INDUSTRIAL PILOT PROJECT

X-ray Absorption Spectroscopy for speciation of hazardous metals in ash from waste incineration – Critical knowledge for recycling

THE INDUSTRIAL CHALLENGE

Today, a considerable amount of household and commercial waste that cannot be reused or recycled is incinerated with energy recovery resulting in large streams of fly and bottom ash. Fly ash most often goes to landfills, while bottom ash has excellent properties as ground construction material, such as for building of roads. However, it must be secured that its use poses no environmental risk. For Cu and Zn, the chemical form/speciation determines the ecotoxicity and some chemical forms are therefore regulated. However, no simple methods exist to identify the chemical speciation of the trace metals in the ash and if not known, a worst scenario is applied - preventing the use of bottom ash. Thus, it is crucial to know the chemical speciation of Cu and Zn in the bottom ash to secure a correct classification and an effective use of resources. For fly ash, the chemical speciation is relevant when evaluating the possibilities for extracting trace metals.

Figure. The storage of processed ash at SYSAVs facility at Spillepengen, Malmö

WHY USING A LARGE SCALE FACILITY

The low concentrations and extremely complex ash matrix make it not possible to use traditional lab-based methods for analysing chemical speciation. Synchrotron-based X-ray absorption spectroscopy (XAS) has been identified as one promising method and in the future, the BALDER beamline at MAX IV will provide possibilities for high throughput measurements.

HOW THE WORK WAS DONE

Samples from both bottom and fly ash were collected from five Swedish facilities. After a pre-study at the BALDER beamline using both XANES (X-ray absorption near edge structure) and EXAFS (Extended X-Ray Absorption Fine Structure), the focus was on XANES. The absorption edge of Zn, Cu, and Pb was scanned for in total 16 samples. The project also included building up an open library of reference materials – which is critical for interpreting the XANES results. The probable chemical speciation of Cu, Zn and Pb in ash was determined using Linear Combination Fitting (LCF) of the references to the ash spectra.

Figure. The project team from Fortum Waste Solutions, Sysav and NOAH with research support from RISE, Chalmers Technical University and MAXIV. Also, representatives from Stena and EON joined at MAXIV. Photo: Stena Metall

THE RESULTS AND EXPECTED IMPACT

We could to a large extent explain the XANES spectra using a subset of reference compounds, however, for some ashes additional reference materials are still missing in order to make a full determination of the speciation. A number of questions were addressed such as differences between (i) bottom vs fly ash, (ii) facilities, (iii) before and after storage, and (iv) after processing of the ash. The requirement for a library containing all relevant reference spectra is identified as a limitation of the method when used for analysing a broad range of ashes, but when in place, the method has high potential to be used for determining the speciation of trace metals in ash. The results are summarized in a paper submitted to J. Waste management.

Contacts: Thomas Von Kronhelm – Fortum Waste Solution AB, Thomas.VonKronhelm@fortum.com
Jenny Rissler – RISE Research Institutes of Sweden, jenny.rissler@ri.se

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INDUSTRIAL PILOT PROJECT

In-situ neutron scattering analysis of the early stages of phase separation in duplex stainless steels at elevated temperature

THE INDUSTRIAL CHALLENGE

There is a strong industrial interest in increasing the service temperature and/or prolonging the service life of duplex stainless steels to make use of their excellent strength and corrosion resistance. Low temperature embrittlement of duplex steels, often referred to as 475 °C embrittlement, however, restricts their application at low temperatures. The embrittlement is caused by the immiscibility of the key alloying elements Fe and Cr in the ferrite phase at temperatures below about 550 °C. If the alloy is exposed in the temperature range between ~250 - 550 °C for extended times, the Fe and Cr will demix and form a nanostructure constituting two ferrite phases. This leads to increasing hardness and decreasing toughness with time, and may cause catastrophic failure.

WHY USING A LARGE-SCALE FACILITY?

Most of the prior research has been devoted to long term isothermally aged materials and *ex-situ* metallography using techniques such as transmission electron microscopy and atom probe tomography. However, little effort has been paid to the early stages of demixing. Hence, *in-situ* measurements of the nanostructural evolution during the thermal cycle could provide new information on the early stages of demixing, which is not possible to capture accurately with post mortem analysis. *In-situ* measurements during thermal cycling are possible using small-angle neutron scattering (SANS).

HOW THE WORK WAS PERFORMED

The SANS experiments were carried out at the ISIS Neutron and Muon Source, UK, by representatives from Outokumpu Stainless and KTH in collaboration with Dr. Stephen King at ISIS. The SANS2D beamline were uesd at an incident neutron wavelength of 2-10 Å for analysis of 10x10x1.1 mm samples from two duplex stainless steels (2205 and 2507). The *in-situ* experiments during heat treatment were carried out at three different temperatures for up to six hours. To supplement the in-situ measurements, heat treatments of *ex-situ* specimens were conducted in advance using three temperatures and aging times. *Ex-situ* specimens were also prepared in advance by thermal simulations using a Gleeble instrument in order to simulate different cooling rates due to different steel plate thicknesses. Specimens heat treated according to the *ex-situ* SANS conditions were subjected to property testing using hardness and impact toughness testing.

THE RESULTS AND EXPECTED IMPACT

The early stages of thermal cycling was found to play an important role on the demixing kinetics. For example, the ex situ specimens slowly cooled in the Gleeble showed a pronounced demixing, whereas this was not found in the fast cooled specimen. The difference disappears after 20h of subsequent aging at 475°C, but the initial condition alters the kinetics in the important early stage when embrittlement occurs. The Figure shows an example from the *in-situ* analysis of the 2507 duplex steel at 450 °C up to six hours of aging. The periodic length scale of the concentration fluctuations can be directly evaluated from the peak position as $2\pi/Q$ (Å^{-1}), while the amplitude requires model-dependent fitting.

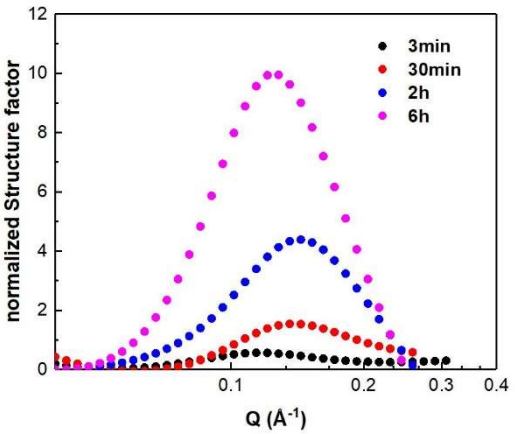

Figure. In-situ SANS data for 2507 during aging at 450 °C. Structure factor vs. Q [Å^{-1}].

The difference in demixing kinetics between alloy 2205 and 2507 were analysed. Finally, an unexpected parameter that can delay demixing was revealed in the in-situ experiments. This is currently explored for commercial protection.

Contacts: James Oliver – Outokumpu Stainless AB, james.oliver@outokumpu.com
Anette Nørgaard Hansson – Haldor Topsoe A/S, annh@topsoe.com
Peter Hedström – KTH Royal Institute of Technology, pheds@kth.se
Sten Wessman – Swerim AB, sten.wessman@swerim.se

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INDUSTRIAL PILOT PROJECT

In-situ experiment to follow kinematics of phase transformation and precipitate formation in duplex stainless steels

THE INDUSTRIAL CHALLENGE

Intermetallic phases and precipitates are commonly identified in duplex stainless steels in heat-affected zones after treatment or welding. Understanding the cause of their formation and following the kinematics of their growth is of great interest for both manufacturers (to optimize the production process) and end-users (to enhance the application and prolong the lifetime).

WHY USING A LARGE-SCALE FACILITY?

In-house techniques such as electron/light microscopy or x-ray diffraction allow quantification of phases or precipitates to a depth of a few micrometres and for the final product only. Synchrotron radiation, on the other hand, enables following the kinetics of phase transformation during relatively fast heating and cooling processes relevant to e.g. welding, several mm into the bulk.

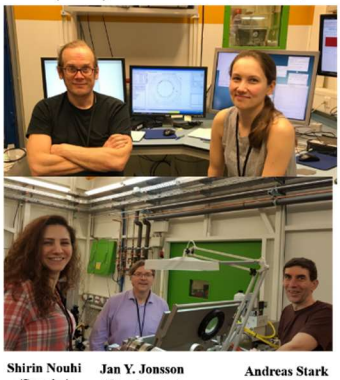
HOW THE WORK WAS DONE

The experiments were conducted at the P07/HEMS beamline of Petra III (Hamburg) by expertise in diffraction techniques and materials experts from Outokumpu (Sweden and Germany). In-situ Wide-Angle X-ray Scattering was performed with a dilatometer in the beam to have accurate control over the temperature of the specimen during the phase transformation. Ex-situ Small-Angle X-ray Scattering was performed to quantify precipitates. Different heating and cooling cycles on three types of steels were tested to explore the effect of holding time at high temperatures and cooling rate on the final structure of the steels.

THE RESULTS AND EXPECTED IMPACT

The results show how heat treatment or welding can alter the phase fractions and precipitate content of duplex steels. Experimental results were used to modify a simulation tool which allows prediction of phase transformations. The results are expected to be implemented directly by the manufacturers to minimize the formation of unwanted phases during the process. The experiment was a great demonstration of the power of synchrotron radiation techniques for industrial process development, showing both advantages and limitations of the techniques for addressing similar challenges.

The project brought together partners from the academy (KTH), research institute (Swerim), steel manufacturers (Sandvik and Outokumpu), material users (Alfa Laval) and staff from a large-scale facility (Petra III). This promoted extensive discussion and collaborations to design the experiment, resulting in increased knowledge/skills for all partners.



Contacts: Jan Y. Jonsson, Outokumpu AB, jan.y.jonsson@outokumpu.com
Shirin Nouhi, Swerim AB, shirin.nouhi@swerim.se

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INDUSTRIAL PILOT PROJECT

Characterisation of hydrogen and strain interactions in the microstructure of duplex stainless steel using x-ray diffraction

THE INDUSTRIAL CHALLENGE

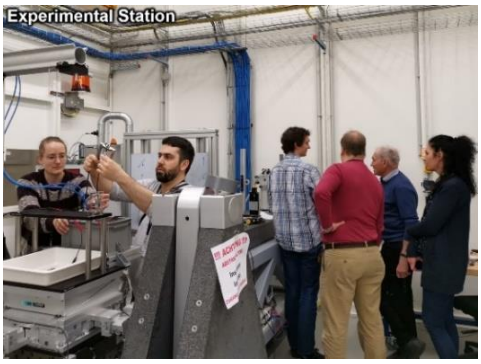
Duplex stainless steels have experienced some failures in subsea applications when subjected to cathodic protection. This is due to hydrogen-induced stress cracking, a type of hydrogen embrittlement failure. Since only large components such as forgings have had failures, but not smaller components such as seamless tubes, this has been linked to the coarseness of the microstructure. Understanding the effect of hydrogen on microstructural degradation of advanced stainless steels has however remained challenging.

WHY USING A LARGE SCALE FACILITY?

The strains in the microstructure associated with hydrogen infusion are very small (sub-Ångstrom metre) which require ultra-high-spatial-resolution measurement of the lattice parameters (sub-Ångstrom). Hydrogen mobility in microstructures is ultra-high, and to study hydrogen-metal require rapid testing while the material of interest is under mechanical loading. While the use of more traditional experimental techniques can't provide sufficient knowledge, synchrotrons enables x-ray diffraction measurements with ultra-high spatial and temporal resolution.

HOW THE WORK WAS DONE

The investigation of the early stages of hydrogen-induced material degradation on commercial duplex stainless steel was carried out with high energy XRD with low angels for surface characterization as well as layer by layer for effects of hydrogen ingress and strains through the material.



This was done under in operando conditions. The Swedish beamline P21.2 at the German synchrotron Petra III (DESY) in Hamburg was used. The conditions were strained material in sodium chloride with a cathodic protection potential imposed. The work was performed by the partners and beamline scientists at DESY (Dr Timo Müller and Dr Ulrich Lienert) present.

The results were correlated with data obtained from various laboratory techniques (EIS, AFM and SKPFM).

THE RESULTS AND EXPECTED IMPACT

The main results were the development of an experimental method and adjustments of an in-situ cell for the P21.2 beamline. Obtained data indicate that this method is promising to for investigation of microstructural influence on hydrogen degradation. The results showed that hydrogen leads to the development of tensile strains in the microstructure and that most strain evolution were concentrated on the surface region where hydrogen first entered the material. More strains were developed in the austenite phase than the ferrite phase during hydrogen charging. The outcome has improved our understanding of material degradation in earliest stages due to hydrogen infusion in stainless steel. The collaboration has set up a network with multi-disciplinary focus on material research for tomorrow's materials. The collaboration has further enabled access to large-scale synchrotron facilities with a special focus to meet industrial demands, which will be utilized for further experiments.

Contacts: Ulf Kivisäkk – Sandvik Materials Technology, ulf.kivisakk@sandvik.com
Jinshan Pan – KTH Royal Institute of Technology, jinshanp@kth.se

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INDUSTRIAL PILOT PROJECT

3D investigation of grain orientation induced braze alloy wetting

THE INDUSTRIAL CHALLENGE

Braze clad on aluminium sheets enables fast and convenient brazing assembly of complex heat exchangers. A well-known, but poorly understood, application problem is that the braze alloy may penetrate into the bulk of the sheet material, which reduces the corrosion resistance.

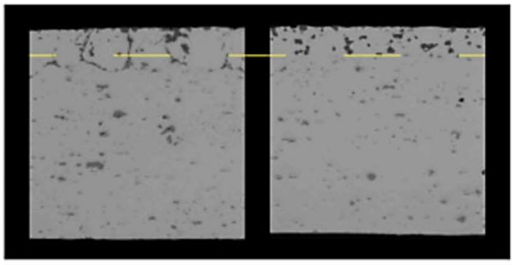

Figure 1. Aluminium sheet with a braze clad before brazing (right) and after brazing (left). The dashed yellow line highlights the original boundary between the aluminium core and the braze clad.

WHY USING A LARGE SCALE FACILITY

Recent studies indicate that the relative orientations of neighbouring grains might play a key role for braze alloy grain wetting. The results indicate that the effect may be critically dependent on the grain orientation mismatch. Obtaining the grain orientation is difficult and time consuming when using conventional techniques. Convenient synchrotron radiation-based methods, on the other hand, can provide 3D images that show orientations, shapes and locations of all grains in a sample.

HOW THE WORK WAS DONE

To obtain detailed 3D images the work has included three synchrotron radiation-based 3D imaging techniques: 1) Phase-contrast X-ray tomography (PCXCT), 2) 3D X-ray diffraction (3DXRD) and 3) X-ray diffraction contrast tomography (DCT). Studies have been performed at BL14B2 at SPring-8 in Japan and at the Swedish beamline (P21-2) at PETRA III in Hamburg, Germany. The SPring-8 session focused on the DCT in combination with PCXCT while the PETRA III session focused on 3DXRD in combination with PCXCT. These studies were complemented by additional X-ray tomography at the 4D Imaging Lab at Lund University. Project participants were representatives from Gränges R&I, Uppsala Synchrotronix AB and Lund University, together with the 3D imaging experts Dr. Daiki Shiozawa (Kobe University) for the SPring-8 session and Dr. Johan Hektor (DESY) for the PETRA-III session.

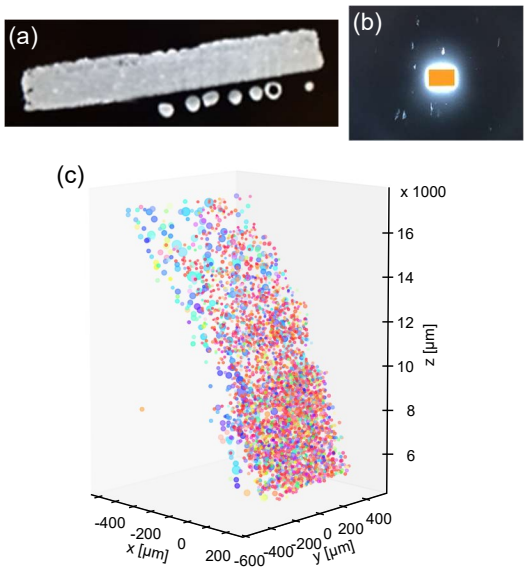

Figure 2. (a) a slice from the tomography imaging at SPring-8. The balls under the aluminium sample are glass beads that make it easier to identify positions in the samples. (b) the DCT data where every bright spot around the centre is a diffraction-projected grain image with a certain orientation. (c) the 3DXRD result image where every circle represents a grain. The diameter and colour of the circles corresponds to the grain size and orientation, respectively.

THE RESULTS AND EXPECTED IMPACT

The acquired data have provided 3D images of aluminium sheet materials showing characteristic signs of different stages of braze alloy penetration. The project has provided new perspectives on the braze alloy penetration process and valuable inputs for new approaches towards reducing this unwanted phenomenon.

"With the knowledge we have acquired we are now at the forefront in this field"
/ Torkel Stenqvist, Gränges R&I

GRÄNGES LUND UNIVERSITY Uppsala Synchrotronix AB

Contacts: Linda Ahl – Gränges Sweden AB, linda.ahl@granges.com
Stephen Hall – Lund University, stephen.hall@solid.lth.se
Mårten Edwards – Uppsala Synchrotronix AB, marten@synchrotronix.se

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INDUSTRIAL PILOT PROJECTS

3D analysis of fatigue cracks in cast irons using in-situ x-ray synchrotron tomography

THE INDUSTRIAL CHALLENGE

Trucks and buses from Scania CV AB have several cast iron parts exposed to cyclic loads. These cyclic loads may lead to fatigue damage. A significant factor in damage development for cast iron components is crack propagation and this very often controls the total life of a component. A deeper understanding of the relation between the different microstructural constituents and the propagation of cracks would enhance material development efforts, as well as the ability to design and cast components with improved fatigue properties

WHY USING A LARGE SCALE FACILITY

Synchrotron experiments at a large scale facility are necessary to achieve sufficiently high spatial and temporal resolution to study the cracking during a fatigue load cycle.

