can address one or more of the following issues (specific content and corresponding time frame can be adapted as needed):

- either
- 1. Improving properties:** The galvanic coupling between Fe and C is responsible for an unwanted corrosion of the Fe-C coating when exposed to humid atmosphere or water. Alloying the coating with elements capable of passivating the surface to prevent corrosion will broaden the applications from being limited to oil based environments. Alloying can be done either by diffusion or co-deposition.

or

 - 2. Pulse plating:** In the typical electroplating process, direct current (DC) is supplied to the plating bath as straight DC current. In pulse plating, the DC power arrives in the bath in a series of short pulses (up to over 10,000 times per second). Precise changes in the regulation of these cycles should affect the plated deposits (e.g. better materials distribution, lower porosity, smaller grain sizes and associated improved mechanical properties).

or

 - 3. Thermal stability of as-deposited coatings:** The nanocrystalline nature of the as-deposited coatings implies the risk of microstructure evolutions with time and temperature, which would affect mechanical strength and wear resistance. The link between the final stable structure and the process parameters is essential for tailoring the coatings towards their long-term applications at elevated temperatures.
- or a combination of ideas within Fe-C coatings.

All projects require literature study and a lot of experimental work with materials synthesis and/or materials characterization and testing.

Improving the quality of hard chrome by pulse plating

Supervisors: Jacob Obitsø Nielsen (jacobon@mek.dtu.dk)

Karen Pantleon (kapa@mek.dtu.dk)

and collaboration with a.h. nichro Haardchrom A/S, Hvidovre

Electroplating is a rather efficient, industrially feasible and widely applied process and provides a huge potential for numerous engineering applications. The predominant coating for mechanical applications is hard chrome, but it is known for forming undesirable macro-cracks during deposition and, therefore, requires the use of sub-coatings like bright-nickel or electroless nickel to ensure efficient corrosion protection of the base material in highly corrosive environments.

In the typical electroplating process, direct current (DC) is supplied to the plating bath as straight DC current. In contrast, during pulse plating the DC power arrives in the bath in a series of short pulses and their duration, current and shape affects the plated deposit. Typical advantages of pulse plating include better material distribution, lower porosity, higher density and larger throwing power.

The Danish company a. h. nichro Haardchrom A/S has started designing and plating multilayer coatings containing separate layers of pulse plated chromium and DC-plated chromium. The adherence of the plated multilayer coating is very good, even after mechanical influences on the coating. The corrosion resistance is excellent (more than 500 hours in salt spray test), but depends very much on the multilayer design and the process parameters during plating.

Various BSc or MSc projects are offered in cooperation with a. h. nichro Haardchrome A/S, where each of the projects can address one or more of the following issues (specific content and corresponding time frame can be adapted as needed):

- Controlling the layer types (DC / pulse), number of layers, the thickness of each layer and their location in the plated coating for optimizing the process parameter towards corrosion resistance.
- Linking the process parameters to the resulting microstructure, morphology and associated properties. This relies on advanced methods of materials characterization.

The projects require literature study and a lot of experimental work with materials characterization and testing.



Large-scale electroplating at
a.h. nichro Haardchrom A/S



Hydrogen in superheater tubes and its influence on steam oxidation in thermal power plants

Supervisors: Karen Pantleon (kapa@mek.dtu.dk),
Melanie Montgomery (mmon@mek.dtu.dk)

Steamside oxidation of superheater tubes applied in thermal power plants can cause serious problems, because oxide spallation can result in blockage of the loops and cause insufficient steam flow through the superheater and, consequently, overheating and failure of the tube.

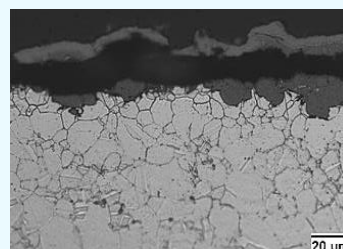
It is anticipated that the presence of hydrogen in steel, originating from hydrogen dissolution during service conditions in the power plant, affects the rate of oxidation on the steamside of the superheater tubes.

The effect of hydrogen in steel is often directly related to the loss of ductility, i.e. hydrogen induced embrittlement. However, the role of hydrogen can be manifold and includes, for example, the initiation of phase transformations, changes of crack growth, localization of plastic deformation, weakening of interatomic bonds, changes in the type, number and interaction of lattice defects. All these hydrogen induced changes, indeed affect the mechanical properties and enhance embrittlement, but simultaneous influences by a certain chemical and thermal environment may further explain the effect of hydrogen on the lifetime of steel in these conditions.

A student project (MSc or BSc) aims to contribute to understanding the mechanisms of steam oxidation in hydrogen containing austenitic superheater tubes. The project requires thorough literature study and comprehensive experimental work including various aspects, as for example:

- systematic hydrogen-charging of austenitic steel (electrochemical treatment compared to thermal treatment in a furnace)
- stability over time as a function of thermal and/or mechanical influences
- testing of hydrogen loaded samples (mechanical tests, chemical analysis),
- high-temperature exposure of samples with and without hydrogen to steam oxidation conditions in laboratory furnaces at industrial relevant conditions,
- microstructure characterization of the near-surface region applying microscopic, spectroscopic and diffraction techniques, with particular focus on hydrogen-induced phase transformations and the evolution of internal stresses.

(note: the content will be agreed according to the type and time frame of projects):



Optimization of a dual phase model alloy

Supervisor: Karen Pantleon (kapa@mek.dtu.dk)

The development of advanced methods for materials characterization often requires well defined, simplified materials both for testing purposes and fundamental materials investigations.

For example, for understanding the elastic and plastic deformation behaviour of multiphase materials on the sub-micron scale, a copper-iron alloy is considered a suitable model system.

As copper and iron are practically immiscible at room temperature, the resulting microstructure of the iron-copper alloy will consist of well-separated regions of copper and iron. These two single phases with their specific crystal structure (FCC copper and BCC iron) represent an interesting combination of essentially different mechanical properties within the multiphase material.

The project aims at

- the **synthesis of such a model alloy** consisting of 50wt% Cu and 50wt% Fe
- the **optimization of the internal structure** of the alloy with special focus on homogeneous distribution of the two phases and uniform grain sizes in the range of only a few micrometers up to several tenths of micrometers
- comparison of such Cu-Fe bulk alloy with a **coating of alternating Cu- and Fe-layers**

The practical work involves, in addition to appropriate heat treatment procedures, thorough analysis of the microstructure applying various microscopic techniques (light and electron microscopy) and X-ray diffraction for the phase-specific determination of preferred crystallographic orientations of grains and internal lattice strain.

The project is meant to be a MSc project, but parts of it can be formulated as a BSc project, if requested.

Refinement of Si containing ferritic alloys for applications in biomass fired power plants

Supervisors: Karen Pantleon (kapa@mek.dtu.dk),
Matteo Villa (matv@mek.dtu.dk)

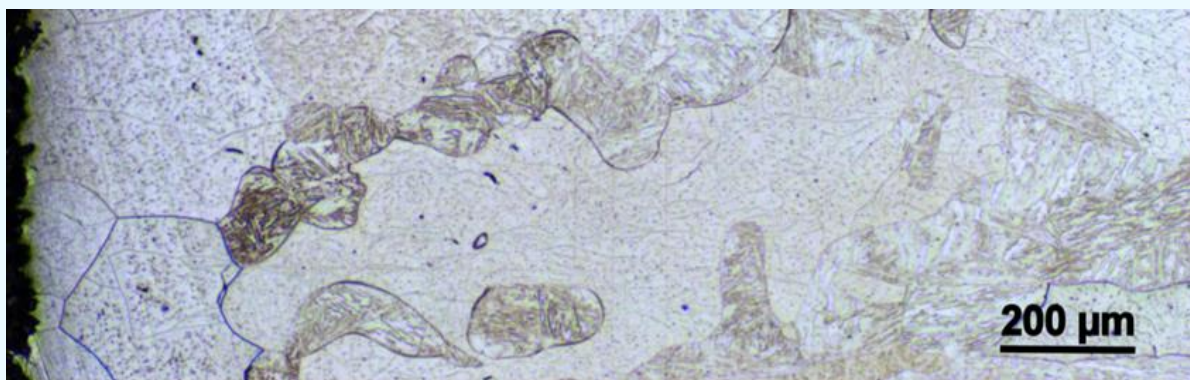
As renewable energy source, biomass becomes more and more attractive for replacing traditional coal firing in thermal power plants. However, during combustion of biomass, superheater tubes are exposed to oxidizing atmospheres containing aggressive solid and gaseous species, e.g. KCl, HCl and SO₂. The resulting more severe corrosion attack when (co-)firing biomass compared to traditional fossil fuels is currently limited by reducing the steam temperature in the plant, which, however, also reduces its efficiency. For full exploitation of biomass combustion with increased plant efficiency and lifetime despite the aggressive conditions, new materials solutions solving the tremendous high temperature corrosion problem are required.

Recent research indicates Si containing ferritic stainless steels as possible candidates for extending the service temperature of metallic materials in oxidizing atmospheres containing aggressive chemicals.

In Fe-Cr-Si alloys, passivation against high temperature corrosion under biomass firing conditions can be obtained by the formation of a continuous silica SiO₂ healing layer underneath the typical chromia layer Cr₂O₃ at the surface. Formation of such protective double oxide layers requires diffusion of the alloying elements from the bulk towards the surface, which strongly depends on the microstructure.

Present alloys obtained by casting reveal an extremely coarse microstructure, which is particularly unfavorable to promote diffusion of alloying elements to the surface. Classical routes for obtaining grain refinement recently revealed, at least, local SiO₂ formation, but a continuous protective oxide layer is not obtained yet.

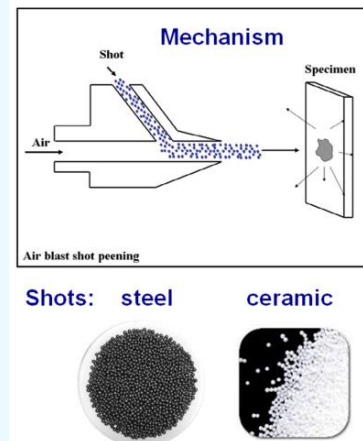
In the present project (MSc or BSc), refinement of the microstructure near the surface should be obtained by an unconventional route, consisting of promoting a series of phase transformations in the material by changing its chemistry during heat treatment. The proposed process consists in high temperature solution nitriding/denitriding and carburizing/decarburizing of the material, which is expected to promote continuous grain refinement by alternatively stabilizing (and forming) different phases. Final exposure of the refined microstructure to conditions mimicking biomass firing will be used to evaluate applicability of the alloy as superheater in thermal power plants.



Surface optimization of steel via shot peening

Supervisors: Karen Pantleon (kapa@mek.dtu.dk)
Xiaodan Zhang (xzha@mek.dtu.dk)
Xiaoxu Huang (xihu@mek.dtu.dk)

Shot peening is a process in which either hard steel spherical shots, or ceramic or glass beads/shots are shot against a target to mechanically treat its surface and to introduce compressive residual stresses and microstructural refinement. These surface modifications improve the fatigue resistance by preventing or delaying cracking, and also affects further properties, like the material's corrosion resistance. Shot peening is a feasible industrial process and widely used for components and parts such as bearings, shafts and gears in the aerospace, chemical, petroleum, automotive and power industries, alone or in combination with other surface engineering methods.



The effectiveness of shot peening depends both on the intensity and coverage. The intensity dictates the amount of energy transferred from the shot stream into the target parts or components, and is closely related to the applied parameters of the shot peening process (type, size, density, hardness, velocity and impingement angle of shots, pressure, surface coverage, etc.).

Student projects (MSc or BSc) aim to understand the effect of peening parameters as described above on the surface morphology and microstructure as well as associated mechanical properties. The projects require thorough literature study and comprehensive experimental work including various aspects, as for example:

- Surface morphology investigation by 3D optical microscopy,
 - Depth-resolved microstructure characterization of the near-surface region applying microscopic, spectroscopic and diffraction techniques,
 - Microhardness testing as a function of distance to surface,
 - Wear testing of shot peened surfaces,
 - Exposure of shot peened samples to extreme conditions as relevant for industrial applications
- (Note: the content will be agreed according to the project type, **several projects can be formulated**)

The gradient structure in a low carbon steel produced by shot peening in the top 60 μm of deformed layer.