HOW THE WORK WAS DONE

Cracks were initiated using a resonant fatigue test machine in the laboratory of RISE. The test samples were imaged by x-ray tomography at the 4D Imaging Lab at Lund University both prior and subsequent to the crack initiation. The tomograph images show the microstructure and defects, including shrinkage porosities, as well as the initiated fatigue crack.

Synchrotron experiments were performed at the Swedish beamline, P21, at the German synchrotron Petra III (DESY) in Hamburg. These experiments focused on tomography imaging of fatigue crack evolution and the microstructure of cast iron under mechanical load. The samples were imaged while subjected to stepwise loading, according to the fatigue load cycle, to study the opening and closure of the fatigue crack. In addition, stepwise loading at load levels exceeding the fatigue load cycle was applied to further grow the crack. Around 150 tomography images were produced to follow the crack growth. Johan Hector at P21 is greatly acknowledged for his assistance during the experiment.

Digital Volume Correlation (DVC) analyses were performed on the tomograph image sequences, which yields the strain distribution within the loaded samples and the possibility to assess the damage and deformation that takes place, e.g., in relation to graphite particles, pores and other defects.

THE RESULTS AND EXPECTED IMPACT

The analysis of the produced data shows that it is possible to observe damage and deformation mechanisms taking place at different microstructural features like graphite particles, porosities and carbides. These findings would not be possible to observe without 3D synchrotron imaging and in-situ loading. This will deepen the understanding and contribute to the development of materials with improved fatigue properties.

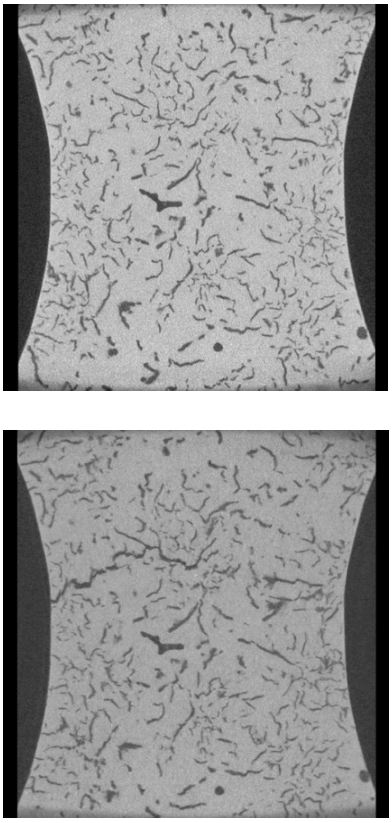

Figure. 2D slices extracted from the 3D tomography Images of the same sample with (lower) and without (upper) an initiated fatigue crack.

Contacts: Jessica Elfsberg – Scania CV AB, jessica.elfsberg@scania.com
Torsten Sjögren – RISE Swecast, torsten.sjogren@ri.se
Stephen Hall – Lund University, stephen.hall@solid.lth.se

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INDUSTRIAL PILOT PROJECT

Development of hydrogen barrier coatings for fuel cell applications

THE INDUSTRIAL CHALLENGE

Sandvik Materials Technology (SMT) provides coated steel strip products for fuel cell applications; Sandvik Sanergy® LT for low temperature fuel cells (PEMFC) and Sandvik Sanergy® HT 441 for high temperature (SOFC) applications. For the next generation of coatings for SOFC applications we investigate hydrogen (H) barrier coatings. The effect of hydrogen is well known as it severely deteriorates the corrosion protection of the interconnect material. However, the exact mechanism is not understood

WHY USING A LARGE SCALE FACILITY

Neutron reflectivity (NR) measurements have the potential to unravel the presence of H deep inside a metal matrix, in a direct and fully non-invasive manner. This is not possible with any other technique. Of specific concern is the application of NR for studies of the effect of a dual atmosphere (i.e. H_2 gas on one side of the sample, and air on the other side) on samples of FeCr alloy. In an operating SOFC, the dual atmosphere is known to damage the corrosion resistant oxide layer formed on the so-called interconnect - the question is why.

HOW THE WORK WAS DONE

SMT and Chalmers have been collaborating for many years in the field of SOFC materials, but the collaboration with their NR expertise was new. A series of samples were exposed to typical SOFC operating conditions at Chalmers and subjected to NR measurements at the Super ADAM instrument at the Institut Laue-Langevin (ILL) in Grenoble, France.

THE RESULTS AND EXPECTED IMPACT

A somewhat unexpected finding from this very first NR experiment was the observation of a large surface roughness, which we believe originate from the thermal growth of the protective oxide layer. To measure on samples with a lower surface roughness, which is normally needed for a high-quality NR pattern, special attention has been directed towards the preparation of more flat samples, which is further described in the following. Figure 1 shows the NR data as measured on the sample exposed to SOFC condition (green) as well as on an unexposed sample (black).

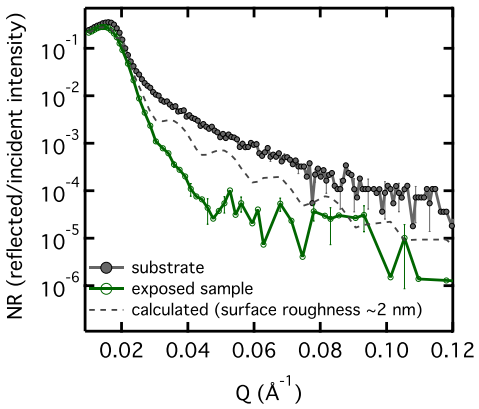

Figure 1. NR curves as measured on a unexposed sample (substrate, black) and an exposed sample (green). A calculated curve for the case of a sample with low roughness (gray, dashed) is also shown.

The NR data suggests that the exposure leads to an increase of the surface roughness from ~1 nm (on the surface of the substrate) to ~5 nm. Furthermore, the analysis indicates that the ~5 nm roughness is mainly at the oxide/air interface, meaning that the low roughness at the substrate/oxide interface is virtually unaltered under exposure. This is an important new result in itself that provides insight into the oxidation mechanism of the material and that encourages further NR studies, such as on a systematic series of films varying in exposure time.

Nonetheless, it should be noted that the increase in surface roughness causes a damping of the oscillations of the NR pattern, see *e.g.* the calculated NR curve in Fig. 1. This effect makes the extraction of the chemical composition and quantification of H in the film very difficult. This encourages the use of alternative sample preparation techniques, such as sputtering, to prepare atomically flat samples. With a view towards the future, we recognize the unique potential of further NR experiments on these samples and, for this reason, both SMT and Chalmers are committed to continue this track outside the scope of this project.

Contacts: Ulf Bexell – Sandvik Materials Technology, ulf.bexell@sandvik.com
Jan Froitzheim – Chalmers, jan.froitzheim@chalmers.se
Maths Karlsson – Chalmers, maths.karlsson@chalmers.se

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INDUSTRIAL PILOT PROJECT

In situ XPS of Al_2O_3 break-up at flux-free brazing conditions

THE INDUSTRIAL CHALLENGE

Braze clad on aluminium sheets enables fast and convenient brazing assembly of complex heat exchangers. The dominant brazing technique today is controlled atmosphere brazing (CAB) which is performed in a furnace with nitrogen (N_2) gas and temperatures around 600 °C. Before brazing it is necessary to dissolve the inherently formed surface oxide. The CAB technique however requires the use of a potassium fluoroaluminate flux for the oxide break-up process. Beside a negative effect on health and environment, the flux residues in the ready-made heat exchanger can interact chemically with coolants and form a gel in the heat exchanger tubes. The tubes become clogged and suppresses the cooling capacity. Due to this, flux-free brazing where magnesium acts as an oxide break-up agent is an attractive alternative. The knowledge about this process is however very limited and it is a challenge to obtain a stable process – so far it has been a trial and error procedure.

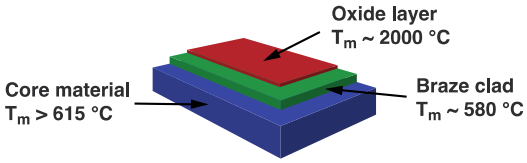

Figure 1. Braze clad aluminium sheet where the braze clad melts at about 580 °C. The surface oxide must be dissolved before the molten braze clad can flow to regions where to form joints.

WHY USING A LARGE SCALE FACILITY

X-ray photoelectron spectroscopy (XPS) is an element sensitive technique that can provide chemical-specific information about the probed elements in a sample. It is, thus, possible to follow the changes in the XPS spectra for oxygen (O), aluminium (Al), and magnesium (Mg) during a heat treatment. That means that the kinetics of the break-up process of Al_2O_3 initiated by Mg is tracked while it occurs. To avoid evaporation of Mg it is necessary to perform the experiment at 1 mbar N_2, which require the high X-ray intensity that can be obtained in a synchrotron radiation (SR) facility. In addition, the high X-ray intensity facilitate fast data acquisition necessary for *in operando* measurements. By using SR it is also possible to tune in the provided photon energy for depth profiling through the oxide overlayer. Hence, this study would be impossible using conventional XPS.

HOW THE WORK WAS DONE

An initial investigation was performed at Linköping university using a conventional surface science XPS system. The samples were then heat-treated at Gränges R&I in a CAB furnace and the obtained spectra showed O 1s, Al 2p, and Mg 2p before and after the heat-treatment. The SR-based *in operando* study was performed at ambient pressure at the XPS beamlines HIPPIE and SPECIES at MAX IV in Lund, Sweden. Project participants were representatives from Gränges R&I, Uppsala Synchrotronix AB, and Linköping University.

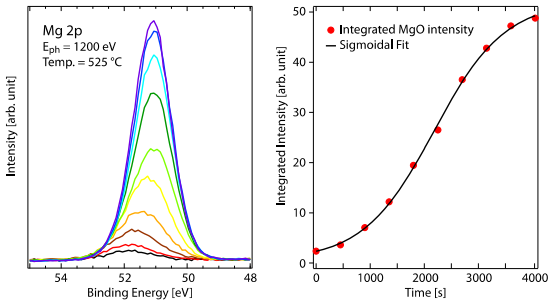

Figure 2. Mg 2p XPS of the oxide layer above the braze clad and how the intensity evolves as a function of time at a temperature of 525 °C.

THE RESULTS AND EXPECTED IMPACT

The study has successfully provided new information regarding changes in the oxide composition, as well as the Mg-kinetics. It also displayed the oxide break-up mechanism, when Mg diffuses from the aluminium sheet core material up to the oxide through the braze clad at temperatures close to brazing conditions. The project has provided new perspectives on the Al_2O_3 break-up process and valuable inputs for the development towards a stable process that facilitates flux-free brazing.

"Nobody has done this study before us. It has simply not been possible"
/ Lars-Åke Näslund, Gränges R&I

LINKÖPINGS UNIVERSITET GRÄNGES Uppsala Synchrotronix AB

Contacts: Linda Ahl – Gränges Sweden AB, linda.ahl@granges.com
Robert Moberg – Uppsala Synchrotronix AB, robert@synchrotronix.se

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INDUSTRIAL PILOT PROJECT

Characterization of secondary carbides in commercial steel grades using small angle neutron scattering experiments

THE INDUSTRIAL CHALLENGE

The hardness and strength of martensitic steels can be increased by formation of nano-sized carbides, so-called precipitation hardening. Molybdenum and vanadium are both strong carbide forming elements and are often added to the steel composition to increase hardness and strength. In order to form carbides that are ideally homogenously distributed within the steel microstructure, the steel is heat treated, quenched and tempered. It is important for SSAB and Uddeholm to better understand the precipitation sequence during tempering. When designing new steel alloys physically based modelling is often used to predict number densities, size distribution and volume fractions of formed particles, related to alloy content and heat treatment. Thus, extensive data regarding number density and size distribution of formed carbides is of great importance to improve and develop better models.

WHY USING A LARGE SCALE FACILITY

Traditionally, transmission electron microscopy (TEM) and atom probe tomography (APT) have been used to study small precipitates in steel. The micro-to-nano structure of commercial steels can be very complex, with local deviations. Since TEM and APT only analyse a very small and local sample volume, it is difficult to get a complete picture of the resulting nano-structure. On the other hand, small angle scattering (SAS) techniques provide significantly more data over much larger sample volume. The penetration depth of neutrons is very high compared to X-rays. Small angle neutron scattering (SANS) thus provides a more viable path for characterization of nano-particles. SANS provides data from a large sample volume, while retaining a similar detected length scale as TEM/APT (1-100nm). SANS also allows to measure the precipitation sequence in-situ, which is not possible with the laboratory-based techniques. This will help to verify and improve models used to predict particle distribution.

HOW THE WORK WAS DONE

SANS experiments were conducted at the Sans2D beamline of ISIS Neutron and Muon Source in England by representatives from both companies supported by experts from Swerim and Chalmers. The two alloys investigated had been heat treated at 500-650°C for up to 40h. They were of high and low alloy content, respectively, and consequently with different amount of small carbides. A magnetic field (1.5T) was applied perpendicular to the incident beam during measurements to separate the nuclear and magnetic scattering.

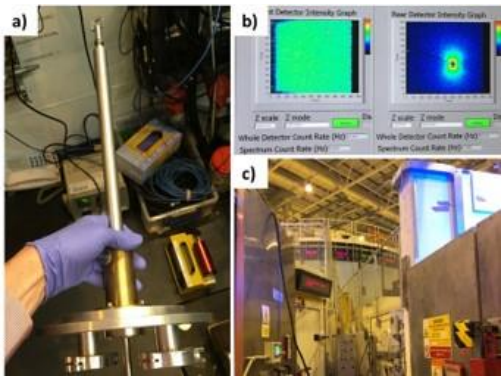

Figure. a) Sample holder, b) live monitoring of detectors, c) the experimental station.

THE RESULTS AND EXPECTED IMPACT

Clear signals could be detected from different particle types in different size ranges. The results regarding precipitation sequence and size and shape of the particles correlate well with the data previously obtained from TEM and APT. The data also allows the size distribution to be determined with very good statistics. More in depth data analysis is planned together with verification of physical based models using the results.

"The high output of data at this length scale is just amazing"*/Robin Nilsson, SSAB EMEA AB*

Contacts: Robin Nilsson – SSAB EMEA AB, robin.nilsson@ssab.com
Christos Oikonomou – Uddeholm AB, christos.oikonomou@uddehom.com
Erik Claesson – Swerim AB, erik.claesson@swerim.se
Mattias Thuvander – Chalmers Tekniska Högskola, mattias.thuvander@chalmers.se

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INDUSTRIAL PILOT PROJECT

Quantitative characterization of chemically complex low alloyed high strength tempered martensitic steels using ASAXS

THE INDUSTRIAL CHALLENGE

The strength of low-alloyed steels is improved by the nano sized secondary carbides rich on molybdenum (Mo) and vanadium (V). To set the properties of these SSAB steels, they are quenched from high temperature and then tempered at 500-600ºC for some hours. It is important for SSAB to better understand the precipitation sequence in order to optimise alloying and heat treatments processes.

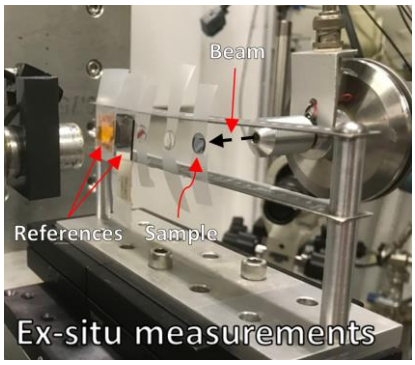
WHY USING A LARGE SCALE FACILITY

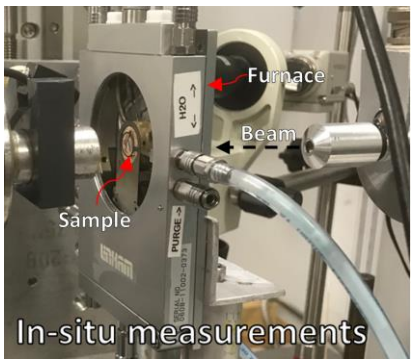
The precipitation sequence of nano-sized particles has previously been analysed with electron microscopy (SEM/TEM) and atom probe tomography (APT). However, quantifying particles with these lab-based techniques is limited by the small probe volume. By using scattering techniques at large scale facilities, a greater sample volume can be analysed which is useful for determining size distributions and phase fractions of carbides. In previous work small angle neutron scattering (SANS) has been used to detect these small carbides, but due to the complexity of the steel with many different but similar carbides, it was difficult to isolate the scattering contribution from different sorts of carbides. Carbides with similar size range can be iron carbides or Mo/V secondary carbides. With anomalous small angle x-ray scattering (ASAXS) at a synchrotron facility it is however possible to distinguish the anomalous effect (AE) which is selectively related to Mo-rich carbides. ASAXS was therefore chosen as suitable supplement to previous characterisations.

HOW THE WORK WAS DONE

ASAXS experiments were performed at the Advanced Photon Source (APS) in USA, by representatives from SSAB, Kungliga Tekniska Högskolan and Swerim. A monochromatic x-ray beam with energies close to the absorption edge of Mo was used to study differences in the Mo-content at different q-ranges. X-ray energies were close to 20 keV.

In total 20 different energies were measured for each sample to study the anomalous effect (AE) related to Mo-rich carbides with ASAXS. The measurements were performed both ex-situ and in-situ at 550 °C.





THE RESULTS AND EXPECTED IMPACT

From the data analysis it is evident that the AE is observed only at high q-values, i.e. the small particles. This indicates that the smallest carbides can be separated through the AE-effect at ASAXS experiments, which gives additional information compared to previous SANS experiments. This effect becomes even more pronounced with longer tempering times, which is reasonable since Mo portioning in the secondary carbides requires time for diffusion. The improved understanding of the precipitation sequence obtained within this project can aid the further optimization of heat treatment and alloying of these commercial materials.

Contacts: Diana Orrling – SSAB EMEA AB, diana.orrling@ssab.com
Erik Claesson, Fredrik Lindberg – Swerim AB, erik.claessonl@swerim.se,
Peter Hedström – Kungliga Tekniska Högskolan, pheds@kth.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Exploring XANES and EXAFS to access wear behaviour of cutting tools during high-speed turning

THE INDUSTRIAL CHALLENGE

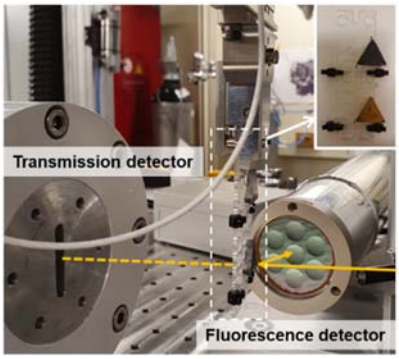
Metal machining requires high-performance cutting tools with excellent wear properties to enable high cutting speeds and thus high production rates. To design next generation high-speed cutting tools, detailed knowledge on the tool wear behaviour is required.

WHY USING A LARGE SCALE FACILITY

The part of the tool that is in contact with the chip is inhomogeneous as a result of differences in temperature and stress on the tool. Thus, characterization methods with a good spatial resolution are required to resolve local changes of the surface.

HOW THE WORK WAS DONE

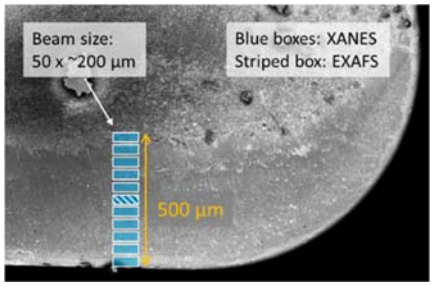
Worn cutting inserts were prepared by turning in stainless steel with TiAlN coated tools. Five samples with different Al-content in the coating were studied. EXAFS and XANES experiments were performed at the BALDER beamline of the MAX IV synchrotron, in Lund. The samples were placed so that the beam was incident at 10° to the surface normal and the fluorescence signal was detected at 80° exit angle.



The chemical changes across the surface of the tool were probed by measurements at the Ti *K* absorption edge. Additional measurements at the Cr-*K* and Fe-*K* edges were made to investigate the nature of the steel species adhered to the tool. Complementary studies of structural changes were performed by transmission electron microscopy (TEM) at Linköping Uni.

THE RESULTS AND EXPECTED IMPACT

The spectral features from XANES data on the as-deposited TiAlN coatings revealed differences in the cubic and hexagonal chemical environments and their hybridization, thus changes in the atomic arrangement as a function of Al content could be identified. The XANES line scans across the surface of the worn tools showed that there are changes in the atomic arrangements in the coating at different spatial areas of the worn cutting edge.



The EXAFS measurements on selected regions of the worn areas also showed changes in bond distances and number of neighbours between the atoms of the coatings compared to the corresponding as-deposited reference coatings. Additional TEM investigations revealed formation of nm-sized domains enriched in TiN.

To conclude, XANES and EXAFS are useful techniques as they are sensitive to very small local changes in the atomic structure of the coatings. The results reveal the possibility of accessing chemical interactions during use of the tool by x-ray based methods. This knowledge will be used for designing future experiment to further explore the material behaviour during machining and in the extent to optimize the material of the tool.

"As researchers we always want to study and understand our materials at the highest possible detail. These techniques help us explore the unknown" /Jon Andersson, Seco Tools

Contacts: Jon Andersson – Seco Tools, jon.andersson@secotools.com
Lina Rogström – Linköping University, lina.rogstrom@liu.se

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INDUSTRIAL PILOT PROJECTS

Improved steel cleanliness analysis with inclusion detection and quantification using synchrotron X-ray microtomography

THE INDUSTRIAL CHALLENGE

Foreign particles (inclusions) in steel, originating from early stages in the steel production process may reduce mechanical properties. As a world leading steel producer SSAB constantly strives to improve the cleanliness of the steel and one key is improved methods for the detection and quantification of inclusions.

WHY USING A LARGE SCALE FACILITY

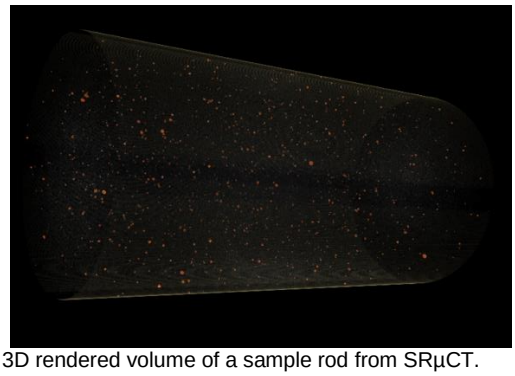
Present standard techniques to investigate steel cleanliness are 2-dimensional (2D), e.g. light optical microscopy (LOM) and scanning electron microscopy (SEM). These methods are limited since interpretation by stereographic methods and assumption of a regular inclusion size distribution must be done in order to obtain size, shape and volume fraction of the inclusions. Difficulties arise when inclusions are few or when there is a varying orientation or shape of inclusion particles and irregularities in the inter-particle connectivity. The 3D nature of non-destructive tomographic imaging techniques can overcome this. The low X-ray intensity of laboratory-based X-ray tomography (µCT) demands for a longer scan time, thus making impeding the technique to be used on a commercial basis. With the high beam brilliance at a modern synchrotron source, X-ray microtomography SRµCT is less time consuming. Following the recent developments and increased availability of SRµCT combined with optimized work-flows for image analysis, this technique could potentially be offered as a standard and routinely used technique for industrial inclusion assessments.

HOW THE WORK WAS DONE

The inclusion detection and quantification SRµCT experiments were performed at the P07 High Energy Materials Science (HEMS) beamline of Helmholtz-Zentrum Geesthacht at the PETRA III storage ring at the DESY synchrotron in Hamburg, Germany. 12 samples of low carbon steel were prepared as rods with 10 mm height and 1-2 mm in diameter. To validate the results of the SRµCT measurements, standard testing was done using microscopy methods, i.e. LOM and SEM.

THE RESULTS AND EXPECTED IMPACT

With the SRµCT method, it was possible to detect inclusions down to the size of 2.4 µm, which is similar to standard 2D techniques, however with a much shorter scan time, about 15 min compared to several hours for standard 2D techniques. The image below shows a 3D rendering of a sample volume, where the inclusions are clearly visible.



3D rendered volume of a sample rod from SRµCT.

For image analysis of individual inclusions, an automatic image analysis script in Python was developed. The quantitative results of SRµCT, LOM and SEM showed strong mutual correlation. Also, the SRµCT revealed the actual size distribution of the inclusions in the samples. Proven as a fast and precise method for 3D inclusion assessment, a very interesting next step would be to test steels for correlation between SRµCT data and relevant mechanical properties.

Contacts: Niklas Kojola - SSAB AB. niklas.kojola@ssab.com
Fredrik Lindberg - Swerim AB, fredrik.lindberg@swerim.se
Emanuel Larsson – RISE Research Institutes of Sweden, emanuel.larsson@ri.se

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INDUSTRIAL PILOT PROJECT

Stress mapping on ultra-high strength steel (UHSS) cut edges using high-energy X-ray diffraction

THE INDUSTRIAL CHALLENGE

During cutting operations in the process route from steel plate to finished product stresses can be introduced that can be detrimental later in the products life. This is especially true in connection with fracture induced by hydrogen embrittlement (HE). A prerequisite for HE is the introduction of residual stresses originating from plastic deformation during the cutting process.

WHY USING A LARGE SCALE FACILITY

The investigation and optimization of the internal stresses introduced during the cutting process is clearly important. Direct measurements of residual stresses on the microscale would open new ways for optimizing materials and cutting processes for high strength steels. The best conventional XRD has probes with a diameter of 0.5 mm and a depth penetration in steel of 20 µm. This makes local measurements with the required geometry impossible. With the deep penetration and a small probe of high energy X-ray sources; however, such measurements become possible.

HOW THE WORK WAS DONE

We used pencil-beam high-energy X-ray diffraction at the Swedish beamline P21.2 of the synchrotron PETRA III, Hamburg, to map stresses down to the microscale at cut edges. The work was done by representatives from SSAB Europe with support by expertise from Swerim and DESY. The UHSS material analysed was a commercial steel Docol™ 1200M from SSAB Borlänge, with approximately 1 mm thickness. This is mainly intended for use in the automotive industry and is fully martensitic with a minimum tensile strength of 1200 MPa. The material was cut using three different methods; shearing, shearing followed by milling, and by using a CO_2 laser. The cross-sections could be inspected perpendicular to the cut edge in transmission mode. For mapping of the residual stresses, a 30 µm beam was scanned over the sample and a diffraction pattern was collected every 50 µm. Residual macro stresses (or type I) were evaluated by examining the peak shift at the north and east direction of the detected Debye rings.

THE RESULTS AND EXPECTED IMPACT

The residual stresses could be studied with impressive resolution.

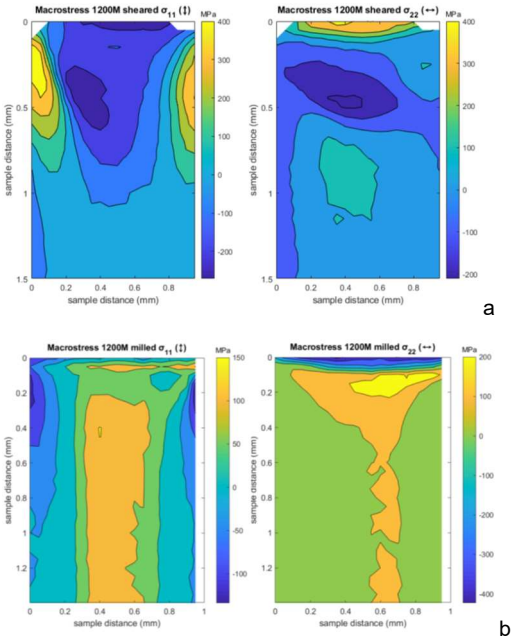

Figure. The constructed macro strain maps calculated by the determined cell parameters in the north (σ11) and east (σ22) direction for the a) sheared sample and b) sheared and milled sample.

The dangerous localized tensile stresses were only found in the sheared samples. These stresses could serve as driving force for crack propagation during HE. Subsequent milling was found to introduce compressive stresses in the immediate surface and therefore reduce the sensitivity for HE.

"This unique XRD method can explain why certain cutting methods should be avoided for processing of UHSS in order to reduce high local tensile stresses and improve HE resistance. It will be a valuable tool for modifications of materials and cutting processes."

/Sven Erik Hörnström, senior expert at SSAB

Contacts: Sven Erik Hörnström and Jonas Östberg - SSAB Europe AB, sven-erik@hornstrom.se
Johannes Brask, Lena Ryde and Fredrik Lindberg - Swerim AB, johannes.brask@swerim.se

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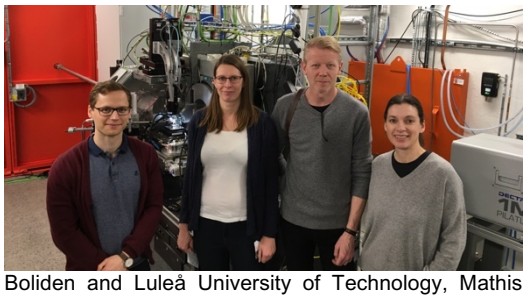
Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Nanoscale characterization of ore samples – optimizing the recovery of metals important for the green shift

THE INDUSTRIAL CHALLENGE

Trace element characterization of complex ores is of importance for Boliden and the rest of the global mining industry as the generation of by-products are becoming more and more common practice in mining. These by-products (metals) typically occur in small concentrations and often irregularly distributed in the ore. To be able to extract these by-products, of which some are vital for the green shift, it is crucial to optimize the analytical procedures used in the planning of metal extraction from mining. Boliden therefore joined forces with ore geological expertise at Luleå University of Technology, to address potential by-product metals in the ore.



Boliden and Luleå University of Technology, Mathis Warlo, Iris McElroy, Glenn Bark and Christina Wanhainen, at the NanoMAX beamline at MAX IV.

WHY USING A LARGE SCALE FACILITY

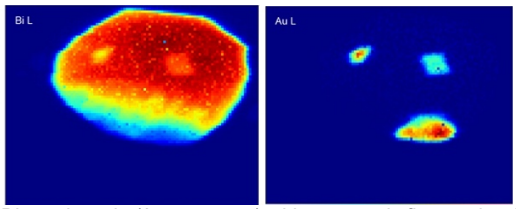
Compared with current analytical routines used by the mining industry (typically different types of electron beam techniques), synchrotron-based nano-XRF allows for a nanoscale spatial resolution of the elemental distribution in the ore samples. Going from conventional micron scale characterization to nanoscale enables frontier science within the scientific fields of ore geology and mineral processing. With the synchrotron analysis (as complementary to the routine techniques used) we aimed at finding out where in the ore sample the metals that are of interest for renewable energy are located. Finding this out, it is then possible to develop mineral processing schemes for an optimized extraction of these metals.

HOW THE WORK WAS DONE

To test the additional value that synchrotron analysis might bring to the analytical routines of the mining industry, we selected a suitable ore sample that is representative of a complex ore that Boliden will mine in the near future. To start with, we went to the synchrotron in Taiwan. There we did some preliminary measurements to test the technique. With an improved understanding of the analytical technique from Taiwan we could then go to the NanoMAX beamline (nano-XRF) at the MAX IV synchrotron in Sweden to do the final and critical measurements. At MAX IV, with the help of expert beamline scientists, detailed maps of the elemental composition of the sample were obtained, using different analytical parameters such as spot size variation (down to 50 nm). This was the first time that ore samples were analyzed at this beamline.

THE RESULTS AND EXPECTED IMPACT

Through this project, we could find out the nanoscale distribution of several metals that are important for green technologies (wind- and solar power), in the complex Boliden ore. Having this knowledge, Boliden can now plan for an improved metal extraction procedure, and recovery of the trace metals. The project team has gained valuable skills regarding synchrotron-based analysis and is now in a really good position to generate new ideas for projects that will use synchrotron-based techniques as a way of generating important scientific data.



Bismuth grain (3 µm across) with extremely fine-grained gold. The nano-XRF image pixel size is 50 nm.

"Future advances in metal extraction enabled by synchrotron nanoscale characterization."
/Iris McElroy, Boliden and Glenn Bark, Luleå University of Technology

Contacts: Iris McElroy – Boliden, Iris.McElroy@boliden.com
Glenn Bark – Luleå University of Technology, Glenn.Bark@ltu.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Residual stress measurements in Hardox™ high strength steel using neutron diffraction

THE INDUSTRIAL CHALLENGE

High strength steels can be sensitive to hydrogen embrittlement (HE) which can limit the application of the steel in certain environments. A prerequisite for HE is the presence of residual stresses built up in the product during production and in service. Residual stresses can be reduced by applying thermal treatments, i.e. tempering, during the production process. However, a limitation is that the time and temperature profile used must not alter the strength of the steel from acceptable values. To optimize the process, there is a need to map the stress distributions. This is the quest for an effective production route that will allow for the full potential of these high strength steel products.

WHY USING A LARGE SCALE FACILITY

It is desirable to map residual stresses throughout the whole of the thickness of steel plates. Non-destructive analysis of the bulk is impossible to perform with conventional laboratory methods (e.g. X-ray diffraction) due to the limited penetration depth. Since neutrons penetrates deep into a material, neutron diffraction is an ideal tool for this.

HOW THE WORK WAS DONE

The neutron diffraction data used to analyse residual stress were recorded from the Engine-X diffractometer at ISIS Neutron & Muon Source, UK.

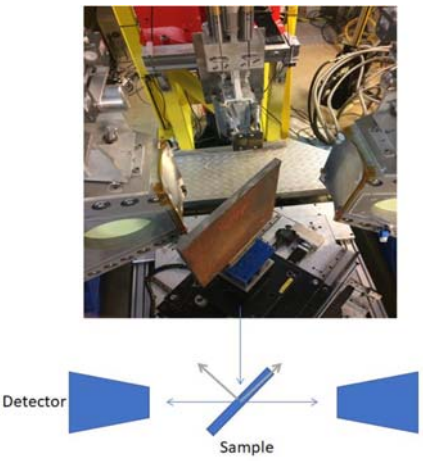

Figure 1. The experimental set-up of Engine-X.

Measurements were performed on samples after quenching, tempering and levelling of the steel plate. The residual strain and stress at each measurement depth was calculated using the bcc cubic cell parameters obtained from the corresponding neutron time of flight data.

THE RESULTS AND EXPECTED IMPACT

Examples of macro stress profiles are shown in Figure 2, where the quenched state is compared to the 600°C tempered sample.