References:

- Zhang X. et al., Hall-Petch and dislocation strengthening in graded nanostructured steel, *Acta Mater.*, 2012, 60, 5933.
Yang R. et al., Effect of shot peening on the residual stress and mechanical behaviour of low-temperature and high-temperature annealed martensitic gear steel, 2017 IOP Conf. Ser.: Mater. Sci. Eng. 219 012046.

TWIP steel for crash absorber for automotive industry

Supervisors: Grethe Winther (grwi@mek.dtu.dk), Kristian Vinter Dahl (kvd@mek.dtu.dk) and Peter Kjeldsteen (Sintex)

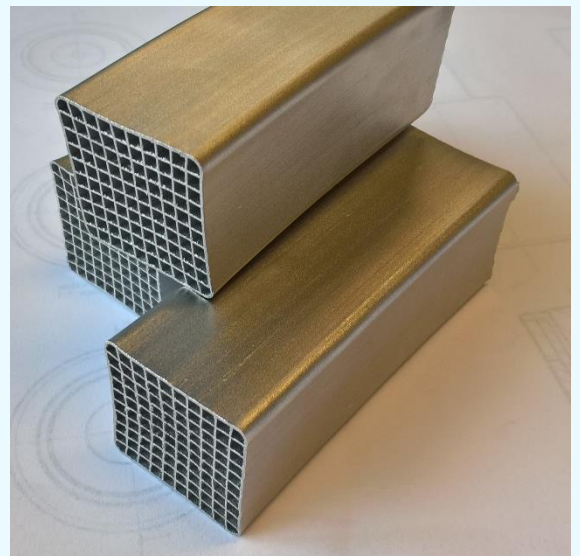
When cars hit sturdy objects at high speed, the first parts of the car to deform should be crash absorbers. Deformation of the crash absorber takes up as much kinetic energy as possible to decelerate the car before the passengers are affected. To optimise the energy take up, crash absorbers are made of a material with high strength and good ductility which is shaped into a honey-comb-structure.

TWIP steels are a new class of high-manganese alloys, which offers an exceptional combination of strength and ductility because the dominating deformation mechanism is twinning instead of dislocation glide as in conventional alloys. Due to the high strength, TWIP steels are, however, difficult to shape mechanically.

The company Sintex currently explores the possibility of producing crash absorbers by sintering of TWIP powders. The first feasibility studies on the production of honey comb structures in sintered TWIP steel are promising.

The project involves thermodynamic modelling of the alloy composition to optimise the sintering parameters. In addition the alloy composition may be optimised to maximise the TWIP effect by thermodynamic calculations and comparison with literature data. The experimental part consists of microstructural characterisation and chemical analysis of sintered parts to evaluate the sintering quality, mechanical testing to evaluate the energy absorption under as realistic conditions as possible and finally microstructural characterisation by e.g. electron microscopy and X-ray diffraction to identify the dominant deformation mechanisms in the alloy.

Parts of the project will be conducted at Sintex.



Quality of laser cladding for repair purposes

Supervisors: Grethe Winther (grwi@mek.dtu.dk) and Jacob Quan Kidmose (Vestas)

Laser cladding is currently used to repair damage in ships but may also find its way into wind turbines. Although the heat affected zone is relatively narrow compared to conventional welding, its properties are crucial for the quality and performance of the repair.

The project is conducted in collaboration with Vestas. The aim is to establish relations between the process parameters of the laser cladding and the quality of the result.

Practical work may include:

- Microstructural characterisation of the initial cladding by microscopy
- Mechanical tests to evaluate the strength of the cladding
- Evaluation of the microstructural changes during mechanical loading



Material optimisation to optimise production of filter house

Supervisors: Grethe Winther (grwi@mek.dtu.dk) and Søren Lassen (Brdr. Jørgensen)

The project is in collaboration with the Danish company Brdr. Jørgensen Components, which specializes in manufacturing components in steel by advanced cold forming techniques. This project concerns optimization of the manufacturing of a specific product, a filter house, which is already in production. The product in service must withstand a high pulsing pressure. The steel in the final product must therefore possess high mechanical strength to resist explosion but also high ductility to resist cyclic fatigue. This combination is not easy to obtain. The product is made of low-carbon steel. The current processing consists of three steps: cup pressing, thermal treatment and a final deformation step. The aim of the project is to evaluate if the production time can be shortened by lowering the time for heat treatments or potentially elimination of the last deformation step. The project involves adjustment of the thermal treatment and/or change of the steel grade.

The project consists of

- Investigation of the microstructure in the product before and after thermal treatment by optical microscopy
- Design of an experimental series of heat treatments based on literature data (incl. CCT and TTT diagrams)
- Evaluation of the mechanical properties of the heat-treated samples. Initial hardness measurements serve as the basis for selection of samples for tensile testing to evaluate the ductility.

BRDR. JØRGENSEN COMPONENTS



Filter house at various steps in the production

Nanoindentation to determine thermo-chemically induced changes in mechanical properties at the grain scale

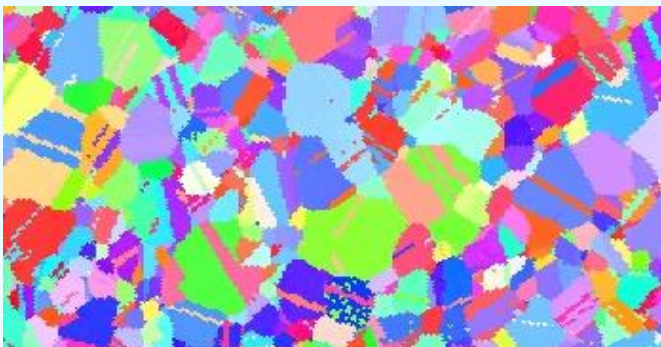
Supervisors: Grethe Winther (grwi@mek.dtu.dk), Thomas L. Christiansen (tch@mek.dtu.dk), Marcel A.J. Somers (somers@mek.dtu.dk)

By thermo-chemical treatments nitrogen can be dissolved in the surface of e.g. austenitic stainless steel to improve the wear resistance. The project aims at detailed investigation of the mechanical properties of individual grains to investigate reports on the reversal of the elastic anisotropy for thermo-chemically treated steel surfaces.

The elastic modulus of austenitic stainless steel is highly anisotropic and depends on the crystallographic orientation of the surface. Upon thermo-chemical nitriding, a reversal of the crystallographic dependence has been observed. The nature and origin of this reversal is yet to be fully elucidated, and also the role of compressive stresses in the surface induced by the nitriding is unknown.

The elastic modulus of individual grains can be probed by nanoindentation. Nanoindentation differs from standard hardness tests by monitoring the full load-unload curve, which allows extraction of the elastic modulus. It is a fairly new technique, which also allows for small indents.

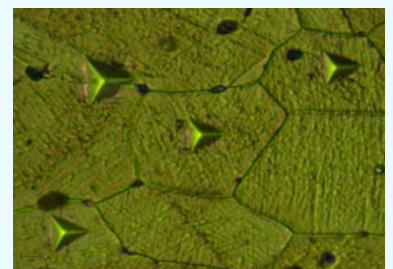
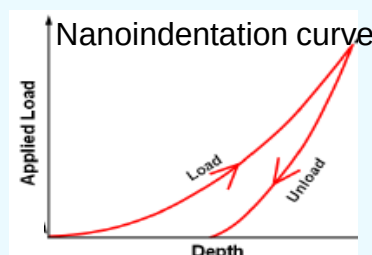
The project combines many experimental techniques: EBSD to measure crystallographic orientations, nanoindentation and sample preparation by nitriding and in the metallurgical laboratory.



EBSD map of grains with differently oriented crystal lattices as designated by the colours



Nanoindentation equipment



Indents in individual grains

Forming limits after strain path changes of ferritic and austenitic steels

Supervisor: Grethe Winther (grwi@mek.dtu.dk)

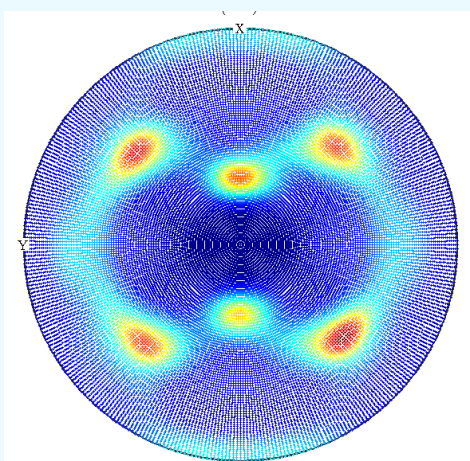
Background:

The forming limit, i.e. the strain applied until fracture, of metals depends on the strain path (e.g. tension or rolling) and also on the deformation history of the metal. Changes in the strain path (e.g. from rolling to tension) typically induce early fracture in the second deformation step, due to the evolution of microstructure in the metal.

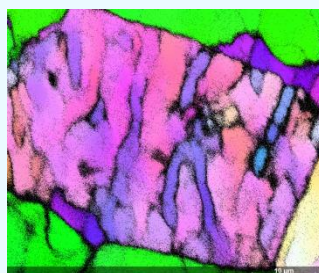
The project:

The forming limits of an austenitic stainless steel (316) and ferritic steel (DC04) are to be determined by mechanical testing covering several types of strain path changes. The experimentally determined forming limit should be correlated with the microstructural evolution in the metal measured by X-rays and electron microscopy (electron back-scatter diffraction). Modelling activities, including crystal plasticity calculations, may be part of the project.

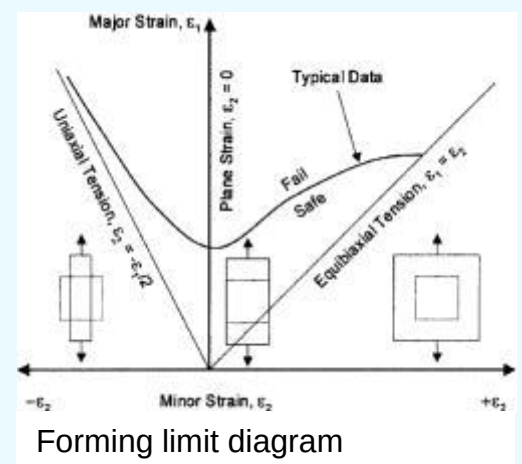
The project can be adapted according to the study type (diploma, BSc, MSc) and the number of students by the choice of samples, number of strain paths and extent of modelling activities.



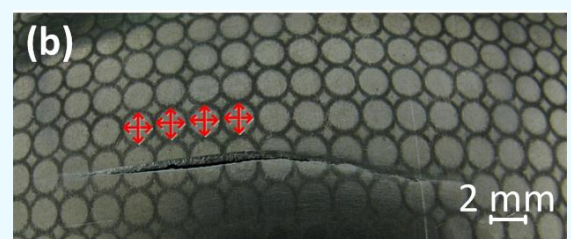
Distribution of crystallographic grain orientations measured by X-rays or EBSD



Deformation-induced microstructure within an individual grain



Forming limit diagram

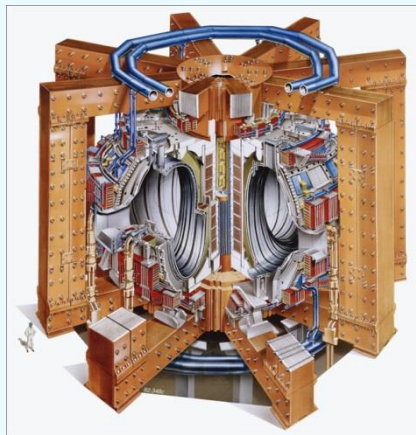


Fracture in metal at the forming limit

Improved materials for Fusion Reactors

Supervisor: Wolfgang Pantleon (pawo@dtu.dk)

One of the key issues in building fusion reactors is the selection and development of materials being able to withstand high temperatures as well as high mechanical and radiation load which alter the microstructures of the materials during service. The suggested projects will focus on the microstructural property relationships of materials relevant for fusion reactors.