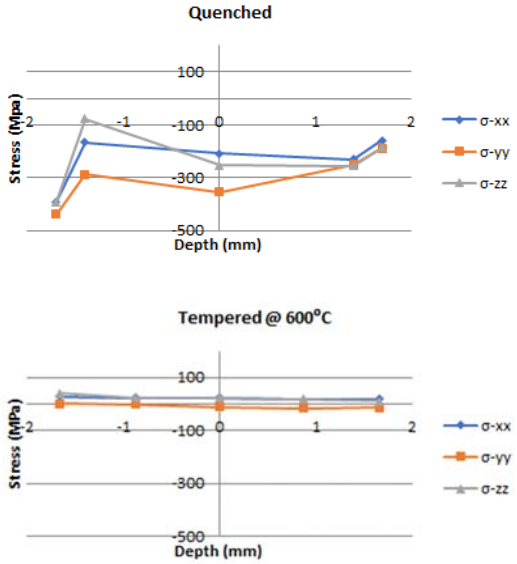

Figure 2. Stress diagrams with stress in MPa vs sampling depth.

The residual stress levels and the through plate stress profile changed between the different process steps and both levelling and tempering reduced the stresses in the plate. The results need to be further analyzed since the stresses differ between the top and bottom side of the plate and the reason for this needs to be clarified.

"For SSAB it is important to see the evolution of the residual stresses in the production route and further to develop steel with higher resistance to hydrogen embrittlement. Also important is the aspect of sharing the possibilities with neutron diffraction within the R&D function throughout the company." /Torbjörn Narström, SSAB Special Steels

Contacts: Torbjörn Narström - SSAB Special steels AB. torbjorn.narstrom@ssab.com
Fredrik Lindberg, Lena Ryde and Sten Wessman - Swerim AB, fredrik.lindberg@swerim.se
Jakob Blomqvist and Christina Bjerkén – Malmö Universitet, jakob.blomqvist@mau.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Study of precipitates in an additively manufactured maraging steel using small angle neutron scattering

THE INDUSTRIAL CHALLENGE

With a good combination of high strength and adequate toughness, maraging steels have been adapted to many engineering applications. Due to their excellent welding behaviour, maraging steels are also among the most promising candidates for additive manufacturing (AM). For those reasons, maraging steels are of great interest to Uddeholms AB as candidate materials for AM products in tooling applications. Understanding the characteristics of the precipitation behaviour of the AM maraging steel would considerably assist Uddeholms AB in tailoring heat treatments to achieve optimized properties with respect to targeted applications.

WHY USING A LAR7GE SCALE FACILITY

Laboratory-based techniques such as scanning electron microscope (SEM) and transmission electron microscope (TEM) can reveal the morphology of precipitates at selected cross-sections. However, analysis of the nano-scale precipitates' volume and size distribution with statistical significance requires small angle neutron scattering (SANS) experiments.

HOW THE WORK WAS DONE

Specimens of 12mm x 12mm x 0.8mm (nominal dimensions) were manufactured by laser powder bed fusion (LPBF) and hot isostatic pressing (HIP) and subjected to various heat treatment condition. SANS experiments were conducted at the SANS-1 station of Heinz Maier-Leibnitz Zentrum (MLZ) at the German neutron facility FRM II. In order to measure precipitates in a large size range, four scattering geometries from combining four detector-sample distances and two neutron wavelengths were used to cover a wide range of scattering vector (q-range: 0.004 Å-1 to 0.4 Å-1).

We sincerely thank Dr. Sebastian Mühlbauer and Dr. Apostolos Vagias from MLZ for their contribution.

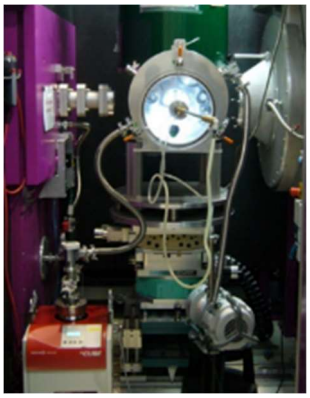

Figure 1. A typical set-up of SANS-1.

THE RESULTS AND EXPECTED IMPACT

An initial analysis of the SANS results was performed, and it was found that there was strong multiple scattering, due to specimens were too thick. Current thickness of around 0.8 mm was used in a nickel-based super alloy to analyse precipitates with SANS-1, and it worked well. However, it did not work for the current maraging steel. A speculation is that the amount of precipitates are too large, which highly increased the chance that a neutron got scattered more than once before reaching the detector. Hence, thinner specimens are required for a successful measurement. Based on the outcome, a specimen thickness of 0.2 mm was then suggested by one beamline scientist at FRM II. The consortium of this project plans to send a new proposal on this very subject to SANS-1 at FRM II.

"There is a great potential in addressing alloy design related topics for complex manufacturing processes with large scale facilities. Meanwhile, also new challenges need to be overcome in order to exploit its maximum potential."
/Christos Oikonomou, Uddeholms AB

Contacts: Christos Oikonomou – Uddeholms AB, christos.oikonomou@uddeholm.com;
Taoran Ma – RISE IVF, taoran.ma@ri.se;

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Understanding heat-treatment of Hybrid Steel® using in-situ and ex-situ synchrotron X-ray diffraction

THE INDUSTRIAL CHALLENGE

The newly developed Hybrid Steel® of Ovako obtains its high strength by combining precipitation of intermetallic NiAl precipitates and carbides. This makes the precipitation process rather complex and a detailed understanding is needed to tailor heat treatments for each application.

WHY USING A LARGE SCALE FACILITY

The precipitates in Hybrid steel are generally very small, below 10 nm. This means that time-consuming techniques like transmission electron microscopy (TEM) or atom probe tomography (APT) are needed. By using synchrotron X-ray diffraction and scattering, valuable information can be obtained quickly. The fast acquisition also allows the precipitation process to be studied in-situ.

HOW THE WORK WAS DONE

Simultaneous Wide-Angle X-ray Scattering (WAXS) and Small-Angle X-ray Scattering (SAXS) was performed at the Swedish Materials Science Beamline P21 at Petra III in Hamburg, with an energy of 60 keV. The experiments were carried out during two visits by researchers from Ovako and Chalmers University of Technology, with assistance from the beamline scientist Dr Timo Müller. The in-situ measurements were carried out using a Linkam furnace with heat treatment times ranging from 1 to 20 hours.

Figure 1. Experimental set-up at P21 with the water-cooled and Ar-protected furnace in the foreground.

During the first visit sheet samples were used, but it was observed that the expected max temperature of 600˚C could not be reached. After modifications of the furnace to allow for match-stick shaped samples, the furnace worked well and reached 600˚C during the second visit.

THE RESULTS AND EXPECTED IMPACT

The experiments generated a large amount of data. Heat-treated samples could quickly be measured ex-situ, giving information on the precipitate structure (WAXS) and size (SAXS). The in-situ tests provided information about the temporal development of the precipitates, and also changes in the phase fraction of retained austenite. Also, changes in the lattice parameters of the austenite and the martensite could be followed during heating, holding and cooling. This type of knowledge cannot be obtained by carrying out only ex-situ measurements.

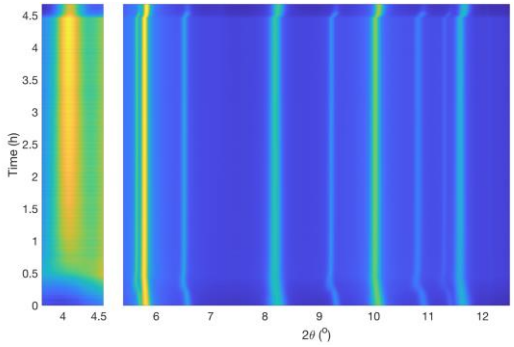

Figure 2. Development of X-ray peaks as a function of time during in-situ measurements. The left peak shows the formation of NiAl. At the top, the drop in the amount of austenite during cooling can be seen.

The results are expected to influence heat-treatment schemes and possibly as guidance when developing new grades of the hybrid steel family.

OVAKO

CHALMERS

Contacts: Fredrik Lindberg – Ovako Sweden AB, fredrik.lindberg@ovako.com
Mattias Thuvander – Chalmers University of Technology, mattias.thuvander@chalmers.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

X-ray Absorption Spectroscopy for in-depth understanding of NiMo catalysts used for biofuel production

THE INDUSTRIAL CHALLENGE

Lignin is a biproduct from the pulp and paper industry that has a large potential as a renewable feedstock to produce value-added chemicals, biofuels, and aromatics. One of the important steps in the production of biofuel from lignin is the catalytic hydrotreatment processes. For this process Sun Carbon has developed a tailored $NiMo/Al_2O_3$ catalysts. A key requisite for designing and optimizing the catalytic process is to be able to link the catalytic properties with the detailed characteristics of the catalysts.

WHY USING A LARGE SCALE FACILITY

Due to the thin layer of active metals (Ni and Mo) on the support (Al_2O_3), the characterisation of the catalyst is a challenge. Synchrotron-based X-ray absorption spectroscopy (XAS) was identified as a very promising technique – both for characterising the catalysts itself but also for in-situ studies of redox reactions and of the activation of the catalyst. The technique would, theoretically, also allow operando studies of relevant catalytic processes. One major advantage is that vacuum is not needed when performing XAS at photon energies above ~2000 eV. This allows studies at high pressures, as well as minimal sample preparation (avoiding introduction of potential artefacts).

HOW THE WORK WAS DONE

The XAS experiments took place at the Balder beamline, MAXIV Laboratory. Two different types of material systems were studied: *i*) industrial catalysts ($NiMo/Al_2O_3$) and *ii*) model systems composed by NiMo nanoparticles (NPs). The NP model system was designed with the attempt to perform operando studies in the future. Both types of samples were produced with two Ni:Mo molar ratios (1:2 and 2:1) and the measurements were performed for samples 'as produced' in ambient atmosphere (ex-situ) and in-situ. In the in-situ measurements, the redox reactions of Ni and Mo was studied using two different chamber set-ups in which the samples could be heated up to 500- 600 °C while flowing either 5% O_2 in N_2 or and 5% H_2 in N_2 over the samples. Primarily XANES (the energy region just above the absorption edge) were used, but also EXAFS were measured for a selected number of sub-samples ex-situ. The measurements were performed both in transmission mode and in fluorescence mode to allow correction for self-absorption.

THE RESULTS AND EXPECTED IMPACT

High quality XANES and EXAFS spectra could be retrieved ex-situ, revealing information of the chemical state of Ni and Mo before and after activation. The measurement reveal that the initial oxidation state of the industrial catalyst was different to the NPs but that after reduction during heating, the samples look very similar. The in-situ studies allowed to follow the redox reactions as a function of temperatures in either an oxidizing or reducing atmosphere. Critical information of the on-set temperature of material transformations could be retrieved. As an example, initially the Ni and Mo in the industrial catalyst did not seem to form a real alloy. This was first formed when heated in a reducing environment.

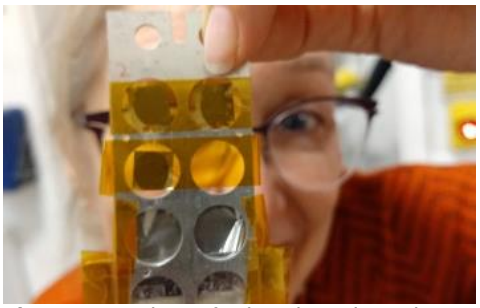

Figure. A variety of NiMo based catalysts were tested with XAS at the Balder beamline at MAX IV.

Based on the learnings about the model particles and the possibilities and limitations with XAS, we conclude that the technique could allow future operando studies during catalytic reaction using a chamber for high pressure experiments.

SunCarbon

Contacts: Christian Hultenberg, SunCarbon, christian@hulteberg.com
Sara Blomberg, Lund University, sara.blomberg@chemeng.lth.se
Jenny Rissler, Research Institutes of Sweden (RISE), jenny.rissler@ri.se

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INDUSTRIAL PILOT PROJECT

Frost durability of concrete addressed by dual-modality neutron and x-ray tomography

THE INDUSTRIAL CHALLENGE

A lot of effort is done to ensure that building material is durable and sustainable but some properties are investigated in a rather coarse way. We are therefore looking into new technologies to help us understand what happens when concrete freeze.

WHY USING A LARGE SCALE FACILITY

Water is an important player in almost all types of degradation. The state and whereabouts of water in the complex pore structure of concrete is in almost every case studied with indirect methods (eg. volume change due to ice formation in the pores). A frost resistant concrete contains air entrainment agents. How efficient the agents are, depends largely on how well they interact with the cement. Neutron scattering measurement allows studies of water and X-ray tomography allows us to study the structure of the solid material. With this combination we have a chance to validate other techniques - and thereby our product development strategy - which is exiting!

HOW THE WORK WAS DONE

Cement mortar with different moisture levels were studied at different temperatures in the D50 NeXT dual-modality neutron and x-ray tomography experimental station at Institut Laue Langevin (ILL), Grenoble. We were excellent supported by instrument scientist Nicolas Lenoir at ILL.

THE RESULTS AND EXPECTED IMPACT

The combination of x-ray and neutron imaging allowed separation of all phases in the samples (air-entrained cement mortar). An example is showed in Figure 1, where it is possible to distinguish between water-filled pores (left) and air-filled pores (right). With that it is possible to study each phase by itself which opens up to the opportunity to target on what that is most important in each and every case.

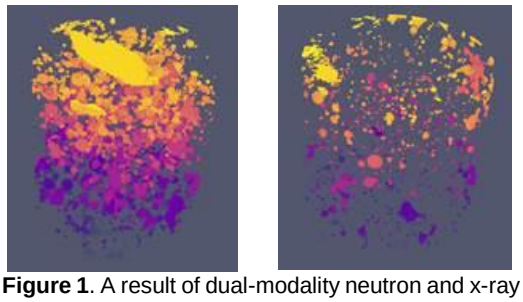

Figure 1. A result of dual-modality neutron and x-ray tomography, where the x-ray detects the structure of the cement mortar and the neutrons detect the water. Figure is showing: to the left all water-filled pores and to the right the air-filled pores.

During the freeze/thaw study it was possible to follow individual air voids and how their degree of saturation changed (Figure 2). The samples were exposed to temperature cycles from room temperature to -13ºC three times.

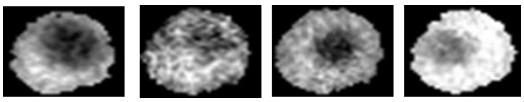

Figure 2. The same air void during four measurements at different temperatures: (1) room temperature, (2) -13ºC, (3) -13ºC at the second freezing cycle (4) after 2nd thawing. Notice the increased amount of water inside the pore. The grey scale indicates from white (water) to black (air).

This study show that this method can be a complement in future product development where we can study the interaction of cement and air entraining agents.

Contacts: Stefan Sandelin – Cementa AB, stefan.sandelin@cementa.se
Katja Frid – Division of Building Materials, Lund University, katja.frid@mau.se
Jonas Engqvist – Department of Construction Sciences, Lund University, jonas.engqvist.lth.se

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INDUSTRIAL PILOT PROJECT

Phase transitions of additively manufactured components investigated by *in situ* X-ray diffraction at high temperatures

THE INDUSTRIAL CHALLENGE

Höganäs AB is the world's largest producer of iron- and other metal-based powders for powders metallurgy but also has a focus on developing alloyed steel powder materials for Additive Manufacturing (AM). In order to make full use of AM techniques for making parts of complex geometries, one has to have a detailed knowledge of the influence on composition and microstructure during AM processing.

WHY USING A LARGE SCALE FACILITY

The goal of the project is to showcase how neutron- and synchrotron based techniques can assist industrial alloy development for additive manufacturing by allowing in situ analysis. The high intensity provided at e.g. the Petra III synchrotron in Hamburg, in combination with a large area detector, makes it possible to collect the time resolved data needed for the successful interpretation of these measurements. The time resolution needed is typically in the seconds range.. Furthermore, the neutron is unique in how it interacts with matter, which makes it possible to distinguish properties invisible to other probing tools, such as elements/ ions with similar or equal number of electrons.

HOW THE WORK WAS DONE

Structural changes as a function of temperature were investigated at the P02.1 beamline at Petra III, using a custom-built sample cell (λ = 0.207 Å). Samples were placed in single crystal sapphire capillaries and heated to 900 °C in Ar atmosphere using a Kanthal wire coiled around the capillary, followed by rapid cooling. (Figure1). Images of the scattered X-rays were recorded on a PerkinElmer XRD1621 fast area detector, and the resulting 2D images were azimuthally integrated to 1D diffraction patterns using the software Fit2D. Instrument peak shape parameters and zero-point error were obtained from refinements based on data from LaB_6. Neutron scattering at the powder diffractometer MEREDIT at the Neutron Physics Institute (NPI) in Rez, Czech Republic was used to obtain complementary crystallographic information.

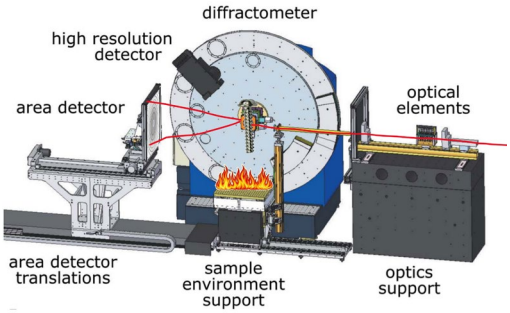

Figure.1 Experimental setup at P02.1. Image adapted from J. Synchrotron Rad. (2015). 22, 675–687.

THE RESULTS AND EXPECTED IMPACT

The results clearly show that in situ X-ray diffraction is an excellent tool to investigate phase transitions in AM-components. Figure 2 display the possibility to evaluate phase stability (composition, microstructure etc) as a function of temperature with high resolution and accuracy. The combination of neutron and X-ray scattering should be highlighted, where the X-rays provide excellent spatial and temporal resolution and neutrons complement with an average value over an entire component. These methods are a great complement to the in house capabilities at Höganäs.