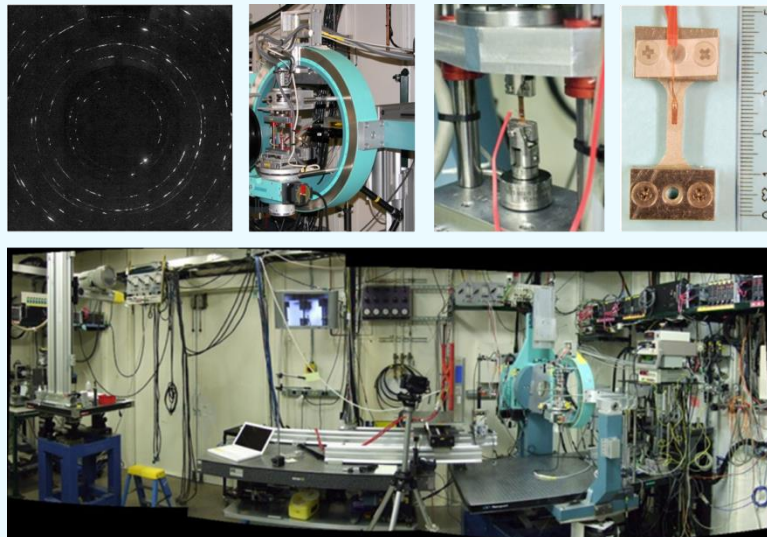
Tungsten and tungsten alloys are considered as preferred candidate for the plasma facing first wall material of fusion reactors. In service, the materials have to withstand high temperature (and radiation loads) altering their microstructure. The replacement of the instable microstructure leads to degradation of the mechanical properties as a loss in strength. The aim of the project is to investigate the thermal stability of differently processed tungsten with focus on the mechanisms (recovery and recrystallization) relevant for property degradation. Additionally, the mechanical properties of the as-processed material as well as after heat treatment will be analyzed.

Monitoring fatigue in fcc metals by x-rays

Supervisor: Wolfgang Pantleon (pawo@dtu.dk)

During plastic deformation of metals, the defects carrying plastic deformation are stored in dislocation walls separating dislocation-free subgrains. These emerging structures require applying higher and higher loads for continuing deformation (work-hardening) and, eventually, cause failure. A peculiar type of deformation occurs during fatigue when the sense of the deformation is reversed repeatedly. Even small plastic strains applied repeatedly e.g. in tension compression cycles cause formation of ordered dislocation structures which after a large number of cycles eventually lead to failure.

The evolution of dislocation structures during cyclic deformation can be monitored *in-situ* by X-ray diffraction. High-energy x-rays available at synchrotron sources provide bulk penetration and the opportunity of *in-situ* monitoring microstructural evolution under realistic conditions. Individual subgrains can be identified in high resolution reciprocal space maps from their characteristic high-intensity peaks and followed during repeated cycles.



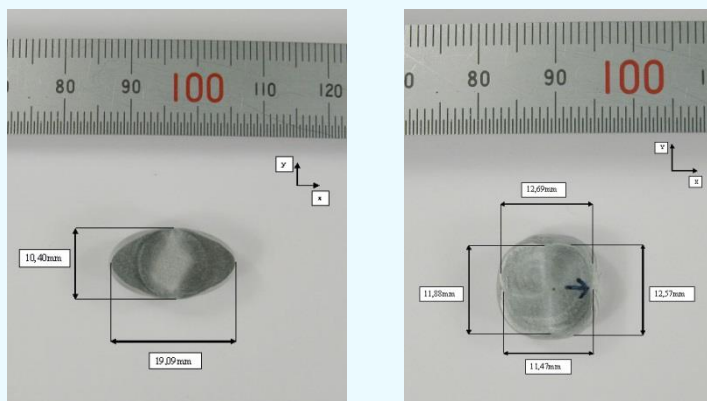
Aim of the project is to analyze and quantify the microstructural changes occurring during fatigue of fcc metals. Preliminary data on copper have been taken already during 80.000 loading unloading cycles. Revealing the microstructure evolution by tracing individual subgrains during cyclic loading from the maps will shed light on their formation mechanism and further the insight in the initiation of failure.

Intrinsic versus enforced anisotropy in plastic deformation of single crystals

Supervisor: Wolfgang Pantleon (pawo@dtu.dk)

Plastic deformation of polycrystals occurs by activation of multiple slip on five or more crystallographic slip systems in each grain. In this manner each grain can follow the imposed shape change.

In single crystals and very coarse grained materials, the individual crystals are not restricted in their shape change in a similar manner. In this way, the crystallites are able to deform by activating a lower number of slip systems. This leads to curious consequences as deviations from the original shape. For example, round cylindrical specimens of aluminum single crystals developed into elliptical cylinders or even more complicated geometries:



The development of shapes with lower symmetries is a result of a very specific selection of slip systems. The specific selection is an intrinsic feature not caused by boundary conditions. It is also reflected in the deformation structure as revealed by electron backscatter diffraction. Establishing of an intrinsic pattern of activated slip systems in individual grains has also consequences for the behavior of polycrystals and the understanding of their texture formation.

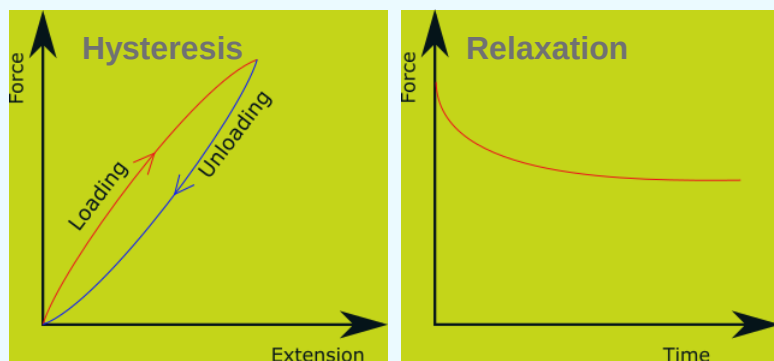
Aim of the project is to study the interplay of the intrinsic activation pattern with opposing boundary conditions. For that it is intended to build a setup allowing the performance of plain strain compression tests and performing such tests with single crystals of particular orientation.

Hysteresis and relaxation of springs in auto injectors

Supervisors: Wolfgang Pantleon (pawo@dtu.dk)

Gert Klarskov Petersen (Medicom Innovation Partner a/s)

Springs are usually considered as perfectly linear elastic with the force strictly proportional to extension or compression. The limitations of this simplification become of importance when the performance of the spring is linked to performance of a medical device.



Medicom Innovation Partner A/S develops auto injectors for the medical industry, used to automatically inject a drug. While electromechanical devices store the energy for injection batteries, other designs are purely mechanical with the energy stored in springs. When the delivery of a potentially lifesaving drug is dependent on the energy stored in a spring, it is quite important that the spring will deliver the expected energy when required. Hysteresis in the loading behavior of the spring and stress relaxation during storage must be considered and understood to ensure reliable performance.

The project aims towards quantification of the observed phenomena from mechanical testing and tracing their microstructural reasons. In this manner, it is hoped to establish an understanding and model description of their dependence on relevant parameters, e.g. selected material, applied stress level and storage temperature.

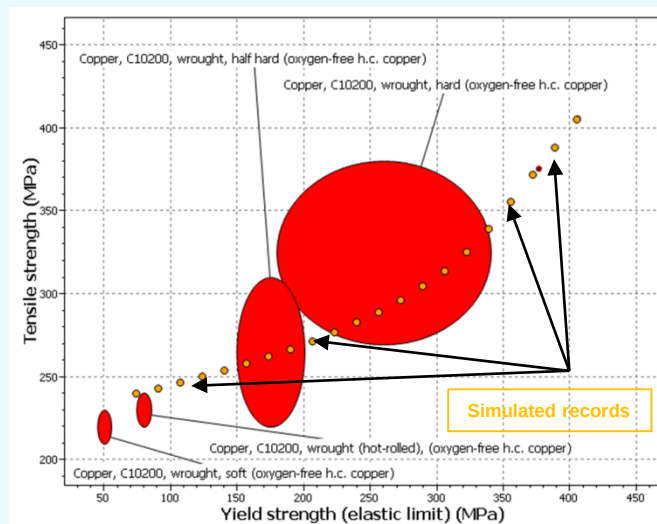
Existing procedures to eliminate set and relaxation of springs should be evaluated and possible limitations identified. By applying higher stress levels or temperatures, relaxation can be accelerated enabling simulation of aged devices and studying the different factors. Medicom Innovation Partner A/S will supply springs that have been stored in compressed configuration and similar unloaded springs for an evaluation of the microstructural changes.

Mapping the microstructural path

Supervisor: Wolfgang Pantleon (pawo@dtu.dk)

Materials selection for structural applications can be conveniently rationalized by means of material property maps reflecting mechanical, e.g. yield and tensile strength. Such maps can be constructed using the Cambridge Engineering Selector. For any given material, specific properties may vary within a certain range, but neither the physical origin of the variation, nor the influence of processing of a material on its properties is revealed. In this manner, insight in the enormous potential for tailoring properties is hindered.

In order to overcome this limitation, a composite-based approach has been developed to simulate the property changes due to thermo-mechanical treatment. As a first application, annealing of deformed metals is considered. During annealing, the hard material representing the deformed state is replaced by softer material representing the recrystallized state. Material resulting from any chosen annealing treatment leading to partial recrystallization of the metal can be simulated using the hybrid synthesizer as composite of material in the two states with respective properties. This approach cannot only be used to simulate the yield strength, under appropriate assumption, ultimate tensile strength and maximum uniform elongation of the partially recrystallized metal can be defined as well.



Such a modeling of microstructural changes in terms of a composite will be applied to other thermo-mechanical treatments leading to precipitation hardening, solid solution strengthening or grain refinement. Aim is to illustrate changes in the mechanical properties due to the microstructural evolution and trace their path in materials property maps.

Grain refinement by combined martensitic transformation and plastic deformation in steels

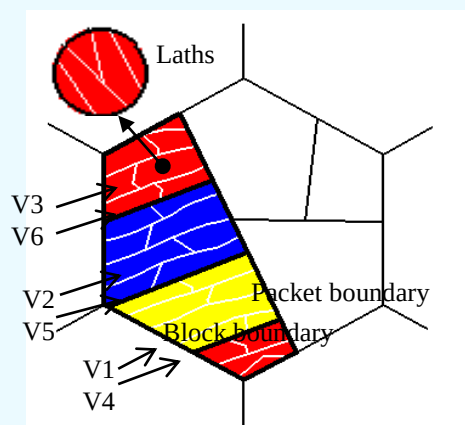
Supervisors: Xiaoxu Huang (xihu@mek.dtu.dk);

Chuanshi Hong (chho@mek.dtu.dk)

Project description

Grain refinement is an important microstructural design approach for strengthening of metals. Martensitic transformation is an efficient grain refinement process, which introduces high density of high angle boundaries, low angle boundaries and dislocations. Martensite in steels shows a variety of morphologies, i.e. lath, butterfly, lenticular and thin plate. Among them, lath martensite has obviously industrial importance because it appears in quenched commercial steels, such as plain low-carbon steels, low-carbon and low-alloy steels, maraging steels and interstitial free (IF) steels. In the martensitic structure of low carbon (below 0.4 wt % C) steels, an original coarse austenite grain is divided at decreasing scale into packets, blocks, sub-blocks and laths, as schematically shown in the figure below. The spacings between lath boundaries can be as fine as 100 nm or less. Plastic deformation (PD) is another efficient grain refinement process, which is achieved by introducing dislocation boundaries in the original grains. Combination of martensitic transformation and plastic deformation of steels is expected to further enhance the grain refinement and as a result to increase the strength of the steels.

The objective of this project is to investigate the effectiveness of grain refinement by the combination of martensitic transformation and plastic deformation in low carbon steels. This requires detailed microstructural characterization of martensite in the as-transformed state and after plastic deformation for example by cold rolling to different strains. In this project particular focus will be on characterization of misorientation angles across various of kinds of boundaries in the undeformed and deformed martensite to understand the structural stability of martensite against plastic strain and the structural refinement effectiveness. Involved techniques will be transmission electron microscopy and SEM (scanning electron microscopy) based electron backscatter diffraction.