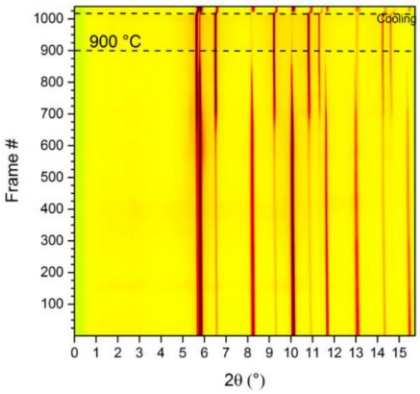

Figure2. An example of *in situ* X-ray diffraction as a function of temperature.

"It is clear that these measurements at large scale infrastructures provide a unique opportunity to understand structure-property relations. We will be back for more!" /Hilmar Vidarsson, Höganäs AB

Höganäs

UPPSALA UNIVERSITET

Contacts: Hilmar Vidarsson – Höganäs AB, Hilmar.Vidarsson@hoganas.com
Martin Sahlberg – Uppsala University, martin.sahlberg@kemi.uu.se

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INDUSTRIAL PILOT PROJECT

Neutron Bragg Edge Imaging of 3rd Generation AHS-Steel.

THE INDUSTRIAL CHALLENGE

The 3rd generation steel called Medium Manganese type is a variant of advanced high strength steels (AHSS) that shows more attractive forming properties compared to presently used steel types. The major phase constituents in this steel product are martensite and retained austenite, and a fine tuning of the ratio can result in an attractive combination of high strength and good ductility which is of interest for e.g., the automotive sector. According to the transformation induced plasticity (TRIP) mechanism, austenite transforms to "fresh" martensite upon deformation such as cold forming. The challenge is to control the stability of the austenite to make it transform at the right stress/strain in the metal forming step.

WHY USING A LARGE-SCALE FACILITY?

The phase fraction of austenite can be analysed using laboratory methods such as X-ray diffraction (XRD) and EBSD, but these methods can only be performed on limited areas or volumes. Understanding how the austenite fraction is dependent on the deformation, requires knowledge of the phase fraction on a macroscopic scale. With XRD or EBSD, this requires many individual measurements at different locations on for example a tensile test sample. Using the imaging capabilities at a neutron scattering beamline and the technique referred to as Bragg Edge Imaging, spatially resolved images of the said phase transformation due to deformation, can be made on a macroscopic scale. The time resolution of neutron sources also opens up for in-situ studies during deformation, for example by tensile testing or forming operations.

HOW THE WORK WAS DONE

Tensile test specimens, 90mm in length, were prepared from the investigated material and drawn to four different deformation levels. These samples went through preparatory measurements at the Conrad instrument at BERII in Germany and were further imaged using the Bragg edge technique at both the Raden instrument station at the J-Parc neutron spallation source in Japan and on the IMAT instrument at ISIS UK. Dr Takenao Shinohara and Dr Winfried Kockelmann are acknowledged for the remotely performed measurements due to the travel restrictions. As validation also quantification with XRD was performed at five different locations from the center of each sample cross-section

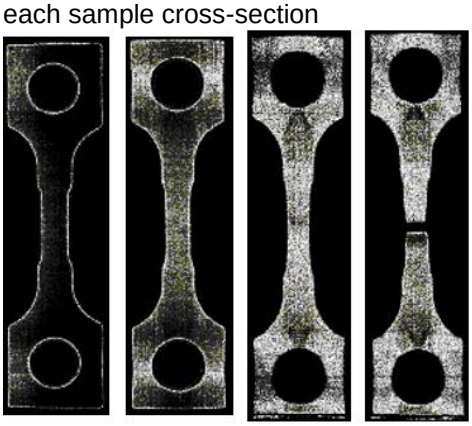

Figure. From left to right: an increased degree of deformation is followed by a larger amount of martensite (brighter regions).

THE RESULTS AND EXPECTED IMPACT

The images computed from neutron Bragg edge data showed the profile of the austenite and martensite phases. At the different stages in the deformation curve an increased signal of the martensitic content could be seen, see Figure, where the brighter regions indicate a larger amount of martensite following the degree of deformation. XRD data confirmed the neutron findings. This project has introduced SSAB to a new type of characterization technique that allows for both larger sample sizes and an in-situ approach to follow phase transformations in the specimen during deformation. This allows for a better understanding of phenomenon's occurring in the material also under other types of loads than deformation.

"This project has opened our eyes to the possibilities made available through the use of neutron sources and Bragg edge imagery in characterizing material and the present investigation of the TRIP effect can successfully be followed using this technique."/ Erik Nymann, SSAB EMEA AB

Contacts: Erik Nymann, SSAB EMEA AB erik.nymann@ssab.com
Fredrik Lindberg, Swerim AB fredrik.lindberg@swerim.se
Robin Woracek, European Spallation Source ERIC robin.woracek@ess.eu

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INDUSTRIAL PILOT PROJECT

Investigating strain age cracking in precipitate hardening nickel-based alloys using in-situ diffraction and tomography

THE INDUSTRIAL CHALLENGE

The next generation precipitate hardening nickel-based superalloys will be used for producing more efficient and lighter jet engines. This will not only reduce the fuel consumption but the amount of NOx emission as well. However, strain age cracking (SAC) caused by heat treatments or reheating in multi-pass welds is of major concern when designing high-performance components from precipitate hardening nickel-based alloys. The problem becomes even more pronounced for alloys that precipitate rapidly and have an elevated volume fraction of precipitates. Some alloys are even treated as “non-weldable”. Hence, it is of high interest to increase knowledge and improve lifetime predictions for components susceptible to strain age cracking.

WHY USING A LARGE SCALE FACILITY

Strain age cracking is a phenomenon that appears where multiple length scales are active simultaneously at elevated temperatures, however the main mechanism is not known. The capacity of synchrotron facilities where it is possible to perform high-temperature in-situ experiments that can be analyzed for multiple length scales is vital in increasing the understanding for the phenomenon. These kinds of multiple length scale in-situ experiments are hard, or if possible, at all, to do using traditional investigation techniques such as SEM or TEM.

HOW THE WORK WAS DONE

Experiments designed to understand the strain age cracking mechanisms were done at the Swedish materials science beamline, P21.2, at the Petra III synchrotron, Germany. Small Angle X-ray Scattering (SAXS), 3D X-ray Diffraction (3DXRD), and tomography were utilized in the experiments during the in-situ heating and loading of the sample using a for the project developed compact load frame. This setup covers length scales from the nm-range to the mm-range. From the results, it is possible to study the misfit strains that occur during the precipitation, but also the change in stiffness of the grains because of heat treatments. The scripting to perform the analysis of the tomography data was done by Emanuel Larsson, Lund University, and the 3DXRD analysis by Johan Hektor, Malmö University.

THE RESULTS AND EXPECTED IMPACT

The conclusions drawn from the SAXS, 3DXRD and tomography data are that an increase in grain stiffness because of precipitation is not an alone mechanism for SAC to occur. Rather it is suggested that several mechanisms at various length scales are responsible for the phenomena.

Prior to finding a suitable sample that ends within a good signal for 3DXRD analysis, numerous manufacturing techniques, and sample preparation techniques were tested out before a good signal was achieved. This means that the deformation of grains from a turning operation is detrimental for the single-crystal diffraction needed in the 3DXRD analysis. The experimental results will serve as support in a larger campaign where the aim is to understand the SAC phenomenon using a combination of experiments and modeling efforts by, e.g. crystal plasticity modeling.

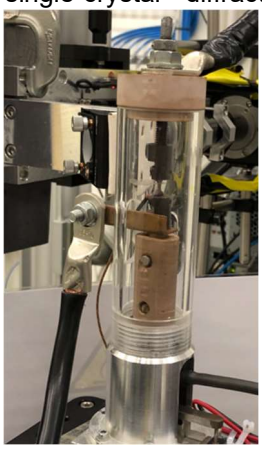

Figure. Compact load frame used to for the in-situ loading and heating 3DXRD, SAXS and tomography experiments. Joule heating are utilized for heating the sample.

“This project has increased the overall knowledge about synchrotron facilities and its possibilities to perform chall-enging in-situ experiments at various length scales” /Ceena Joseph, Materials Application Engineer, GKN Aerospace Sweden AB

GKN AEROSPACE

MALMÖ UNIVERSITY

Contacts: Ceena Joseph – GKN Aerospace, ceena.joseph@gknaerospace.com
Martin Fisk – Malmö University, martin.fisk@mau.se

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INDUSTRIAL PILOT PROJECT

Exploring Rheo-SANS for deducing structure and orientation of particles in paints under flow

THE INDUSTRIAL CHALLENGE

Linking the nano-scale structure to the macroscopic flow behavior is believed to be an important guide in the development of coatings for specific applications, for example when producing acoustic surfaces. For Saint-Gobain Ecophon there is therefore a need for industrially useful advanced tools for characterizing thixotropic industrial paint systems.

WHY USING A LARGE SCALE FACILITY

Small angle neutron scattering (SANS) allows for structure characterization on the nano-scale, 1 nm - 1 µm. Due to the isotope dependence of neutron scattering it is possible to effectively hide or highlight certain parts of an aqueous formulation, such as a water-based paint, by partially exchanging the H2O with D2O. At only a few neutron facilities, a combination of SANS experiments and rheological measurements is available (Rheo-SANS), enabling information gathering on nano-scale structures formed in the paint under flow.

HOW THE WORK WAS DONE

A model system of an industrial paint with relevant rheological behavior was investigated by Rheo-SANS at the SANS1 beamline at the Swiss neutron spallation source (SINQ), Switzerland, with assistance of instrument scientist Joachim Kohlbrecher.

Figure 1. Rheo-SANS experiment with a white paint under shear in the central upright standing cylinder. The neutron beam comes from the right side through the red and blue colored apperature and continues through the sample. Scattered neutrons pass the left circular window before detection by a 2D detector.

A stress-controlled rheometer was placed in the neutron beam on a movable table, enabling scattering measurements at different positions of the sheared sample. The H2O/D2O composition of the samples were chosen to highlight the scattering signal from the latex binder.

THE RESULTS AND EXPECTED IMPACT

At applied continuous shear, a weak anisotropic scattering pattern was visible. Figure 2 shows the angular dependence of the scattered intensity in the velocity-vorticity plane.

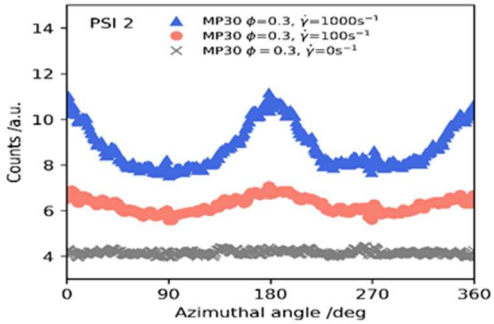

Figure 2. Ordering of particles in the paint was demonstrated as an anisotropic scattering pattern. The graph shows the radially integrated scattering intensity as a function of azimuthal angle.

This shows that the applied shear induces an anisotropy in the binder particles of the model paint on the 100 nm length scale. The specific ordering of the binder particles has not yet been possible to deduce, but the extension of binder material occurs along the flow direction. Upon the cessation of shear, the anisotropy is gone within a single frame of a scattering experiment (30 s). It became clear that previously non determined parameters play a large role in the collective rheological behaviour of the paints.

A specific set of measurements were chosen to be implemented in future paint characterization. When the data have been fully analysed the results will be disseminated inside Saint-Gobain Ecophon, which can lead to continued use to develop new sustainable building materials.

Contacts: Johan Borgström – Saint-Gobain Ecophon, johan.borgstrom@ecophon.se
Ulf Olsson – Lund University, ulf.olsson@fkem1.lu.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Exploring the plasticity of CVD Al_2O_3/TiCN multilayer coatings during nanoindentation

THE INDUSTRIAL CHALLENGE

Growing demands for high production rates in the metal cutting industry requires the development of wear resistant cutting tool and coating materials. Possibilities to conduct a detailed in-situ characterization of the mechanical behaviour and structural evolution of thin coating layers during load would allow Seco to obtain deeper knowledge that can be used to design and optimize coating materials with enhanced properties to achieve a predictable performance in metal machining.

WHY USING A LARGE SCALE FACILITY

Characterization methods with a good spatial and temporal resolution are required to resolve local changes of the mechanical state of thin coating layers in situ.

HOW THE WORK WAS DONE

Thin lamella samples of CVD Al_2O_3/Ti(C,N) coated carbide tools with approximate thicknesses around 300 μm were cut from cutting tool inserts by a low speed diamond saw. The specimens were further thinned down to a "lamella" with a thickness of around 40 μm using FEI Versa 3D focused ion beam-scanning electron microscope (FIB SEM) at Chalmers University.

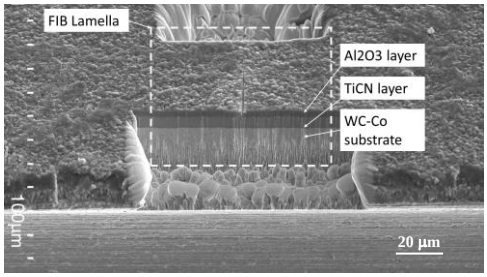

Figure 1. FIB-SEM cross section of the CVD Al_2O_3/Ti(C,N) coated tool.

The coating in the remaining 40 μm wide lamella was indented from the top using a 50 μm long diamond wedge (wider than the lamella width) nano indenter aligned with the X-ray beam of the NanoMAX beamline of MAX IV. Three lamellae with different crystal orientation of the Al_2O_3 layer were subjected to controlled loading conditions of wedge indentation (up to 0.3 N) while synchrotron X-ray nano diffraction data was collected at high spatial resolution (down to 100 nm scale) in an area of approximately 7x7 μm^2. Diffraction patterns were recorded in transmission using the Pilatus 1M detector at a photon energy of 14 keV. During acquisition of each map, the applied load was held constant (force control mode). Maps were acquired at different load levels as well as before and after indentation.

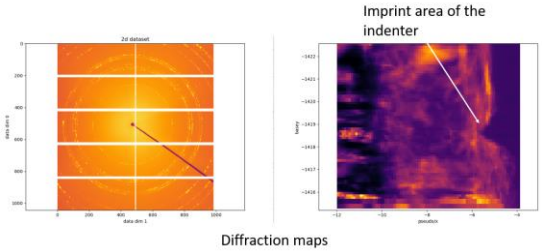

Figure 2. Typical diffraction patterns obtained in the indented CVD coating.

THE RESULTS AND EXPECTED IMPACT

The set-up allowed the evolution of elastic strains and related internal stresses in the individual coating layers to be determined with a reasonably good accuracy and a dependence of the elastic strain/stress on crystal orientation and applied load could be established. Complementary studies of the indented coating specimens were also initiated at Lund University to study the structural changes in the layers with high resolution transmission electron microscopy (TEM). This combined knowledge will be used for designing future experiments to further explore the coating material behaviour during indentation for other coating architecture, compositions, and surface treatment in the extent to optimize the coating materials for the cutting tool.

Contacts: Rachid M'Saoubi – Seco Tools, rachid.msaoubi@secotools.com
Filip Lenrick – Lund University, filip.lenrick@iprod.lth.se

Vinnova's project No: 2019-02590 **Duration:** Aug 2019 -- Apr 2022

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INDUSTRIAL PILOT PROJECT

Micromechanical response of nitrocarburized steel studied by in-situ X-ray microdiffraction during nanoindentation

THE INDUSTRIAL CHALLENGE

Steel nitrocarburizing is a surface treatment process that increases fatigue, wear, friction and corrosion properties. For example, is it used by project partner Volvo Trucks on various engine components. The process produces a complex surface compound layer, which consists of two iron nitrides ε and γ'. Moreover, it can be combined with post oxidation to yield a few microns of oxide to further improve corrosion properties. However, a challenge is to understand the micromechanics of this microstructure and thereby tailor the process for performance.

WHY USING A LARGE SCALE FACILITY

X-ray diffraction (XRD) is a direct probe to study the local crystalline deformation and explore micromechanics. At synchrotron radiation sources, the high flux and available micro/nanofocus X-ray beamsize make the scanning diffraction technique feasible tool to map multiscale structures of metal from um to Ångstrom in minutes to hours. This is unlikely to be accomplished by any inhouse X-ray source.

HOW THE WORK WAS DONE

The micro-/nanofocused X-ray diffraction was performed at the P03 'MiNaXs' beamline at PETRA III in Hamburg, where a nanoindentation setup is available at the Nanofocus end-station operated by Helmholtz-Zentrum Hereon. By using a beamsize of around 1 µm, it is possible to map the indentation deformed region at the step of the beamsize. The samples were cut from nitride steel bars and subsequently milled with sinker electrical discharge machining and finally polished by focused ion beam. This made a final thickness of approx. 50 µm, with parallel surfaces for incident and exiting beam.

THE RESULTS AND EXPECTED IMPACT

Via microfocused X-ray diffraction, a set of peaks intensity allows to identify crystalline phases and map its spatial distribution. After each indentation step, the shift of the XRD peak position could be used to analyze strain distribution with regards to scanning position, indentation load and diffraction orientation. In the figure an SEM micrograph of a FIB-milled section has been overlayed by this strain field analysis, together with a schematic of the indentation tip. This spatial resolved data unravels the micromechanical response of compound layer during loading.

In total three different steel grades were heat treated by Bodycote using two different nitriding recipes. By characterizing the compound layers formed and their micromechanical response we aim at establishing more detailed requirements on microstructure for optimum performance. In addition, the project developed a data-pipeline for onsite data processing and visualization, which enable prompt data evaluation and rapid experiment improvement.

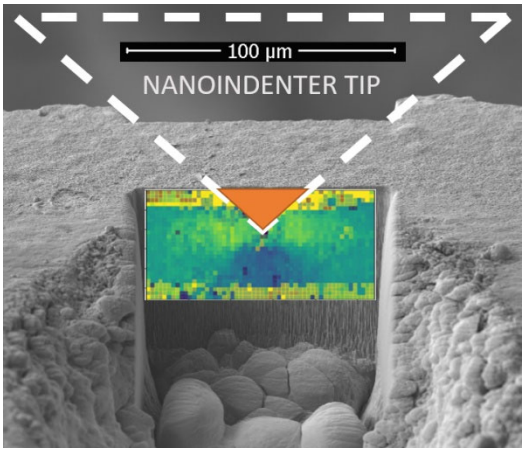

Figure. Strain field analysis overlayed on a SEM micrograph of the FIB-milled section.

"We see an opportunity for tailor-made nitrocarburizing to replace hard chrome in tough applications" /Erik Spolander, Bodycote

Contacts: Erik Spolander – Bodycote, erik.spolander@bodycote.com
Shun Yu – RISE, shun.yu@ri.se, Albin Stormvinter – RISE, albin.stormvinter@ri.se

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INDUSTRIAL PILOT PROJECT

Utilisation of synchrotron based in-situ XRD and CT to study the effect of the processing gas in laser powder bed fusion

THE INDUSTRIAL CHALLENGE

Recent studies indicate that the use of Helium (He) and its mixtures as processing gas in laser powder bed fusion (LPBF) improve process stability and reduce generation of the process by-products (spatters), which are precursors for defects in LPBF-processed components and powder degradation. Still, the understanding of how He influences the material solidification and the resulting component properties are limited and hinder the development and implementation of gas solutions.

WHY USING A LARGE SCALE FACILITY

Effect of the process gas on the LPBF process stability can be depicted following the kinetics of the phase transformation by X-ray diffraction (XRD) during solidification of the micron-sized melt pool, that is characterised by very high cooling rates (10^4-10^6 K/s). Such analysis could only be performed at synchrotrons, which allows utilising of high flux of the radiation source and ultra-fast detectors, together with the *miniSLM* test rig designed by Paul Scherrer Institute (PSI). The use of synchrotron X-ray computed tomography (SXCT) allows to distinguish porosity distribution in as-printed samples at high resolution as well as measure residual stresses.

HOW THE WORK WAS DONE

The experiments at PSI were conducted at the MicroXAS beamline of the Swiss Light Source synchrotron, using the *MiniSLM* rig designed PSI to enable in situ XRD. The system was used in reflection mode with a 12 keV beam energy. Gases including Ar, He and their mixtures were studied. The SXCT work on LPBF fabricated samples was associated with the Federal Institute for Materials Research and Testing (BAM) and performed at the BAMline at BESSY II synchrotron in Berlin. A monochromatic X-ray beam energy of 45 keV allowed a pixel size of 0.88 µm.

THE RESULTS AND EXPECTED IMPACT

The in-situ XRD measurements performed using the *miniSLM* rig highlighted that utilisation of He allows to reach improved process stability in comparison to the standard Ar as well as slightly higher cooling rates. Decreased spatter generation under He was detected as well, resulting in the improved powder reusability. These results have an important impact on the understanding of the effect of He on the process and further development of the gas solutions at Linde.

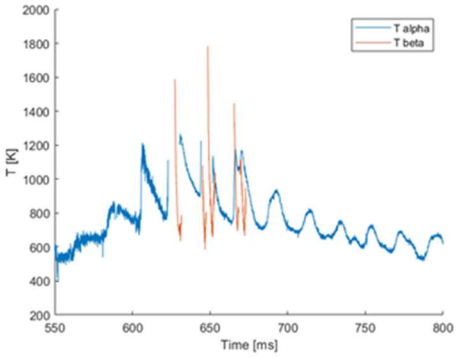

Figure 1. Temperature profile reconstructed from the X-ray diffraction signal collected during printing.

The SXCT measurements showed that application of He does not result in additional residual stresses in the built part and pore distribution and characteristics are comparable between different gases, see Fig.2.

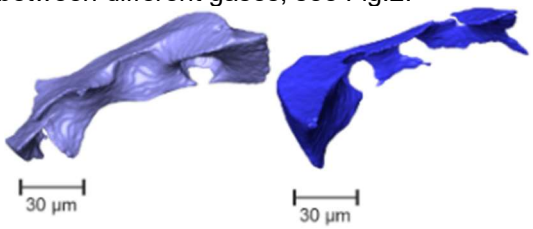

Figure 2. Reconstructed porosity from the SXCT (performed at BAM by T. Mishurova et al.).

In all, the results have permitted Linde to discover current cutting-edge possibilities of monitoring and characterization of both the LPBF process and the produced component at large-scale facilities. In addition, the collaborations led to initiation of several new projects between the PSI, BAM and Chalmers, allowing the Swedish additive manufacturing competence centre CAM² and its industrial partners to strengthen their research activities in that regard.

Contacts: B. Williamsson – Linde Gas AB, bo.williamsson@linde.com
P. Forêt, S. Dubiez-LeGoff –Linde Gmbh, pierre.foret@linde.com, sophie.dubiez-le.goff@linde.com
C.Pauzon, E.Hryha -Chalmers University of Technology, pauzon@chalmers.se, hryha@chalmers.se

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INDUSTRIAL PILOT PROJECT

Non-destructive residual stress analysis on thick welds of Ti-6Al-4V plates for aerospace using neutron diffraction

THE INDUSTRIAL CHALLENGE

The aerospace industry tends to replace large casted structures with smaller free form sections which is assembled with welding. This approach increases the flexibility in manufacturing and reduce costs. However, it may introduce unfavourable residual stresses that could cause geometric distortions and out of tolerance problems. In order to supress the welding induced stresses, it is important to adjust the welding process. This can be accomplished using finite element (FE) simulation, but these numerical tools need to be experimentally verified.

WHY USING A LARGE SCALE FACILITY

The accuracy of FE-simulations can be verified in the surface region with X-ray diffraction measurements and deeper internal stresses by the contour method. X-ray diffraction measure the strains within the crystalline lattice while the contour method measure the relaxation of strains when the part is sectioned. Both methods are however destructive and in order to verify internal stresses of thick components non-destructive neutron diffraction (ND) is the only possibility.

HOW THE WORK WAS DONE

The project focused on the Electron Beam welding process. A sample was produced, with the dimension 400x100x55 mm. The sample was further prepared for ND by sectioning in order to access the stains in the centre of the sample which on the same time gave the input data to the contour measurement. The stresses were characterised with conventional lab-XRD and the Contour method. The neutron diffraction measurements were done remote at the beamline Engin-X at the ISIS Neutron and Muon Source in England. Beamline scientist Saurabh Kabra is acknowledged for the support.

THE RESULTS AND EXPECTED IMPACT

The selected samples size was really pushing the limit for ND but in discussion with the beamline we planned for strategies of how to as assess the most possible data. Even though these strategies were employed, the results showed that the sample thickness limited the amount of data that could be retrieved for this sample thickness. Unfortunately, a smaller sample geometry could not be used due to the welding process and it was not possible to make the sample smaller before measurements since this will influence on the internal stress state. The retrieved strain data was compared to FE-simulation and the other measurements, lab-XRD and contour method. The ND data showed similar strain-profiles but a large difference in magnitude compared to the strains measured with the contour method. Instead, the residual stresses measured with the contour method verified the FE-simulation and showed high correlation.

The project did verify the FE-simulation but could not retrieve enough strain data with ND to calculate the stresses which requires strain data in all three directions for each position in the sample. The project has gained important knowledge when planning for future ND studies where the sample size needs to be designed taking the maximum neutron traveling distance in mind. We also plan to perform future measurement using neutron imaging at ISIS which is a technique that measured large areas in the sample with very high spatial resolution.

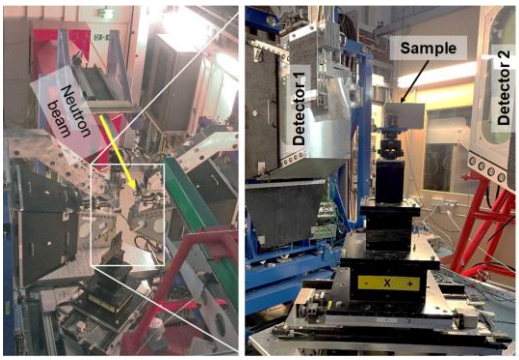

Figure. Sample setup at Engin-X showing the neutron beam entrance from the cut cross section setup (left)and the xyx-stage holding the sample between the two detectors.

Contacts: Joachim Nordenström-Steffenburg–GKN Aerospace AB, Joachim.steffenburg-nordenstrom@gknaerospace.com
Jonas Holmberg – RISE AB, jonas.holmberg@ri.se
Tuerdi Maimaitiyili – Swerim AB, tuerdi.maimaitiyili@swerim.com

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INDUSTRIAL PILOT PROJECT

Verification of cleaning of pores and surfaces using Ultra-pure water using coherent X-ray scattering (CoSAXS) at MAX IV

THE INDUSTRIAL CHALLENGE

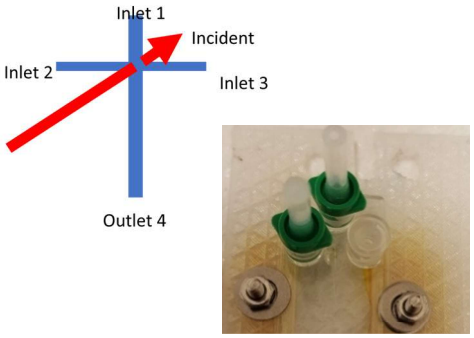
Every year billions of kg of detergents and cleaning agents are produced and used globally. Unfortunately, a large part ends up in the aquatic system and accumulate in living organisms. The Ultra-Pure DIRO-water produced by SWATAB has been showed to have the ability to clean without the need for chemicals and also at room temperature. The mechanism how different types of water can lift, and transport particles is not fully understood but crucial to optimize the process of cleaning without chemicals.

WHY USING A LARGE SCALE FACILITY

Lab-based methods have shown that particles and oil is dispersed readily in DIRO-water but was unable to show the release of particles from pores. In order to mimic the process by which water can release dirt particles from pores or surfaces, a small angle X-ray scattering experiment was designed to track the dispersion by water of nanoparticles from a pore. The CoSAXS technique allows the quantification of the size and distribution of nanoparticles within a scattering volume.

HOW THE WORK WAS DONE

A small angle X-ray experiment was carried out at the CoSAXS beamline of the MAX IV synchrotron by SWATAB and the University of Lund in strong collaboration with the beamline personnel at MAX IV and Malmö University. A thermoplastic (COC) micro-fluidic chip (ChipShop) with a crossed slot design was installed within a 3D printed plastic holder on the motorized sample stage at the sample position. The channel width and depth were 200 x 200 nm. The microfluidic chip was positioned such that the X-ray beam was incident in the centre of the cross (see illustration). The particles was 22 nm.



THE RESULTS AND EXPECTED IMPACT

The experiment gave us the opportunity to actually for the first time be able to see what happens in real time when DIRO-water hits the soil, lift it and transport it away. We were also able to compare it with other type of pure water as well as a salt solution to mimic tap water. DIRO-water was able to extract the nanoparticles from the pore in a continuous manner whereas de-ionised (milli-q) water showed a variation in the extraction rate. The DIRO-water caused no degradation to the nanoparticles as the dimension of the nanoparticles did not change. The results show that DIRO-water can indeed enhance the dispersion of nanoparticles from a pore, when compared to de-ionised water (milli-q) or tap water.

This experiment has already lead to more research and these results will be presented in articles during the summer.
The investigation highlighted that different methods to produce pure water are not equivalent, supporting observations by SWATAB and others. This will reduce or eliminate the need for detergents and surfactants generating a positive impact for both nature and man.

"This project show the importance of collaboration between state of the art synchrotron-techniques, science and the industry" / Mats Marklund, SWATAB

Contacts: Per Hansson – Scandinavian Water Technology AB, per@swatab.com
Ann Terry – MAX IV (CoSAXS beamline), ann.terry@maxiv.lu.se

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INDUSTRIAL PILOT PROJECT

In-situ X-ray diffraction of additive manufacturing to aid in tool steel grade development

THE INDUSTRIAL CHALLENGE

A major challenge in the design of new martensitic tool steel grades for producing hot-work tools by additive manufacturing (AM) by laser-powder bed fusion (L-PBF) is to overcome their crack susceptibility. The L-PBF process results in a complex thermal history as the laser repeatedly melts the surface and heats the adjacent material. The hot-work tool steels undergo phase transformations both during solidification and in the solid-state as martensite forms. It is a real challenge to deconvolute the microstructure and stress evolution necessary to understand how to improve the material and its printability.

WHY USE A LARGE-SCALE FACILITY?

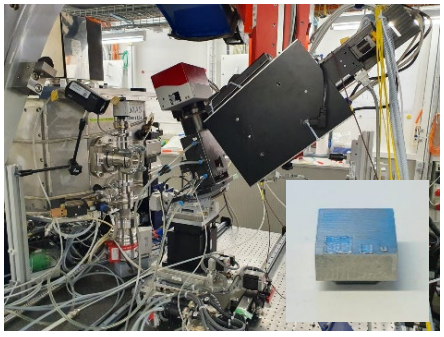
using conventional lab-based X-ray diffraction (XRD) techniques after the process since the thermal history is unknown. The high intensity and spatial resolution of synchrotron sources, combined with ultrafast detectors, allows X-ray diffraction (XRD) studies of how the material evolves during the printing process. In this manner, the phase and stress evolution can be captured during the process and compared to the thermal history.

HOW THE WORK WAS DONE

At the Paul Scherrer institute a research group have constructed a miniaturized L-PBF printer to be used at the MicroXAS beamline of the Swiss Light Source (SLS). This allows real-time diffraction studies while building small structures. The focused X-ray beam with low energy (3-23 keV) makes it possible to perform diffraction measurements of a fixed volume, smaller than a melt pool, as the laser scans across the surface. In this way, one can monitor as the material is heated and melted by the laser, solidified, and eventually transformed to martensite. It is further possible to see the influence of heat flow as the laser scans adjacent areas during the build. The ultrafast detectors with acquisition rates of up to 40 kHz permit sufficient time resolution to capture the process. Due to the travel restrictions during the pandemic, the experiments were kindly conducted by Steven Van Petegem at PSI.

THE RESULTS AND EXPECTED IMPACT

An example of the small structures built, and the information acquired during the build can be seen below.



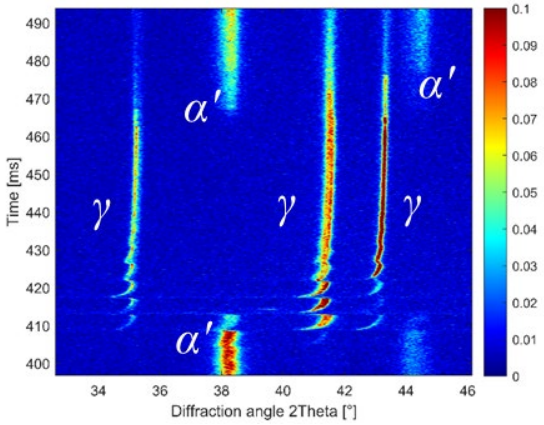


Figure a) The miniSLM mounted at MicroXAS, and small printed structures (4x4, 2x2 and 1x1 mm^2). **b)** Phase evolution (including heating, melting, solidification of γ austenite and later α' martensite formation) in the 2x2 mm^2 structure as the laser is scanning across one layer. Each laser scan is completed in ~4 ms, and the bending of the X-ray lines is due to the changing temperature.

The measurements have resulted in novel information about the local thermal history and resulting microstructure evolution during the L-PBF process for these types of steels. The information gives clues to the continued development work at Uddeholm.

Contacts: Christos Oikonomou – Uddeholm, christos.oikonomou@uddeholm.com
Niklas Holländer Pettersson– Royal Institute of Technology, KTH, nikpet@kth.se
Greta Lindwall – Royal Institute of Technology, KTH, gretal@kth.se

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INDUSTRIAL PILOT PROJECT

In-situ photoelectron microscopy study of σ-phase growth in DSS

THE INDUSTRIAL CHALLENGE

Outokumpu Stainless and Sandvik Materials Technology are the global producers of advanced steels, including duplex stainless steels (DSS). It is of great importance to control and predict intermetallic phase formation and homogenisation for producing DSSs with long lifetime and better properties. Formation of σ-phase in DSSs at these elevated temperatures causes segregation of alloying elements which can lead to deterioration of steel properties even at low fractions of σ-phase. Numerical models have been developed to describe the phenomenon, but lack of experimental verification hinders their application.

WHY USING A LARGE SCALE FACILITY

Laboratory-based electron microscopy is often used for ex-situ studies, e.g., to illustrate σ-phase after performed annealing. Studying of chemical and structural changes accompanying σ-phase formation, however, requires a very high spatial and good temporal resolution as well as capability to mimic the process of annealing while simultaneously measuring. Such performance and equipment for in-situ heating requires synchrotron light sources.

HOW THE WORK WAS DONE

Samples of 2507 DSS were polished down to 0.25 um and studied in Spectroscopic PhotoElectron and Low Energy Electron Microscope (SPELEEM) at the MAXPEEM beamline of MAX IV. The in-situ annealing and characterisation was performed in the T range 600 – 1000°C.