Corrosion of materials in oil and gas applications

Supervisor: Rajan Ambat, DTU MEK

Co-supervisor: Magdalena Rogowska, DHRTC

Background:

Large number of materials are used in oil and gas applications. These materials should withstand highly corrosive conditions, which are observed in the topside and downhole conditions. The environment can be extremely aggressive, i.e. high pressures and temperatures, with CO₂ and/or H₂S and vigorous flow of fluids. Corrosion of materials is undesired as it risks the integrity of the pipes. Consequently, understanding of corrosion behaviour of various materials under extreme conditions with or without static or cyclic stresses is important.

Project aim:

A number of master level projects can be defined in this area based on the expertise of the student focusing on various aspects related corrosion and corrosion prevention. Key aspects are understanding the corrosion mechanisms of materials, corrosion scale formation, and the impact of chemistry on their corrosion behavior using lab scale experiments and simulated test rigs.



Internal corrosion in a crude oil pipeline

Practical experiences gained:

- Material use in oil and gas, and corrosion requirements
- Corrosion testing in general and specific to oil and gas conditions
- Experimental methods used for corrosion investigation and material analysis

The project is in collaboration with Danish Hydrocarbon Research and Technology Centre (DHRTC)

A number of Master/Bachelor project themes are available in this area in collaboration with DHRTC. Exact nature of the project and theme will be formulated in discussion with the interested students. Interested candidates, please contact:

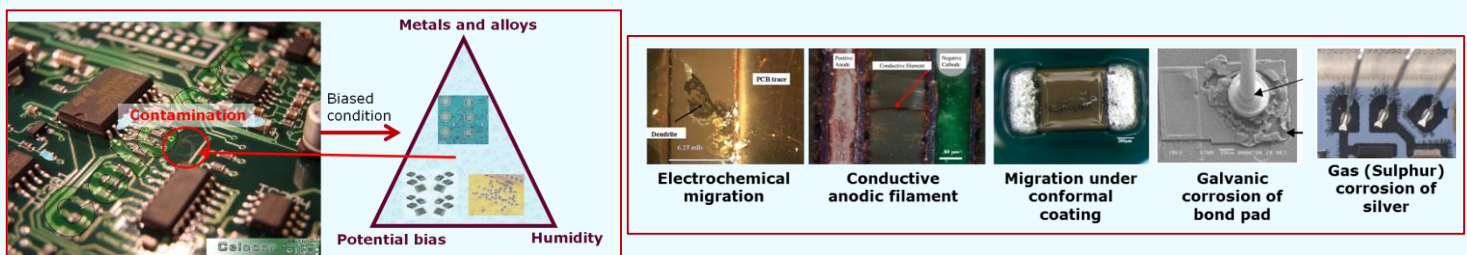
Rajan Ambat, ram@mek.dtu.dk
Magdalena Rogowska, mrog@dtu.dk

Climatic reliability of electronic devices

Supervisors: Rajan Ambat and Morten S Jellesen

Description

Climatic reliability of electronic devices has become an important issue due to the use of multimaterial combinations, potential bias, miniaturized devices, production practices, and wide spread use. At DTU-MEK, the "Centre for electronic corrosion (CELCORR, www.celcorr.com)" investigates various aspects of electronic reliability issues in collaboration with Vestas Windsystems, Danfoss, Grundfos, Velux, Widex, Bosch, etc. and many other electronic industries.



Schematic showing factors causing electronic corrosion and failure modes

A number of Master/Bachelor project themes are available in this area in collaboration with the industries mentioned above. Presently many master students are carrying out their project investigating various issues of climatic reliability in collaboration with above mentioned industries. Broadly the subject for the projects belongs to:

- Climatic reliability issues of electronics in harsh environments
- Semi-empirical modelling of humidity inside electronic enclosures
- Climatic reliability issues of automotive electronics
- Corrosion prevention of electronic devices using conformal coatings
- Climatic Reliability modelling of circuit board design for early prediction

Exact nature of the project and theme will be formulated in discussion with the students who are interested. Interested candidates, please contact:

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Morten S Jellesen, msj@mek.dtu.dk
Vadimas Verdingovas, vaver@mek.dtu.dk

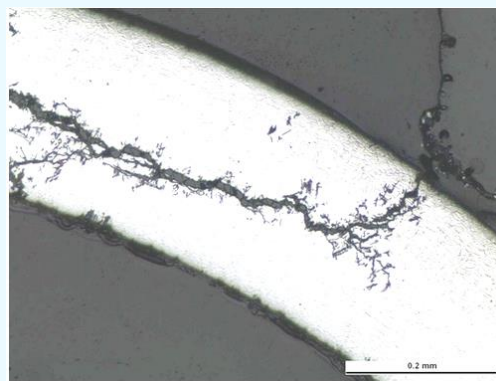
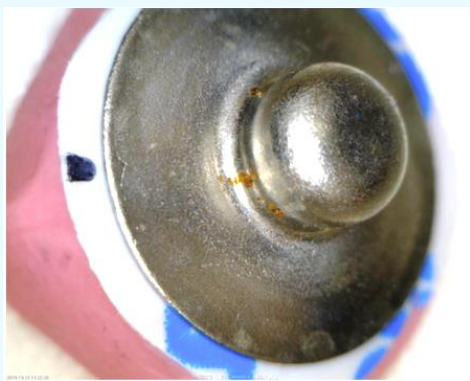
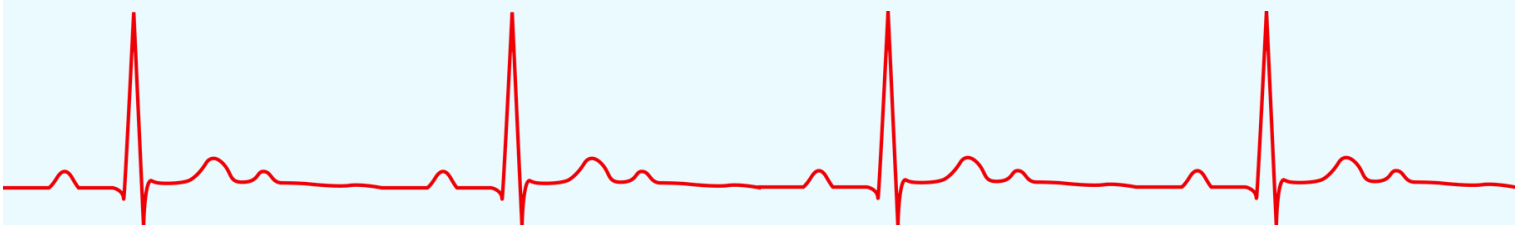
Material characterization of ECG electrodes

Supervisors: Morten S. Jellesen, Rajan Ambat & Rune J. Christiansen

Electrocardiography (ECG) is the recording of electrical activity generated by the heart. The signal is obtained by a series of ECG electrodes placed on the chest of the patient, which is post-processed by sensitive amplifiers and signal filtering. ECG is without comparison the most used method for monitoring and diagnosing patients with cardiac problems in modern medicine. Thus, there is an increasing demand for cheap and reliable ECG, however, keeping the production price low requires the use of new and cheaper materials compromising the long-term reliability of the ECG electrode.

Stainless steel is used as the conducting part of the ECG electrode and is formed by a deep drawing process. Under normal conditions stainless steel possesses good corrosion properties, but cold deformation during the manufacturing process makes it susceptible to corrosion attacks.

The aim of the project is to study and characterize the material properties responsible for corrosion and failure of the ECG electrodes. Two different materials are considered for mechanical and electrochemical testing. Samples from an industrial partner will be available.



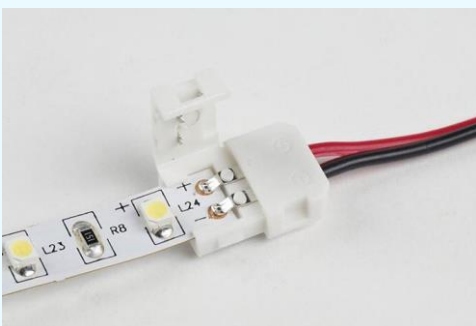
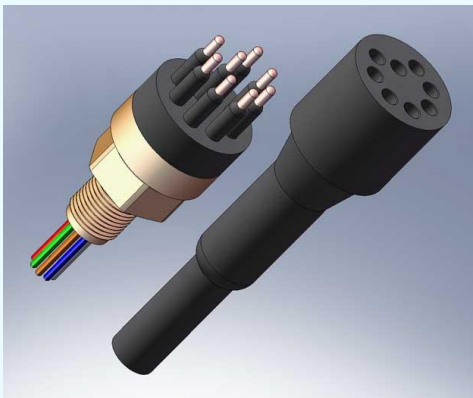
ECG electrode showing local corrosion and stress corrosion cracking after exposure to chloride environment

Materials and surfaces for electrical connectors

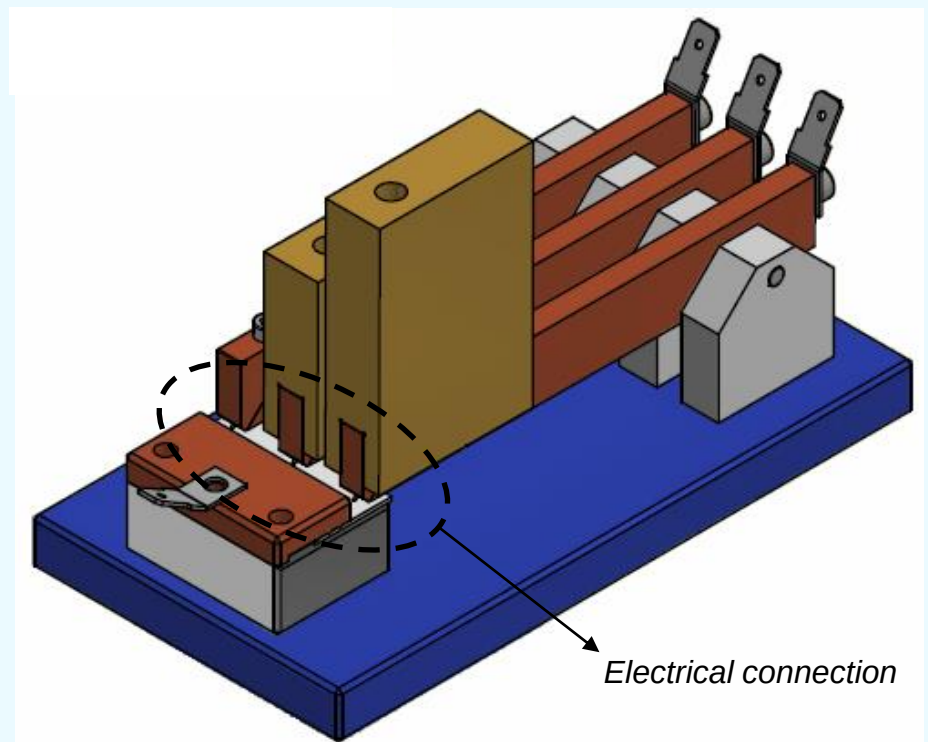
Supervisors: Morten S. Jellesen & Rajan Ambat

The electrical conductivity of contact materials can be largely reduced by corrosion and in order to avoid failure and select optimal materials for electrical connections a deep understanding of the reason behind the failure is needed. One phenomenon that leads to increasing contact resistance is fretting corrosion. Fretting corrosion is the degradation mechanism of surface material, which causes increasing contact resistance. Fretting corrosion occurs when there is a relative movement between electrical contacts with surfaces of ignoble metal. Avoiding fretting corrosion is therefore extremely challenging in electronic devices with pluggable electrical connections. Gold is one of the most commonly used noble plating materials for high performance electrical contacts because of its high corrosion resistance and its good and stable electrical behavior, but it is expensive and there is always a need for minimizing the gold layer thickness. In some cases it is not possible to use gold and other materials as silver tin, nickel, or palladium are being used.