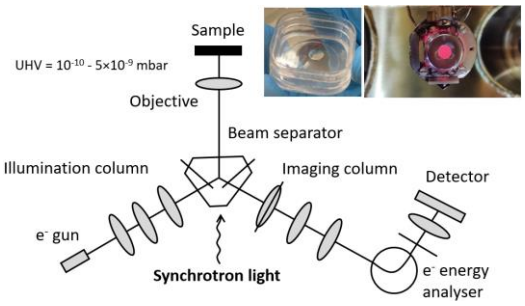

Figure 1. Schematic diagram for SPELEEM. The sample can be illuminated with low energy electrons or photons. Sample in the sample box and mounted in the cartridge during heating is shown. Dr. A. Zakharov and Dr. Lin Zhu from MAX IV operated the instrument.

THE RESULTS AND EXPECTED IMPACT

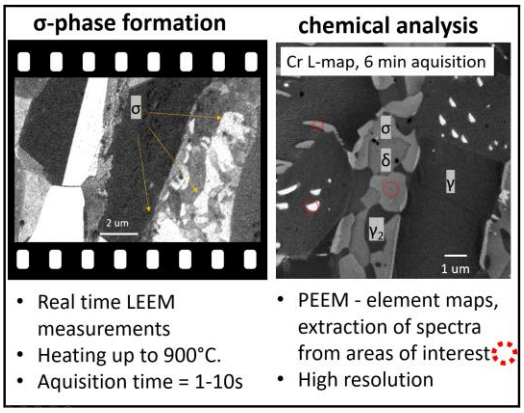

Figure. 2 Left: LEEM image recorded at 786°C. Right: Cr PEEM map.

Growth of σ-phase was observed in-situ in real time with low-energy electron microscopy (LEEM) (Fig 2, Left). Chemical analysis with photoemission electron microscopy (PEEM) allowed to record high-quality element maps at the Cr L-, Fe L-, Ni L-, Mn L-, Mo M2-, N K-, and O K- absorption edges, to identify chemical state of species, and to obtain element profiles – all in one dataset (Fig. 2, Right). The high surface sensitivity of PEEM and LEEM also allowed to observe surface segregation of active elements during annealing. The fact that the instrument can operate only at high vacuum conditions, however resulted in formation of larger number of precipitates than expected at true process conditions in air. This complicated the homogenisation of σ-phase and comparison with computational models. Information on σ-phase nucleation, surface chemistry, and precipitate growth during in-situ annealing was, however, never reported before and additional in-depth analysis of data is planned.

"Results indicate that precipitation on a surface can be examined. It may become a useful tool to study precipitation at grain boundaries which could be very beneficial."
/J. Y. Jonsson, Outokumpu

SWERIM SANDVIK outokumpu

Contacts: J. Y Jonsson – Outokumpu Stainless, jan.y.jonsson@outokumpu.com
U. Borggren – Sandvik Materials Technology, ulrika.borggren@sandvik.com
K. Simonov – Swerim AB, konstantin.simonov@swerim.se

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INDUSTRIAL PILOT PROJECT

Synchrotron SRµCT and XRD investigations of crack mechanisms in parts produced by laser powder bed fusion additive manufacturing

THE INDUSTRIAL CHALLENGE

Porosity is a critical issue in additive manufacturing (AM) of metals. By modifying the process parameters, the density of printed parts can be varied. However, in certain alloy systems, higher density increases cracking. Low levels of porosity important for good properties but the presence of crack is detrimental. It is therefore important to understand why and when in the process chain these cracks form in order to produce high quality parts.

WHY USING A LARGE SCALE FACILITY

Previous microstructure studies using light optical microscopy have indicated a higher susceptibility to cracking in high-density parts. However, for volumetric quantification of the pores and cracks, SRµCT-data is required. Compared to light optical microscopy, synchrotron radiation sources with high flux and brilliance enables rapid assessment of large volumes, while providing comparable resolutions.

By using synchrotron based XRD, information can be collected from a volume instead of just the surface as in laboratory XRD. This is crucial for an accurate correlation of residual strain with the porosity measurements.

HOW THE WORK WAS DONE

The experiments were performed at the Swedish Materials Science (SMS) beamline P21 by Ulrich Lienert at the Petra III synchrotron in Hamburg, which offers the combination of XRD and SRµCT. In order to investigate the effect of porosity levels on crack initiation and propagation in AM parts, 3x4x10mm samples with various porosity levels was analysed (as-built samples with different densities from 97.7% to >99.9%) as well as stress relived samples and samples still attached to the build plate. The six samples required 8 hours of beamtime for the XRD analysis.

THE RESULTS AND EXPECTED IMPACT

Clear increase and stronger variation in strain was seen as the porosity decreased while samples still attached to the build plate had a different strain profile. After stress relieving, a low and uniform strain profile was found. Together with the SRµCT-data it is possible to better understand the link between porosity, residual strain and strain relaxations due to cracking. However, more work using this methodology is needed to fully understand this link.

With the higher richness of detail and the volumetric data that the large-scale facilities offer, Kanthal gets a new powerful tool for solving future challenges in AM. Rapid implementation of this technology in industry for similar investigations is possible through the methodology developed in the project.

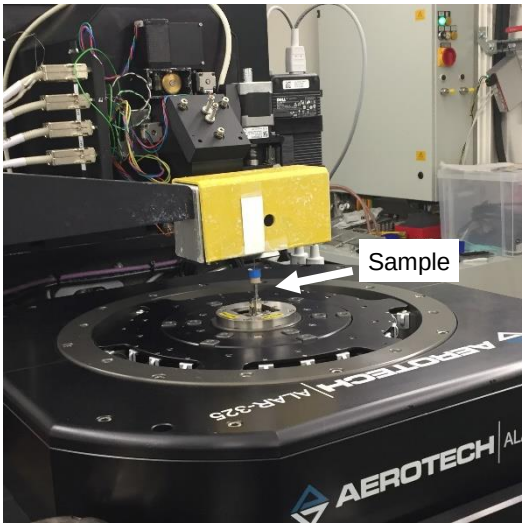

Figure. Experimental setup of the SRµCT.

Contacts: Roger Berglund – Kanthal AB, roger.berglund@kanthal.com
Emil Strandh – Swerim AB, emil.strandh@swerim.se

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INDUSTRIAL PILOT PROJECT

Using synchrotron radiation to study TRIP effect in AHSS

THE INDUSTRIAL CHALLENGE

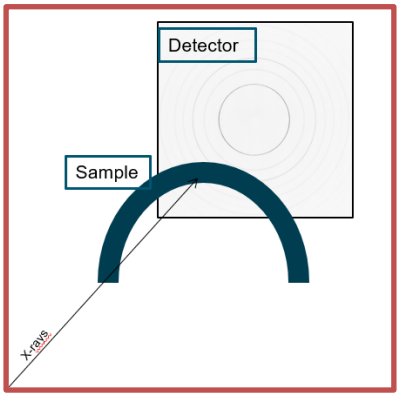
The 3rd generation advanced high strength steels (AHSS) in which major constituents are the steel phases martensite and austenite, show an attractive combination of high strength and good ductility. These properties are beneficial especially for the automotive sector and are achieved by the TRIP (transformation induced plasticity) mechanism when austenite transforms to "fresh" martensite upon deformation. The challenge of developing these steels is to control the stability of the austenite to make it transform at the right stress/strain.

WHY USING A LARGE-SCALE FACILITY?

Both the phase fraction of austenite and the residual stress can be analysed using laboratory methods but not at the required spatial resolution or with the geometrical aspects required in this specific case. With a synchrotron source, it is possible to map the material with a resolution of 50 µm in transmission mode. This could result in a new method for simultaneous mapping of the strain and stress, as well as the amount of transformed austenite.

HOW THE WORK WAS DONE

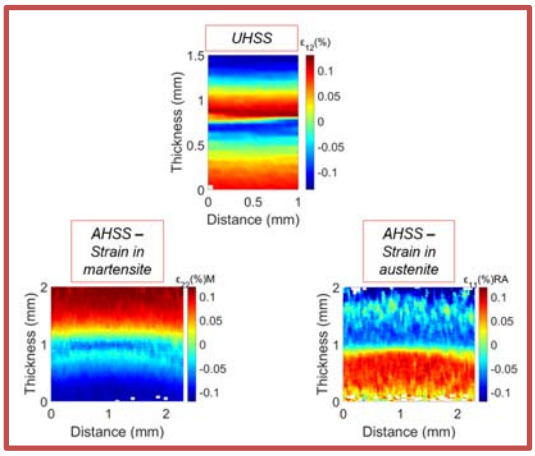
The matrix consisted of two different 3rd generation steels with different austenite contents, as well as an ultra-high strength, fully martensitic steel (UHSS) for reference. Synchrotron high energy X-ray diffraction measurements were performed in transmission mode on thin sections of bent and undeformed steel sheet (see illustration) using the P21.2 experimental station of Petra III in Hamburg.



THE RESULTS AND EXPECTED IMPACT

From the X-ray diffraction data, it was possible to extract both macro and micro strain introduced by bending as well as the austenite fraction.

An interesting finding was the effect of spring-back on the residual stresses in the AHSS. In the single-phase UHSS material, the spring-back after bending leads to compressive strains and stresses on the outside of the bend and tensile stresses on the inside. This is indicated by, respectively, blue and red color in the strain map bellow. The 3rd generation AHSS material show the opposite behavior of the martensite (majority) phase, tensile strain on the outside and compressive strains on the inside of the bend. This while the remaining austenite is affected by the spring-back as expected.



These findings open-up several interesting questions both in fundamental aspects and for the application of 3rd gen AHSS. What is the microstructural basis of the strain behaviour, and how will the strain behaviour of the two phases affect the material's properties in case of for example fatigue and hydrogen embrittlement?

"A strong in-house knowledge of synchrotron techniques gives SSAB a stronger ability to refine materials and properties"/ Sven Erik Hörnström,SSAB EMEA AB

Contacts: Sven Erik Hörnström, SSAB EMEA AB, sven-erik.hornstrom@ssab.com
Fredrik Lindberg, Lena Ryde,Johannes Brask,Shirin Nouhi – Swerim AB, fredrik.lindberg@swerim.se

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INDUSTRIAL PILOT PROJECT

Diffraction studies of precipitate development of Nb/TiNb microalloyed steels aiming for analysis during hot rolling /cooling

THE INDUSTRIAL CHALLENGE

SSAB is a highly specialized steel company and a producer of HSLA steels. One of the key controls on the strength of HSLA steels is precipitates, nucleated both in the austenite and in the ferrite during hot strip rolling and cooling. To optimize the compositions and the rolling parameters it is crucial to have knowledge about the precipitation evolution.

WHY USING A LARGE SCALE FACILITY

It is challenging to investigate precipitation development during hot rolling because of the difficulty in acquiring samples during processing and maintaining the processing conditions/states. Generally, laboratory simulations are required that enable freezing (quenching) of samples at key stages of the process. However, the phase transformation and the possibility for precipitation to occur during quenching makes it impossible to know the size and fraction of precipitates at high temperature. In-situ measurements under processing conditions would provide a solution to this challenge. Due to the fast process times, scales of interest and challenging experiment conditions, this can only be performed with synchrotron X-ray scattering techniques. Simultaneous small (SAXS) and wide angle (WAXS) X-ray scattering measurements would provide invaluable information of precipitation during phase transformation.

HOW THE WORK WAS DONE

With the aim of performing rolling simulations in-situ with X-ray measurements, it was first necessary to establish that the size distribution of very small fractions of nano-sized precipitates could be sufficiently detected in samples of relevant thickness and at a rate of a minimum of measurements every second. Hence, ex-situ measurements of different states of precipitates were performed in 4 mm samples from laboratory simulations and full-scale industry tests, using high-energy X-ray scattering at the P21.2 beamline of the Petra III synchrotron in Hamburg. The possibility for rapid simultaneous acquisition of SAXS and WAXS at P21.2 also enabled a study of both precipitation and phase transformation with in-situ heating/cooling and measurements every second. The expertise of personnel at P21.2, Malte Blankenburg and Ulrich Lienert (DESY), is greatly acknowledged.

THE RESULTS AND EXPECTED IMPACT

It was established that the simultaneous acquisition with SAXS and WAXS can be used for microalloyed steels with low fractions of nano-sized particles. This is especially important in the foreseen next-step of the project to perform measurements during in-situ rolling simulations to investigate the key questions around the timing of the different processes. To perform measurements during rolling simulations, a dilatometer at P21.2 would be the perfect solution. The measurements so far provide SSAB with valuable information about Nb and TiNb rich precipitates. An example of the acquired data is shown in the figure.

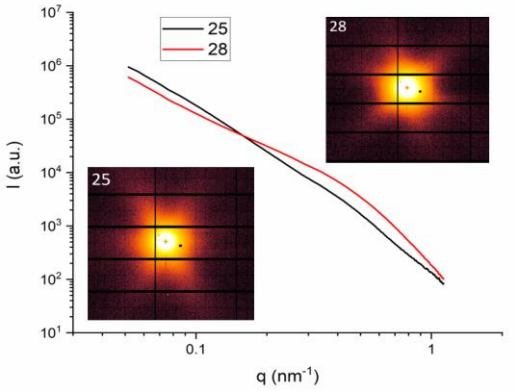

Figure. Ex situ 2d-images and scattering curves for two laboratory samples showing a difference in SAXS intensities (fraction and size of precipitates) for a quenched sample (25) and a sample soaked at 600°C (28).

"This work has provided us with important information about our materials and new contacts in the field of LSI." ***/Linda Bäcke, SSAB***

Contacts: Linda Bäcke – SSAB, Linda.Backe@ssab.com
Stephen Hall – Lund University, Stephen.Hall@solid.lth.se

Vinnova's project No: 2019-05294 **Duration:** February 2020 -- March 2022

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Synchrotron nano-diffraction aiding development of advanced CVD coatings for increased cutting tool performance

THE INDUSTRIAL CHALLENGE

The application of a thin (~10 µm) coating on a cemented carbide tool for metal machining can lead to improvements in tool performance of more than 100 times. $Al_xTi_{1-x}N$ grown by chemical vapour deposition (CVD) is a new coating system within the metal cutting industry and at present only a few companies offer CVD $Al_xTi_{1-x}N$-coated products. The residual stress state in the coating is believed to be a key parameter for performance and tool life, why the ability to tailor the residual stress profiles could allow further enhancements. However, residual stress measurements with standard in-house methods have spatial limitations and cannot resolve gradients through the CVD coating thickness.

WHY USING A LARGE SCALE FACILITY

Through the technical developments associated with the third and fourth generation synchrotron sources it is today possible to perform X-ray diffraction experiments with a spatial resolution well below 100 nm, so called nano-diffraction. In this case the beam size is much smaller than the coating thickness, which allows measurement of stress profile.

HOW THE WORK WAS DONE

Sandvik Coromant and Chalmers jointly performed transmission X-ray nano-diffraction experiments at the NanoMAX beamline at MAX IV, which used an energy of 14 keV and a beam size of ~60 nm. Samples were machined from cutting tools, and carefully prepared through focused ion beam (FIB) milling to preserve the stress state in the coating (Fig. 1a-c). The coating thickness was around 5 µm and the probed width (in the beam direction) in the order of 60-100 µm. Diffraction patterns were collected by a downstream area detector (Fig. 1d-e), which allowed subsequent calculation of the residual stresses as a function of position using the traditional $\sin^2\Psi$ method.

THE RESULTS AND EXPECTED IMPACT

Even in challenging industrial materials, such as coarse-grained CVD AlTiN, synchrotron X-ray nano-diffraction enabled accurate and reliable measurements of residual stresses with sufficient spatial resolution (~100-300 nm) to capture the through-thickness gradient. It also provides simultaneous information of the phase composition, which is extremely important for multilayer coatings (Fig. 2). This provides a unique tool to further understand the relationships between process, structure, stress state, and performance in cutting applications. Several coatings with different processing conditions were investigated, and the results can be correlated with performance measurements to allow knowledge-based design of processes for optimal properties.

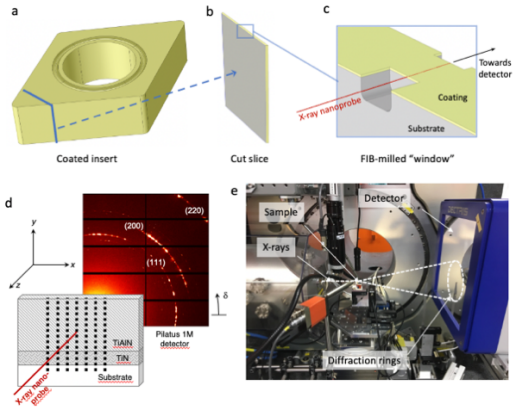

Figure 1. a-c: Sample preparation. d: Schematic of the set-up. e: Photograph of the experimental station at NanoMAX.

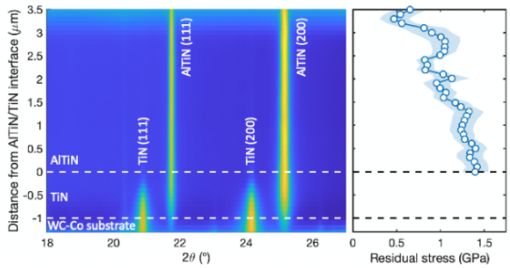

Figure 2. Example of phase composition and residual stress as a function of position in an AlTiN coating.