In this project we aim to investigate the effect of contact pressure, temperature and humidity on a silver aluminium electrical coupling. Also different plating thicknesses and materials can be investigated. A new build test setup will be used to online measure the contact resistance as a function of the surrounding environmental parameters. In addition detailed analysis of failed parts are to be carried out.



Connectors are widely used, also in humid environments.



Setup for investigating electrical connections under various humidity and temperature conditions.

Surface modification of aluminium alloys

Rajan Ambat, Morten Jellesen, V.C. Gudla

Description

Demand for light weight aluminium alloys are increasing due to the possibility of translating light weight into higher efficiency, fuel saving in transportation, and for environmental protection. However, efficient use of aluminium alloys for various applications requires high performance surfaces with various functionalities. These include ability to withstand corrosive environmental conditions, optical quality surfaces, wear resistant surfaces, anti-bacterial surfaces to name a few. Materials and Surface Engineering Division works on a number projects in this areas in collaboration with industries such as Sapa, Bang and Olufsen, Velux, research institutions namely DTI, TU Delft, VUB Brussels, and many other partners. The aim of the work is to produce aluminium surfaces with more functionalities, which could replace a number heavy structural materials used in various technological applications.



A kilo of aluminium, used as a substitute for heavier metals in car industry, reduces gas consumption by 8.5 liters and produces 20 kg less CO₂ emissions

A number of Master/Bachelor project themes are available in this area in collaboration with industries. Broadly the topics for project belongs to the following subjects:

- Optical quality surfaces on aluminium alloys (B&O)
- Microstructure and corrosion of aluminium alloys and painted profiles (Velux, Denmark)
- Microstructure and corrosion of heat exchanger components (SAPA, Denmark, Aluventa, Denmark)
- Surface modification of aluminium using steam (DTI, Denmark)

Exact nature of the project and theme will be formulated in discussion with the students who are interested. Interested candidates, please contact:

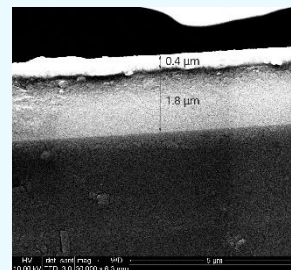
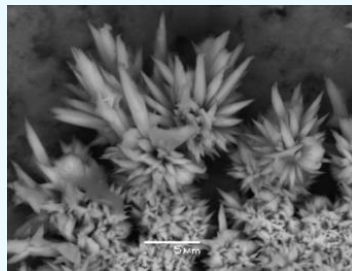
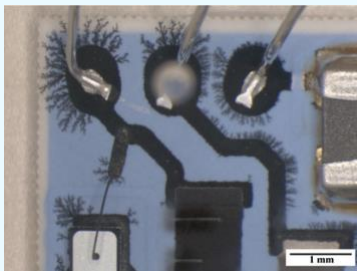
Rajan Ambt, ram@mek.dtu.dk or
Morten Jellsen, msj@mek.dtu.dk or
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Sulphur corrosion of silver

Supervisors: Morten S Jellesen, Vadimas Verdingovas, Rajan Ambat,

Background:

Environmental conditions such as humidity, temperature, contamination and gases affect the reliability and lifetime of electronic products. Sulphur polluted environments include regions near volcanic activity, pig farms, rubber manufacturing plants, oil refineries, coal-generation power plants, paper and pulp industry. Failure analysis of electronic products exposed to an environment containing sulphur gas typically show uniform corrosion and/or migration of silver and copper. The reaction depends on humidity, temperature as well as the existence of other contaminants – e.g. flux residue. The reaction kinetics and dependency of these parameters are very useful when trying to predict lifetime of electronics used under these conditions.



Project aim:

To investigate silver sulfide dependency of humidity, temperature, gas concentration and to look into the effects of flux residues on silver sulfide formation.

Practical experiences gained:

- Electronic corrosion; especially Sulphur corrosion related failure mechanisms and countermeasures
- Test methods employing use of climatic/gas chambers relevant to electronic corrosion testing
- Use electrochemical methods and quartz crystal microbalance to characterize silver sulfide formation.
- Sample characterization using scanning electron microscope (SEM) and X-ray Diffraction (XRD)

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Investigation of martensite formation in Fe-29%Ni model alloy

Matteo Villa (matv@mek.dtu.dk), Marcel A.J. Somers (somers@mek.dtu.dk)

Description

Martensitic transformations are among the most studied topics in materials science.

In the case of steel, martensite formation is fundamental for hardening and is obtained during cooling austenite from high temperature to, or beyond, room temperature.

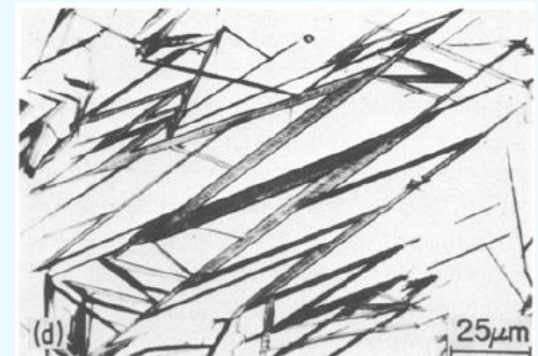
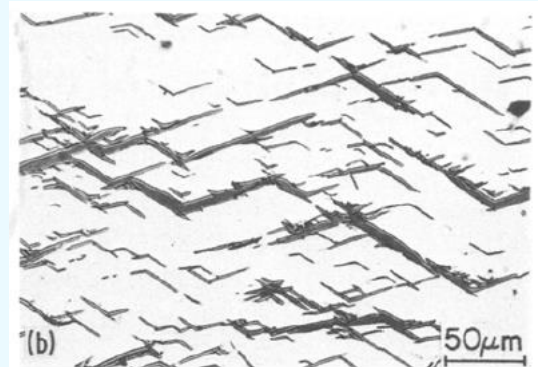
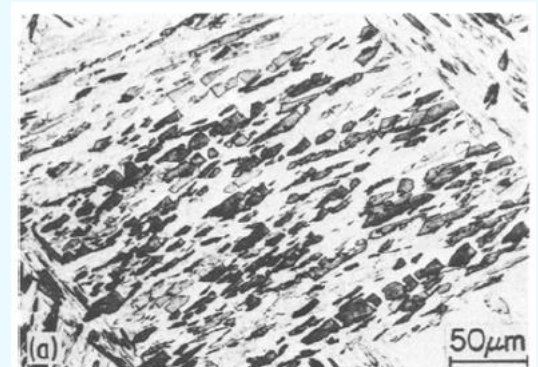
Fe-Ni alloys have historically been taken as reference for the investigation of martensite structures.

At 29% Ni, the morphology of the martensite shows a transition, from lath type (a) to plate type (d). The transition in morphology is accompanied with a transition in kinetics of the transformation.

The connection between kinetics of transformation and martensite aspect is not clarified in the literature. Its investigation is part of the scope of the fundamental research activity in our group and of this project.

Practical work

- Investigation of the material “as received”
- Definition of the thermo-mechanical steps to obtain an homogeneous and fine microstructure
- Implementation of a method to reveal the formation of martensite
- Investigation of martensite transformation temperature and morphology versus austenite grain size
- Characterization of the material with various microscopy techniques



Martensite formation in AISI 301

Matteo Villa (matv@mek.dtu.dk), Marcel A.J. Somers (somers@mek.dtu.dk)

Description

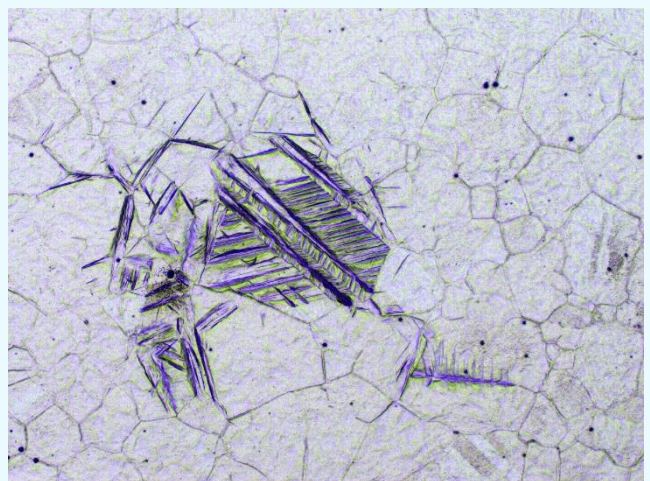
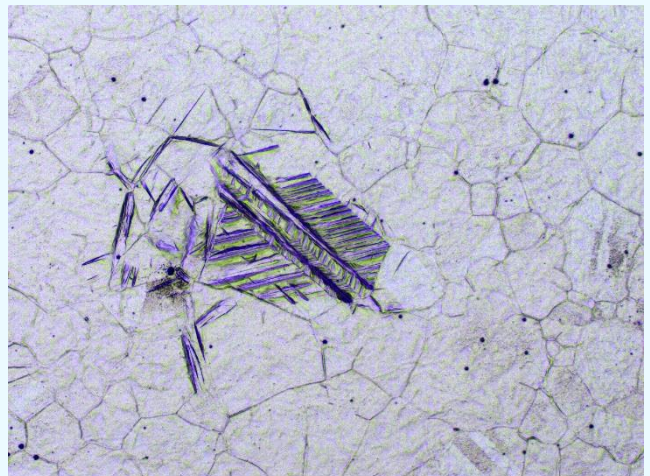
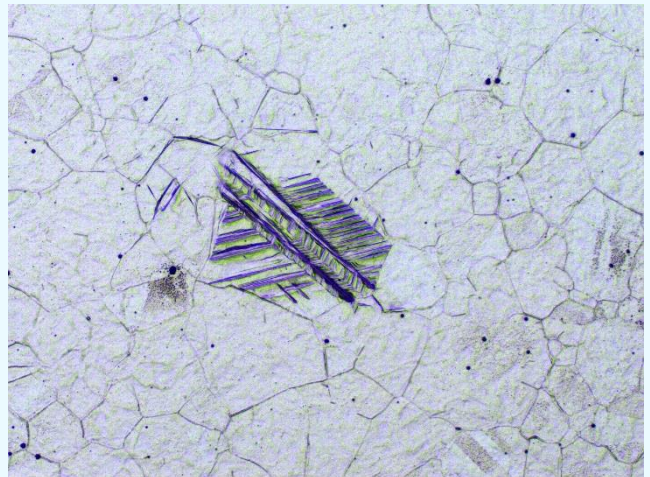
Austenitic stainless steels are materials commonly applied for applications at cryogenic-temperatures. What makes them particularly suitable for cryogenic applications is their f.c.c. crystal structure, which remains ductile also at cryogenic temperatures.

Unfortunately, it is well known that in two of the most used stainless steel grades, AISI 301 and AISI 304, the f.c.c. crystal structure may become unstable at cryogenic temperatures and (partly) transform into martensite. An example of such unstable material was recently obtained by a supplier and is being investigated.

The scope of the present project is to study the stability of AISI 301 stored at -80°C as well as at -196°C versus various treatment parameters. Focus will be placed on the evolution of the martensitic transformation versus storage time for various austenitization conditions.

Practical work

- Heat treatment of AISI 301 in various conditions
- Investigation of the evolution of martensite by X-Ray Diffraction
- Characterization of the material's microstructure (OM, SEM)
- Comparison of the results with literature data



Can advanced Fe-N steels be produced?

Matteo Villa (matv@mek.dtu.dk), Thomas L. Christiansen (tch@mek.dtu.dk),

Marcel A.J. Somers (somers@mek.dtu.dk)

Description

Despite increasing interest in lighter materials, the development of 3 generations of advanced high strength steels, AHSS, confirmed the central role of Fe-C alloys in a large number of applications. The 2nd and 3rd generations are the ones of interest for this project.

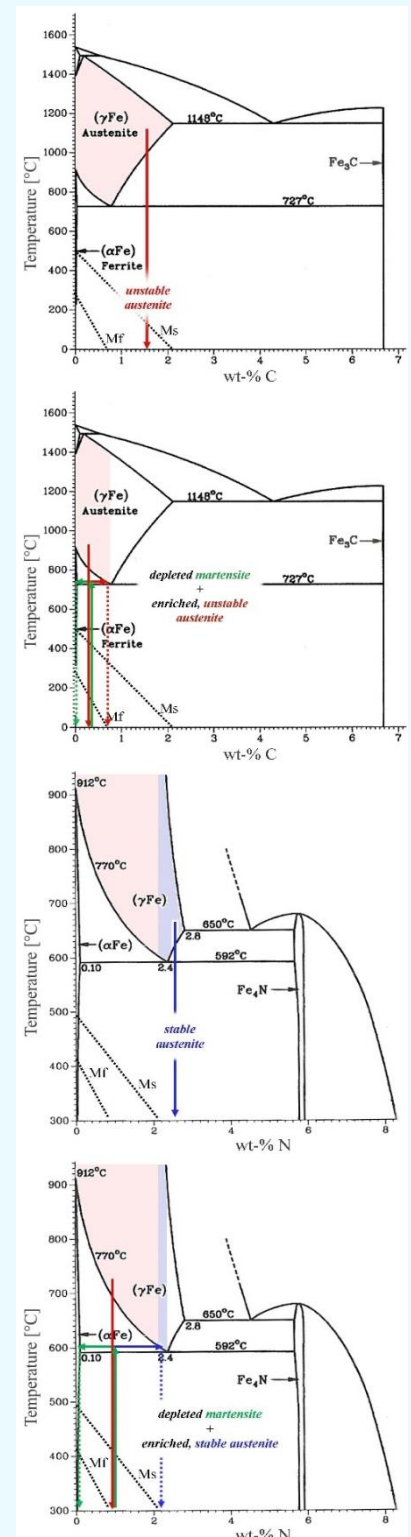
The 2nd generation includes alloys that are fully austenitic after heat treatment, which is key to superior mechanical properties. Unfortunately, Fe-C alloys cannot produce fully austenitic structure at room temperature because the maximum solubility of C in austenite is insufficient. The stabilization is obtained by alloying with elements that are more expensive than C.

The 3rd generation presents a microstructure of fine regions of metastable austenite and C depleted martensite. This is obtained as follow: the austenite is transformed into martensite; the transformation is partially reversed by re-warming the material to a temperature where austenite and ferrite co-exist; austenite is enriched in stabilizing elements. In Fe-C alloys, metastable austenite cannot be obtained because the maximum content of C in austenite at temperatures where this phase coexist with ferrite is too low. The steel is then alloyed with Mn or Ni.

The solubility of N in austenite can easily exceed the one needed to obtain stable austenite. The Fe-N system can be considered to obtain both the 2nd and 3rd generations of AHSS with no additional alloying. Unfortunately, introduction of N in steel is challenging. The purpose of this work is to face the challenge and to try obtaining small parts of a 3rd generation type N based AHSS.

Practical work

- Synthesis of Fe-N thin foils with low porosity at various N contents
- Heat treatment of the material to form microstructures characteristics of the 2nd and 3rd generation AHSSs
- Characterization with SEM, X-Ray Diffraction and EBSD



Effect of Cu precipitates on the microstructure of low C martensitic steels

Matteo Villa (matv@mek.dtu.dk); Marcel A.J. Somers (somers@mek.dtu.dk)

Description

Refinement of the microstructure is the most effective way to enhance the mechanical properties of steel. In the case of low C martensitic steel, which is the most commonly used solution for structural applications, the martensite presents so-called lath morphology.

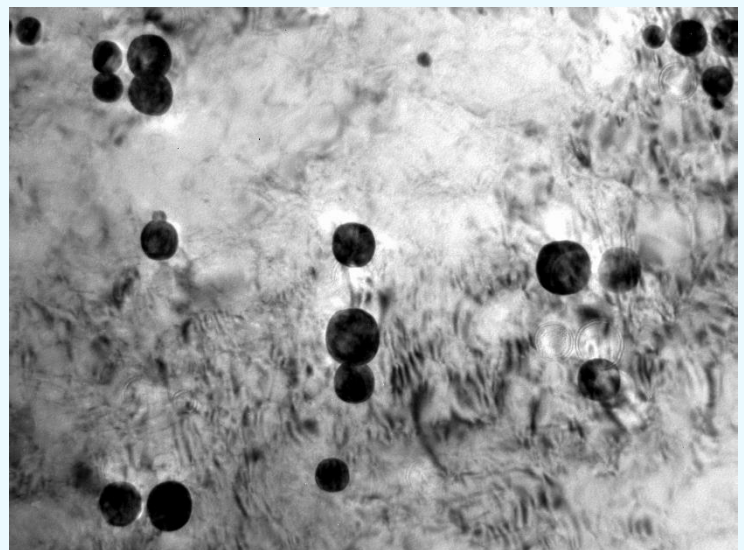
The microstructure of lath martensite is highly hierarchical. Firstly, austenite grains are divided into groups of laths with the same habit plane, so-called packets. Each packet contains several blocks, which are groups of laths with one or two coupled variants of the orientation relationship with the parent austenite, defined as sub-blocks. The individual laths represent the smallest level of sub-division.

It is established that the block size is the effective grain size determining the strength of lath martensite, whereas reducing the packet size is of fundamental importance to depress the ductile-to-brittle transition temperature, making the use of martensitic steels feasible at low temperatures.

In 2010, the most important Japanese research group in the field suggested that finely dispersed particles in the austenite matrix can act to refine the martensitic structure. Not much work in this direction is reported in the literature to confirm / disclaim this hypothesis.

Practical work

- Heat treatment of commercial steel to obtain finely dispersed Cu particles prior to martensite formation;
- Electron microscopy, XRD and hardness measurements to confirm the presence of the particles;
- EBSD investigation and optical microscopy to study the scale of the microstructure.



Studying bainite formation on heating?

Matteo Villa (matv@mek.dtu.dk), Marcel A.J. Somers (somers@mek.dtu.dk)

Description

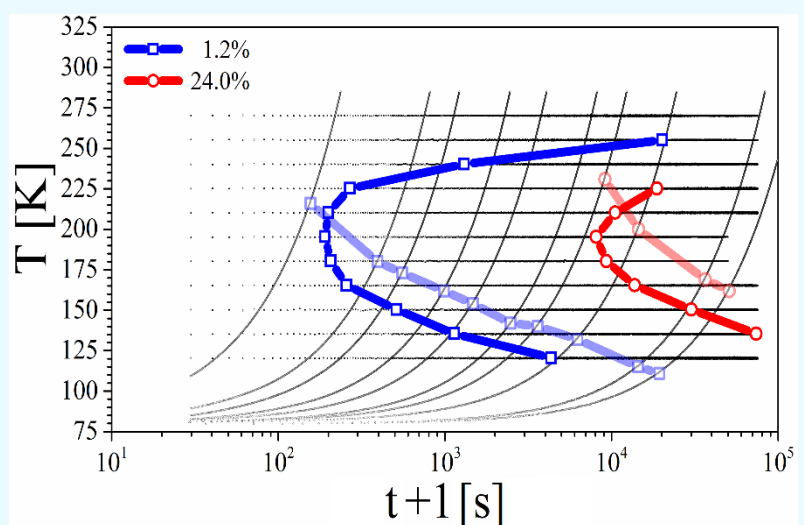
In conventional heat treatment of steel, the material is exposed to high temperature to obtain a soft phase, austenite, which is transformed into various products with strongly varying mechanical properties during subsequent cooling to room temperature. The final properties of the material are determined by the cooling conditions, so that the rate of austenite transformation versus temperature and time is information of major technological interest.

In response to this interest, researchers developed Continuous-Cooling-Transformation, CCT, and Time-Temperature-Transformation, TTT, diagrams, where the various stages of austenite transformation are reported versus temperature and time during continuous, and interrupted, cooling, respectively. Data allows to study the kinetics of transformation, yielding information on the transformation mechanisms, which is of interest to develop predictive tools. Significant results have been obtained. Nevertheless, none of the current transformation theory can reconcile all experimental data obtained during the formation of martensite and bainite that, among all the transformation products, are the ones yielding best performance.

In recent work in our group, various steels were cooled to -196°C and the transformation of austenite into martensite was systematically investigated for the first time during heating. Similarly to the more established investigation approaches, data can be presented versus temperature and time, in this case in the form of a Continuous-Heating-Transformation, CHT, curves. The investigation revealed unique information, never obtained before, like the fact that two types of martensites exists. What about investigation of bainite formation on heating from room temperature?

Practical work

- Preparation of a high C steel fully austenitic at room temperature (carburizing of commercial steels)
- Heat treatment (austenitization and bainite formation on heating)
- Studying of the kinetics by XRD
- Characterization of the material by conventional metallography, hardness measurements, SEM and EBSD



Investigation of reverse austenite formation in stainless steels

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Description

Dual phase stainless steels containing ferrite and austenite, i.e. duplex stainless steels, are extremely resistant to corrosion. Their chemical composition is tailored to obtain a dual phase microstructure in equilibrium conditions at high temperatures. Good mechanical properties are obtained by work hardening. The applicability of such steel grades is in the temperature range -50°C to 300°C .

The aim of the present project is to consider a different approach to the preparation of dual phase microstructures. Commercially available martensitic stainless steels will be austenitized at high temperatures. Thereafter, austenite will be transformed into martensite by cooling. Finally, dual phase microstructure is obtained by partially reverse formation of austenite and simultaneous tempering of the martensite and partition of the chemical elements upon reheating and isothermal holding at intermediate temperatures.

This approach is expected to yield materials with mechanical properties which are superior to currently available duplex stainless steels and with long term applicability at temperatures as high as 400°C . On the other hand, at present nothing can be said on their resistance to corrosion in different environments.

Practical work

- preparation of the material and decarburization in hydrogen;
- austenitization at high temperature and following formation of martensite on cooling to room temperature;
- reverse austenite formation. In situ investigation of reverse austenite formation by Magnetometry (DTU nanotech) will be considered;
- investigation of the microstructure by SEM and XRD. Additional investigation by EBSD (DTU-CEN) will be considered;
- investigation of hardness and corrosion resistance in different environments.

Application of cryo-treatment for surface-hardened martensitic stainless steel

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Description

When commercially available martensitic stainless steels were developed, their chemical composition was tailored to obtain a fully martensitic structure at room temperature. Martensitic structure is necessary for hardening of steel.

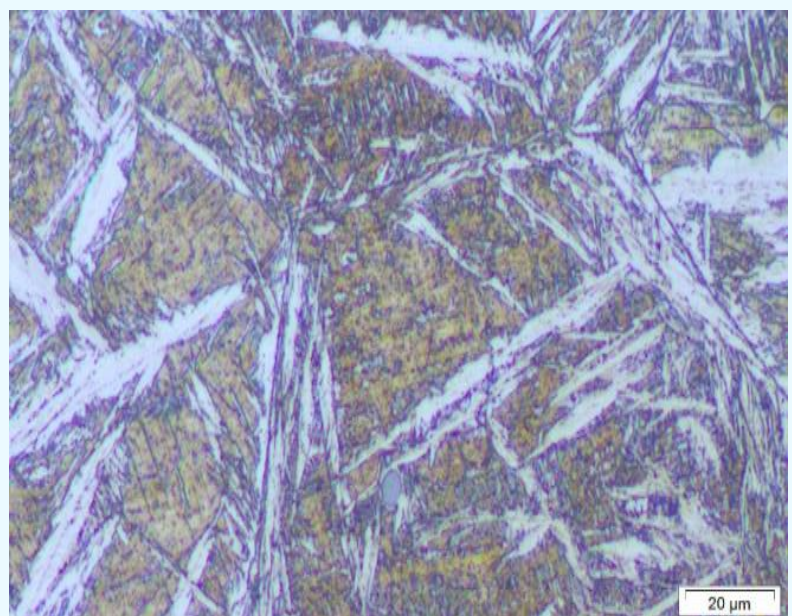
Case carburizing and solution nitriding are techniques to improve the surface properties of steel by the introduction of C and N. Among others they can improve hardness and wear resistance.

Unfortunately, martensite formation occurs at a lower temperature the higher the content of alloying element. Introduction of C and N in the near surface region may result in significant loss of properties by halting the formation of martensite.

The scope of the project is to investigate how cryo-treatments, i.e. heat treatment performed well below zero degrees Celsius, can be applied to overcome the problem.

Practical work

- addition of C/N by thermo-chemical treatment of various commercially available martensitic stainless steel;
- application of different cryogenic treatments to form martensite in the case (ex. storage in dry ice / boiling nitrogen / at -40°C);
- investigation of material microstructure and properties (hardness measurements, XRD, microscopy) also in connection with additional heat treating (i.e. tempering).



Synthesis and characterization of High Entropy Alloys with interstitials

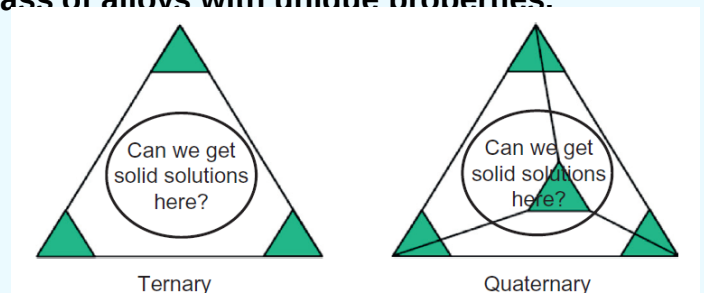
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Description

High Entropy Alloys (HEAs) establish an entirely new class of alloys and derive their stability (and name) from the high configurational entropy by forming a (equi-atomic) solution of at least 5 metallic elements. This class of alloys was first introduced in the 2003. Essentially, the number of possible HEAs is larger than the number of atoms in the universe. Usually, alloys are based on a major metallic element, for example iron-based, aluminium-based or titanium-based alloys, as indicated by the green corners in the figure below. Whereas in ordinary alloys the solid solution often has limited stability and intermetallic phases develop (un)intentionally, in HEAs the solid solution of at least five elements is very stable and the development of intermediate phases is effectively prevented, both thermodynamically and kinetically. As HEAs often are a mixture of different metal atoms with different affinities for elements like carbon, nitrogen and oxygen there is an unexplored potential for making interstitial solid solutions, either to achieve unique properties of strength or to surface engineer HEAs to build in compressive residual stress to enhance their performance with respect to surface-induced cracking and fatigue. It is anticipated that the dissolution of interstitials in the distorted HEA lattice in combination with frustrated diffusion of the metal and the interstitial atoms will lead to a supersaturated solid solution. This alone can lead to an entirely new class of alloys with unique properties.

Practical work possibilities

Under the umbrella of Hes there are many Opportunities for different kinds of projects, From fundamental to applied.



Synthesis of HEAs with one or more components with a high affinity for one or more interstitial elements.

Use thermogravimetry for diffusion of interstitial elements in the HEA and synthesize homogeneous iHEAs or surface engineered functionally graded HEAs.

Characterization of iHEAs and /or functionally graded HEAs with a combination of techniques like LOM, SEM, XRD (including residual stress) and GD OES.

Determination of fundamental parameters as solubility and diffusivity of interstitials in iHEAs.

Development of porous iron and metastable iron phases

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Description

Porosity in iron-based nitrides is a well known by-product during nitriding and nitrocarburizing of steels. The driving force for such porosity is thought to be the thermodynamic meta-stability of iron nitrides with respect to pure iron and pure nitrogen gas. In this sense porosity could be interpreted as the precipitation of nitrogen gas in the solid state. This metastability can be used to synthesize extremely porous iron which could be a precursor for the formation of meta-stable iron-based phases which otherwise are impossible to synthesize as pure phases, as for example transition carbides and nitrides.

Theoretical work

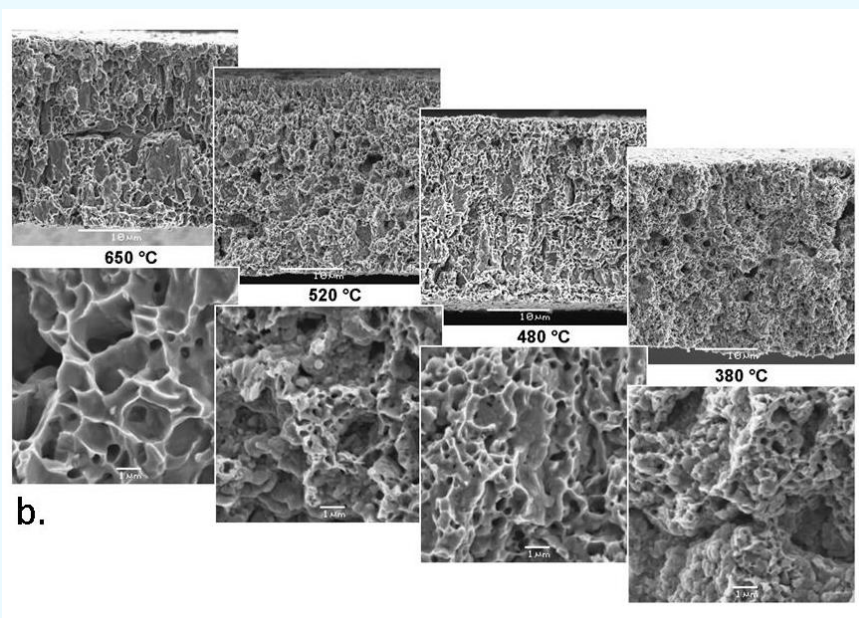
Literature study on porosity and critical evaluation of the various hypotheses put forward to explain this phenomenon. Thermodynamic calculations of the driving force for N_2 development in relation to composition of iron-nitrides.

Practical work

Synthesis of extremely porous iron and metastable iron-based phases.

Kinetic analysis of decomposition of the nitrides with thermogravimetry.

Identification of metastable phases with X-ray diffraction.



Development of porosity in iron-nitrides

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Description

Porosity in iron-based nitrides is a well known by-product during nitriding and nitrocarburizing of steels. The driving force for such porosity has been recognized to be the thermodynamic meta-stability of iron nitrides with respect to pure iron and pure nitrogen gas. In this sense porosity could be interpreted as the precipitation of nitrogen gas in the solid state. So far, the actual scientific evidence for N_2 filled pores is missing; all evidence is circumstantial. The present project focuses on proving that N_2 formation is the origin of porosity. Furthermore the kinetics of the precipitation of N_2 will be investigated.

Theoretical work

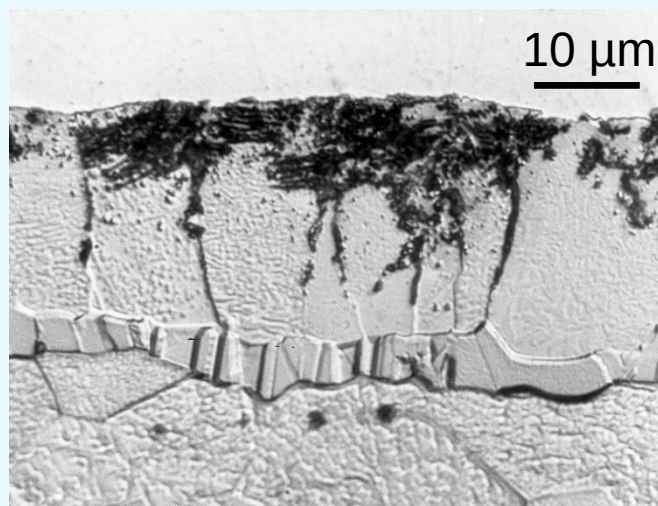
Literature study on porosity and critical evaluation of the various hypotheses put forward to explain this phenomenon. Thermodynamic calculations of the driving force for N_2 development in relation to composition of iron (carbo)nitrides.

Practical work

Synthesis of homogeneous iron nitrides with different nitrogen (and carbon) contents.

Kinetic analysis of decomposition of the (carbo)nitrides with thermogravimetry and differential scanning calorimetry.

Identification of N_2 as precipitates in the solid state with diffraction and or spectroscopic techniques.



Surface hardening of titanium alloys

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Description and motivation

The increasing number of implants, joint replacements and revisions due to adverse body reactions represent an enormous socioeconomic challenge. Up to 20% of the patients undergoing a hip-joint replacement develop peri-implant bone destruction within 10 years after surgery, necessitating revision and replacement surgery. Such implant failure is considered to be strongly correlated with metal release from the implant by corrosion and wear and metal-allergy towards implant components. Longer implant durability and longer lifespan are needed without the risk of rejection of the artificial joint implants. Tailored surface engineering of implants is expected to be an effective means of combating metal release due to corrosion and wear.

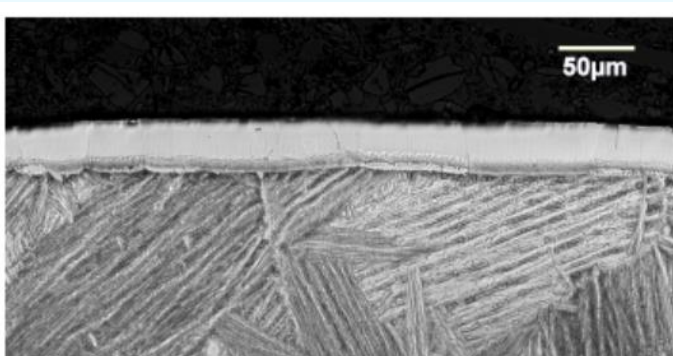
To this end newly developed thermochemical methods for surface hardening of titanium can be applied for improving the performance of such light-weight materials. Very high hardness and thick surface layers can be obtained by these new methods.

Theoretical work

Literature study on existing surface modification technologies used for titanium and titanium alloys.

Practical work

Gaseous thermochemical treatment of titanium and titanium alloys will be carried out various temperatures and in different gas mixtures. The hardened surfaces will be characterized by light optical microscopy, X-ray diffraction, microhardness and scanning electron microscopy. The corrosion resistance of the surface engineered materials will be tested by electrochemical characterization techniques (open circuit potential monitoring and polarization curves). Wear resistance will be investigated using a pin on disc sliding wear setup.



3D printed stainless steel impellers

Supervisors: Thomas L. Christiansen, tch@mek.dtu.dk
Uffe Bihlet, FORCE Technology, udb@force.dk

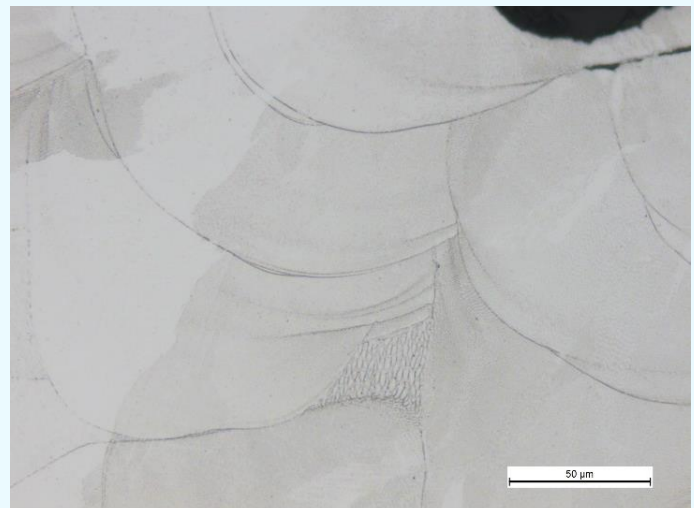
Grundfos has experimented with additive manufacturing (AM) of impellers in stainless steel for water pumps.

The current impeller design is currently either fully cast using lost-wax techniques or assembled of sheet metal, joined by laser welding. These designs, and the production facilities of Grundfos, are optimized for mass production of parts. Additive production of stainless parts enables a versatile small-scale production of certain impeller designs.

The AM equipment currently installed at Grundfos uses the powder bed fusion method, where a mirror-directed laser fuses metal powder particles layer by layer into a finished part. In the present project, a sample impeller has been produced by 3D printing of stainless steel 316L. Some initial corrosion testing has shown that corrosion occurred predominantly from melt pool interfaces. During 3D printing, the laser beams creates a molten pool, fusing powder particles. After solidification, the melt pools are still clearly visible in the microstructure. Another interesting observation in 3D printed microstructures is that grain boundaries overlap the melt pool boundaries.

The aim of the present project is two-fold, and is summarized in two key points below:

- Full characterization of the microstructure, specifically the melt pool lines Involving, but not limited to, optical microscopy, SEM, X-ray diffraction and EBSD.
- Propose and test a post-treatment or printing process adjustment which optimizes the corrosion resistance of the printed parts



Development of irregular white etching cracks in bearings for wind turbines

The project is done in collaboration with Siemens Wind Power A/S

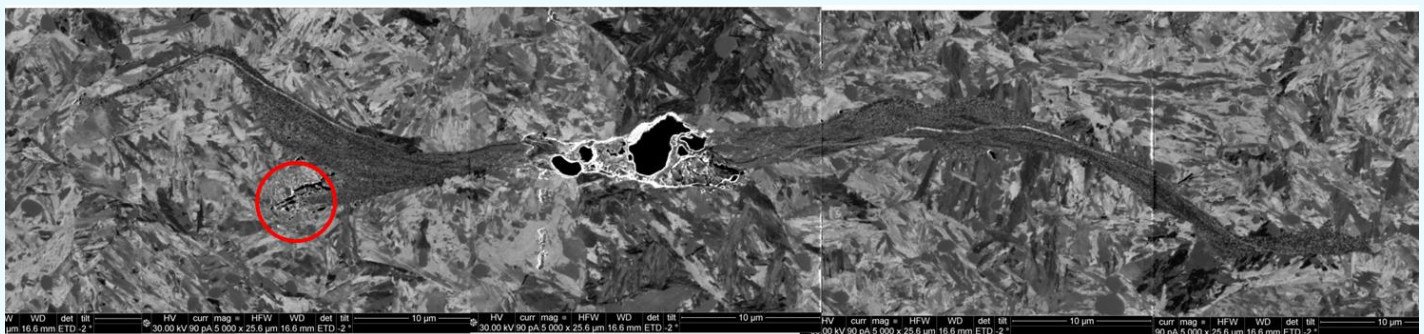
Kristian V. Dahl (kvd@mek.dtu.dk)

One of the major failure manifestations found during metallographic inspections of bearings in wind turbine gears (WTG) is flaking associated with irregular cracks that are always accompanied by white etching areas; hence the name irregular white etching cracks (IrWEC). Such failures have been found to appear long before the predicted life of WTG bearings. There is no common consensus about the root cause for IrWEC formation in WTG bearings, but it is suspected that hydrogen entrance into the bearing steel plays a major role.

Recently IrWEC has been found to form in carburized martensitic bearings containing a high amount of non-martensitic transformation products. It has been hypothesized that the (unfavorable?) heat treatment leading to presence of the non-martensitic regions also has an influence on the kinetics of the IrWEC formation.

In the present project bearing steels containing both non-martensitic transformation products and large IrWEC networks will be investigated using state of the art microscopy techniques to investigate formation mechanism of IrWEC and if possible find a relation to the presence of the non-martensitic regions.

The microscopy techniques to be employed are scanning electron microscopy, ICCI and ECCI (Ion and Electron Channeling Contrast) combined with EBSD as well as transmission electron microscopy on thin foils prepared by lift out done using focused ion beam milling.



ICCI image of a branching "butterfly" in a martensitic bearing steel. A crack bordered by white etching regions has formed from a non-metallic inclusion. Is the branching the start of network crack formation?

Long-term microstructure stability of 12%Cr steels in Danish power plants

Supervisors: John Hald (jhald@mek.dtu.dk)

High temperature components in steam power plants are normally designed for a creep life of 200,000 hours (more than 20 years).

The 12%CrMoV martensitic steel known as X20CrMoV121 has been used for steam pipes in power plants since the mid 1960ies. A number of Danish power plants using this steel are now approaching the creep design life of 200,000 hours, but due to the energy transition there is a desire to maintain the steam pipes in operation for up to 300,000 hours.

This raises fundamental questions about the long-term stability of the microstructure, since long-term creep testing has only been performed up to 200,000 hours.

The project will address these questions by microstructure investigations of steel samples removed from steam pipes in Danish power plants after long-term service. The microstructure degradation of the steels will be investigated by light microscopy and electron microscopy (SEM and TEM) as well as assessment of the chemical compositions of the steels with thermodynamic equilibrium calculations.

The project is carried out in collaboration with the power companies DONG Energy and Vattenfall.



High N martensitic steel for high temperature steam turbines

John Hald (jhald@mek.dtu.dk)

Due to their high creep strength martensitic steels with 9-12%Cr are used for pipes, casings and rotors in steam turbines operating at temperatures up to 600°C in thermal power plants.

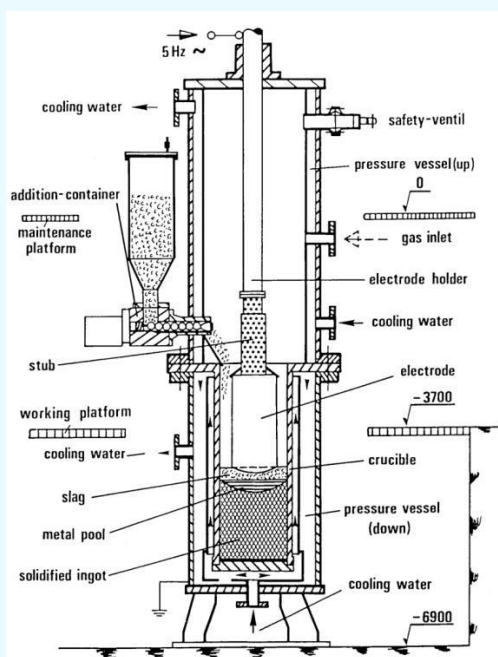
Ongoing research aims at improving the creep and oxidation resistances of such steels in order to increase operating temperatures up to 650°C for better efficiency.

The high creep strength of the 9-12%Cr steels is to large extent achieved by precipitation hardening from a fine dispersion of nano sized nitrides in the martensitic microstructure. However, the amount of strengthening nitrides is limited by the solubility of Nitrogen in the steel melt. For N contents above some 0.08 wt % gas bubbles will form in the melt.

The present project explores the possibilities to increase the amount of nitrides by melting the steels under pressure. This allows N contents up to more than 0.2 wt %, and a similar increase of the amount of Nitrides in the steel.

The project will include investigations of the evolution of microstructure and hardness in high N steels during long-term exposure to high temperatures between 600 and 700°C. Various microscope techniques can be applied including light microscopy and electron microscopy (SEM and TEM). Microstructure modeling of phase equilibria with Thermocalc can be included.

Steel samples will be delivered by Energietechnik Essen, who has equipment for pressurised casting of steels.



Kinetics analysis of the formation and retainment of δ -ferrite in cast low-carbon martensitic stainless steel

The project is in collaboration with Frese metal og stålstøberi A/S

John Hald (jhald@mek.dtu.dk), Niels Skat Tiedje (nsti@mek.dtu.dk),

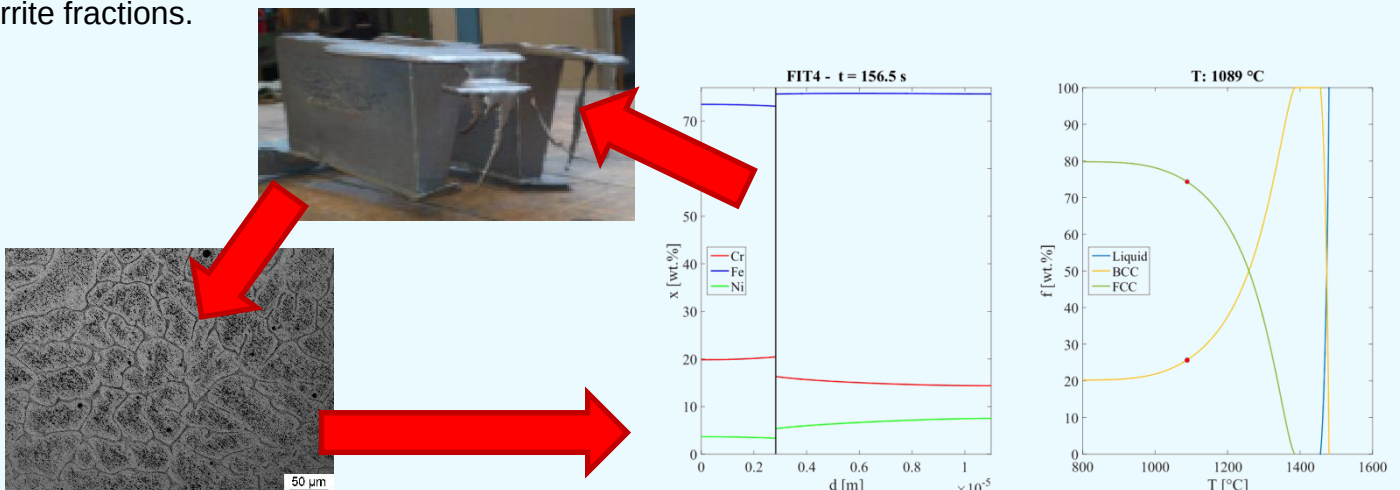
Frank Nießen (frannie@dtu.dk)

Cast low-carbon martensitic stainless steels are a popular choice when mechanical strength combined with good anti-corrosion properties for a reasonable price are demanded, e.g. in oil-pipelines, pumps, ship propellers and water turbines. It is common to retain δ -ferrite upon cooling from high temperature down to room-temperature. δ -ferrite has a detrimental effect on the mechanical properties. Therefore various empirical and analytical models were developed to predict and eventually avoid the retainment of δ -ferrite.

Under thermodynamic equilibrium δ -ferrite should transform to austenite during cooling. The equilibrium conditions can under fast cooling however only be seen as a target point for the occurring phase transformation, the actual phase distribution at room-temperature is determined by the kinetics of the phase transformation during cooling, i.e. to which extent the transformation is able to reach the equilibrium.

Recent investigations have shown that kinetics modelling in DICTRA can be used to model the solidification and cooling of cast low-carbon martensitic stainless steel. The model revealed that the cooling rate and the alloy composition mainly determine the amount of retained δ -ferrite. Proper validation, and eventual adaptation, of the model **to** monitored experimental castings is needed to demonstrate the applicability of the model.

In the present MSc project experimental castings should be made in cooperation with the foundry Frese A/S. The castings should be carried out in different conditions, e.g. cooling rate, alloy composition or geometry. Information from the monitored casting and the as-cast microstructure should be used as input to the kinetics model DICTRA. The predictions from DICTRA should then be validated and, if necessary, the model should be adapted to establish a solid tool for the prediction of δ -ferrite fractions.



Microstructural Evolution in Centrifugally Cast High Temperature Austenitic Alloys

The project is in collaboration with Haldor Topsøe A/S

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Anette N. Hansson (annh@topsoe.dk)

Centrifugally cast pipes made from high temperature austenitic alloys are widely used in steam reformers in petrochemical industries. The tubes are exposed to high temperatures (up to $\sim 980^\circ\text{C}$) and high pressure (up to ~ 40 bar) over a long period of time (10-15 years), therefore creep and thermal damage can be expected.

The alloys contain high amounts of Ni and Cr in order to ensure austenitic microstructure, high creep and corrosion resistance. Great improvement of the high temperature properties of the alloys has been achieved by small additions of other elements such as Nb, Si, Ti or Zr.

The as-cast alloy contains primary precipitates of Cr carbide that coarsen during the exposure at high temperatures. Furthermore, various types of strengthening precipitates in the as-cast alloy are dissolved and other larger precipitates are formed. This has a negative influence on the mechanical and creep properties of the alloys.

The aim of the project will be to study the evolution in microstructure of the high temperature centrifugally cast austenitic alloys. The study could include:

- Literature survey on the microstructure evolution
- Thermodynamic simulation of stable precipitates
- Microstructure characterization of service exposed material by Light Optical Microscopy (LOM), Hardness measurements, Scanning Electron Microscopy (SEM) and possibly Transmission Electron Microscopy (TEM)