Read more at: *https://www.maxiv.lu.se/news/metal-industry-giant-conducts-experiments-at-max-iv/*

Contacts: Linus von Fieandt – Sandvik Coromant, linus.von_fieandt@sandvik.com
Mikael Kriitkos – Sandvik Coromant mikael.kritikos@sandvik.com
Magnus Hörnqvist Colliander – Chalmers University of Technology, magnus.colliander@chalmers.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

HAXPES study of oxide films on AM stainless steel materials

THE INDUSTRIAL CHALLENGE

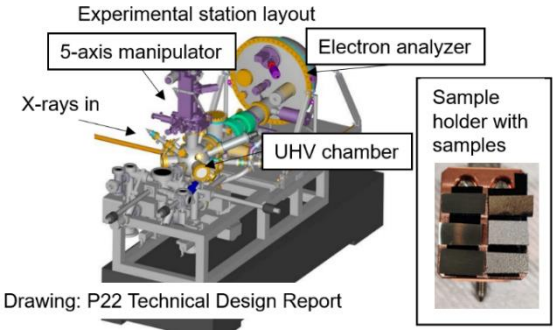
The replacement of conventional stainless steel (SS) with additive manufactured (AM) SS material has many strategical benefits. The printed components used by Alfa Laval often require further treatments to improve their corrosion and structural performance. In turn, Quintus Technologies is specializing on the post-processing of AM components and works together with Alfa Laval and Swerim on improving the corrosion performance of AM SS. For prediction of corrosion performance, it is important to understand how post-processing protocols affect thickness and chemical composition of the surface oxide films.

WHY USING A LARGE SCALE FACILITY

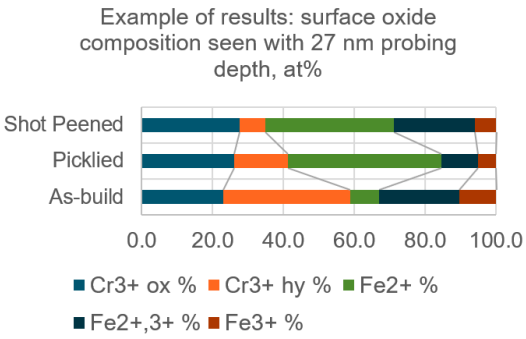
Unlike conventional X-ray Photoelectron Spectroscopy (XPS), the synchrotron-based hard X-ray version (HAXPES) allows to tune the probing depth by changing the incoming photon energy for performance of non-destructive chemical depth profiling of relatively thick oxide films (up to 30 nm). Thanks to the very high brightness, the rough surfaces of as-build AM SS can be studied, and good signal-to-noise ratios can be achieved in a short time.

HOW THE WORK WAS DONE

The experiment was performed at the P22 beamline of Petra III, Hamburg. Samples of AM 316L SS were cut from the printed columns and mounted on the sample holder. The HAXPES spectra were measured using 7.5 keV and 2.5 keV photon energies and at two different incidence angles. The 13 samples tested had been subjected to high pressure heat treatment, shot peening and pickling (in total about 400 spectra during 40h beamtime).



THE RESULTS AND EXPECTED IMPACT



The results revealed changes in chemical composition of the surface oxide introduced by post-processing. Illustration above shows results for as-build, shot-peened and pickled surfaces. The surface oxide of non-treated AM steel appeared to be thick (about 20 nm) and with non-uniform composition (the top 10 nm rich with Fe oxides and the bottom 10 nm rich with Cr_2O_3 in presence of metallic Fe and Cr). Shot peening and pickling surface treatments made surface oxide more uniform and homogeneous with a thickness of 5 nm. Effects of interaction with pickling solution and glass beads were obtained for pickled and shot peened surfaces, respectfully, which can potentially impact their corrosion properties. The results will contribute to the strategical efforts to develop guidelines for production of AM parts with predictable corrosion properties.

"Possibility to participate in a project and join on-site during the measurements, has provided a better understanding of how the HAXPES technique works and how to interpret data"/ D.Klint, Alfa Laval

"The project is extremely useful in understanding the effect of Hot Isostatic Pressing on surface alloy and oxide composition"
/J. Shipley, QuintusTechnologies

Contacts: D. Klint – Alfa Laval Lund, daniel.klint@alfalaval.com
J. Shipley – Quintus Technologies, james.shipley@quintusteam.com
K. Simonov – Swerim AB, konstantin.simonov@swerim.se

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INDUSTRIAL PILOT PROJECT

Investigating residual stresses and SCC in additively manufactured stainless steel by neutron diffraction

THE INDUSTRIAL CHALLENGE

Residual stress and porosities are known to have a detrimental effect on the mechanical and corrosion properties of any stainless-steel (SS) parts made by Powder bed fusion – Laser Beam (PBF-LB, SLM) technologies. During the PBF-LB process, it is not possible to avoid stress and pore formation in the built part. To ensure the satisfactory material performance of SS parts made by PBF-LB it is therefore common practice to undergo a series of post stress and porosity removal operations. Hot Isostatic Pressing (HIP) is one of the most effective material densification processes and is often included in the PBF-LB manufacturing chain. Quintus Technologies is a world leader in high-pressure technology and has strong expertise in the HIP treatment of SS. To reduce the manufacturing cost and shorten the lead time of PBF-LB parts, it is desired to combine the stress relaxation operation with other treatments, such as the HIP. However, the effect of such post-processing combination possibilities on stresses is still not known.

WHY USING A LARGE SCALE FACILITY

Because of the resulting rough as-built surfaces, the anisotropic stress state and microstructure, it is challenging to characterize stress in PBF-LB built SS by conventional analytical techniques such as hole drilling. Neutron diffraction/imaging and high-energy synchrotron x-ray diffraction (XRD) are the only techniques that allow to non-destructively measure/map macro and micro residual stress/strain at any part of a large SS sample, including all important strain components.

HOW THE WORK WAS DONE

Austenitic SS (316L) samples with size 20x20x40 mm^3 were manufactured using the PBF-LB and HIP treated by Quintus. The residual strain was then mapped in all parts of seven samples (including a reference), using both neutron Bragg-edge imaging setup at the IMAT and neutron diffraction setup at the Engine-X beamline at ISIS (UK). The measurements were performed by Dr. G. Burca and J. Kelleher from ISIS. To capture the stress gradient at the near surface (7-150 µm) with improved special resolution, complimentary synchrotron XRD-based stress measurements were performed at P61A beamline at the Petra III synchrotron in Germany. The residual strains were evaluated by examining the diffraction peaks and Bragg-edge shifts.

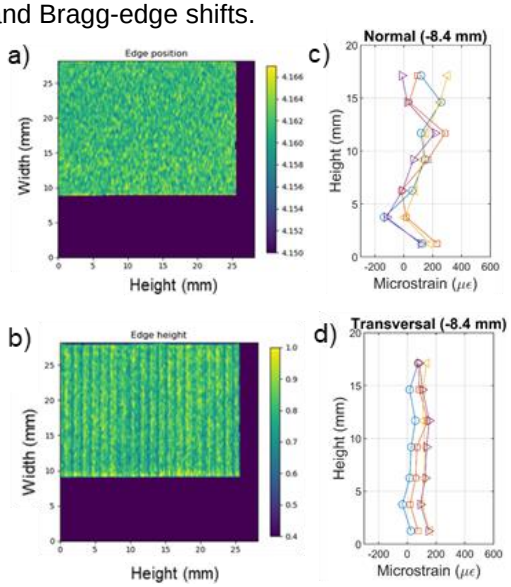

Figure 1.a) and b) display the variation of Bragg-edge and height in given sample area from neutron imaging; c) and d) shows the variation of normal and transversal strains from neutron diffraction.

THE RESULTS AND EXPECTED IMPACT

All measurements show similar results, and the work display the advantage and disadvantages of different neutron and synchrotron-based stress measurement techniques for characterizing phase and grain-specific residual stress in large metallic materials at different length scale. The results showed that it is possible to remove or reduce the stress in SS that originated from PBF-LB printing only by adjusting HIP condition without adding another stress relaxation operation (Fig.1). High-quality data and detailed strain maps from the project were also used for validation of a previously developed numerical model aimed to simulate the stress in SS built by PBF-LB related to the subsequent HIP. Results of the work are expected to deepen Quintus' understanding which might help the industry to cut one step out from the PBF-LB manufacturing pipeline.

Contacts: James Shipley– Quintus Technologies, james.shipley@quintusteam.com
T. Maimaitiyili – Swerim AB, tuerdi.maimaitiyili@swerim.se

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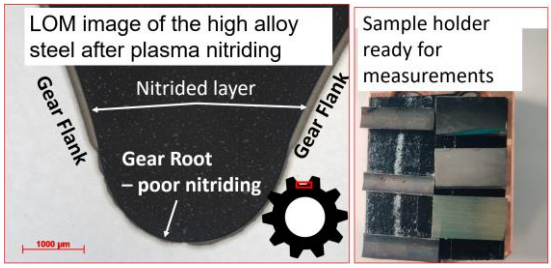
INDUSTRIAL PILOT PROJECT

XPS-study of passivating films and their effect on plasma nitriding

THE INDUSTRIAL CHALLENGE

The plasma nitriding hardening method is extremely important for improving service life of gears. However, the heat treatment subcontractor Bodycote faces challenge when working on plasma nitrided gears manufactured from advanced high-alloy steel grades. The core problem is that unwanted surface passivation seems to inhibit efficient nitriding in the gear root and lead to lack of nitriding depth. It is therefore important to understand what causes this surface passivation that leads to a lack of nitriding depth.



WHY USING A LARGE SCALE FACILITY

To measure the surface chemistry of the passive layers to a deeper depth than ordinary XPS, the higher energy of incoming X-rays of synchrotron based Hard X-ray Photoelectron Spectroscopy (HAXPES) results in an increased inelastic mean free path and increased escape depth. The ability to tune the probing depth combined with excellent chemical sensitivity of XPS allow to perform composition and chemical analysis of the surface and sub-surface layers of material.

HOW THE WORK WAS DONE

Samples of ~4x4x10 mm were cut from vacuum heat-treated gear flanks and gear roots from two different materials, AISI steel H13 and the Ovako Hybrid Steel. After vacuum treatment the flanks were ground, while the roots were left untreated. In addition, samples of similar size were cut and ground and left to age in room temperature up to six months prior to measurements. The measurements were performed at the P22 beamline of Petra III in Hamburg during a 40 hour slot.

THE RESULTS AND EXPECTED IMPACT

The results showed that aluminium oxide (Al2O3) was present in high concentrations in the gear roots of both H13 and Ovako Hybrid Steel. In the case of Hybrid Steel this was not unexpected since it contains a couple of wt% aluminium. For the H13 alloy it was more surprising since it contains only ~0.02 wt%. Aluminium evidently diffuses to the surface during the vacuum hardening performed after gear hobbing and forms an oxide. The room temperature experiments showed that aluminum diffuses to the surface of the H13 alloy, however the amounts are much lower (Figure).

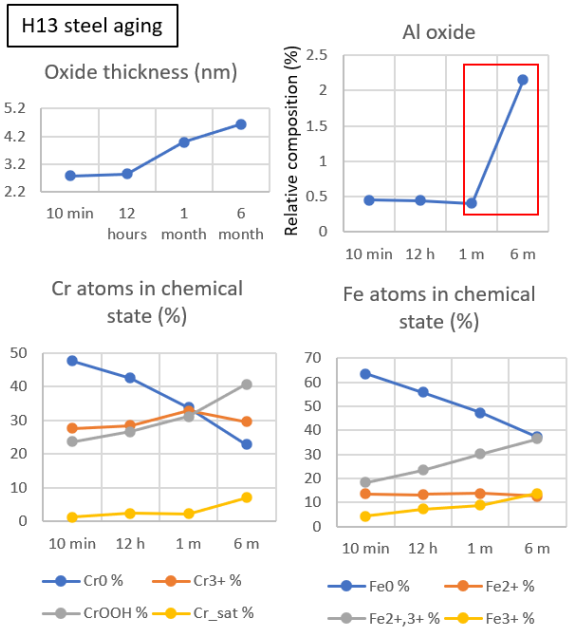


Figure. Results of HAXPES illustrating diffusion of Al in H13 steel after 1 month ageing in air.

It has previously not been known what causes the passivation in gear roots, but following the HAXPES experiment it is clear that Al2O3 is present in high concentrations. Since Al2O3 is a very dense oxide, known to pacify surfaces, it is likely that it plays a large role for the resulting passive films at the gear roots. Now it is possible to start working on solutions to circumvent this passivation.

Contacts: Steve Ooi – Ovako Sweden, steve.ooi@ovako.com
Arvin Ghorbani – Bodycote Värmbehandling, arvin.ghorbani@bodycote.com
Albin Stormvinter, RISE IVF, albin.stormvinter@ri.se
Sven Haglund, Swerim, sven.haglund@swerim.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Transition metal valence in commercial glasses analysed using X-ray Absorption Spectroscopy at Balder beamline, MAX IV

THE INDUSTRIAL CHALLENGE

The stone wool manufacturer Paroc (a part of Owens Corning) considers blending in additional waste materials into the production to obtain a more sustainable product. By using waste material that otherwise would go to the landfill also less virgin raw material (volcanic rock) would be used. The waste material contains manganese (Mn) which may potentially affect the iron (Fe) redox equilibria that greatly affects the melt and product properties. Paroc therefor wish to understand and simulate the effect of blending in additional waste material in their product.

WHY USING A LARGE SCALE FACILITY

Low concentrations and extremely complex glassy matrix of stone wool melts makes it impossible to use lab-based methods for analysing the chemical speciation. Lab-based method also requires considerable sample preparation, possibly affecting the chemical state. Synchrotron X-ray absorption spectroscopy (XAS) is a powerful method to determine the oxidation state and local structural environment of Mn and Fe. It can be applied to the glassy state of matter and provides the necessary energy to get accurate results. In the future, the BALDER beamline at MAX IV will also provide possibilities for high throughput measurements.

HOW THE WORK WAS DONE

Stone wool is an extremely complex composition matrix and is the reason why six conventional soda-lime-silicate (SLS) glasses containing various known amounts of Mn and Fe were prepared in the laboratory as materials with simpler glassy matrix. In addition, six industrial glass samples were prepared with different blends of waste material. The glass samples were powdered and mixed with polyethylene powder to reduce the concentration of the element to be analysed and bind the powder together so that tablets can be pressed.

The experiments were performed at the Balder beamline of MAX IV, Lund, primarily using XANES but some EXAFS spectra were also acquired. The Mn and Fe absorption edge were acquired from 12 glass samples in total. Most were measured in transmission mode, however, for some samples fluorescence mode had to be used due to their low concentration of Mn. In addition, five crystalline Mn/Fe oxide reference materials and the waste material were measured and these were essential for interpreting the XANES results. The absorption edge positions were compared with the reference samples to determine the oxidation states of Fe and Mn in the samples.

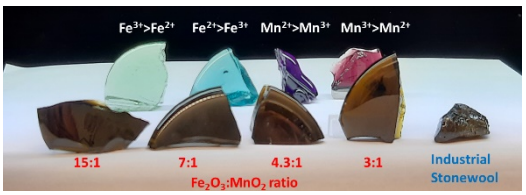

Figure. A photo of selected glassy samples that were measured at Balder (MAX IV).

THE RESULTS AND EXPECTED IMPACT

The XANES results show that the redox pair Mn^{2+}/Mn^{3+} in oxidized soda-lime-silica melts affects the Fe^{2+}/Fe^{3+} redox by increasing the amount of Fe^{3+} in a redox equilibria reaction: $Mn^{3+}+Fe^{2+} \rightleftarrows Mn^{2+}+Fe^{3+}$. In reduced stone wool melts, there is also an increase in the amount of Fe^{3+} when Mn is present, however, the increase is not directly reflected by the Mn-concentration and the amount of Mn^{2+} does not necessarily increase simultaneously. The project has led to increased understanding for Paroc on the implications of blending additional waste materials into their production but also an opportunity for Paroc (Owens Corning) to utilize XAS to quantitatively determine the redox equilibria in the glassy state.

"A fantastic collaboration giving results from the fascinating facility MAX IV that will be an important step towards a more sustainable stone wool product" /Veronica Sjödin, Paroc AB.

Contacts: Veronica Sjödin, Paroc AB (a part of Owens Corning), Veronica.Sjodin@owenscorning.com
Jenny Rissler, LSI-expert – RISE Research Institutes of Sweden, Jenny.Rissler@ri.se
Stefan Karlsson, Lina Grund – RISE Research Institutes of Sweden, Stefan.Karlsson@ri.se , Lina.Grund@ri.se

Vinnova's project No: 2020-03776 **Duration:** November 2020 -- September 2022

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

In-situ analysis of ultrathin passive film of advanced tooling alloy by synchrotron X-ray photoelectron technique

THE INDUSTRIAL CHALLENGE

Uddeholms AB is specialized in developing and producing advanced tooling alloys for demanding applications. A great challenge is to achieve sufficient corrosion resistance and desirable mechanical properties of the alloys simultaneously, which requires a detailed understanding of the passive film formed on the alloy surface and the influence of heat treatment.

WHY USING A LARGE SCALE FACILITY

Tooling alloys are martensitic steels with magnetic property, which is problematic for surface analysis using lab-source X-ray photoelectron spectroscopy (XPS). Moreover, normal XPS is done in ultrahigh vacuum condition, so the information of the passive film is not really relevant for service conditions in air or aqueous environments. Synchrotron-based XPS, with high flux and tunable energy of the beam, and especially the newly-developed ambient pressure XPS (APXPS) measuring system, can tackle the challenge, allowing us to obtain reliable and relevant information of the passive film.

HOW THE WORK WAS DONE

Tooling alloy samples were prepared and heat treated by Uddeholms AB, and electrochemical measurements to define the experimental conditions for the synchrotron APXPS measurements were performed at KTH. By using a special designed sample environment (electrochemical cell) and the dip-and-pull method, we were able to do XPS measurement at a pressure up to 17 mbar, immediately after lift-up of the sample from the corrosive solution that was subjected to electrochemical polarization. The APXPS measurements were performed at the HIPPIE beamline of MAX IV, in collaboration with Prof. Edvin Lundgren's group at Lund university, and supported by the APXPS team leader, Andrey Shavorskiy, and beam scientist, Mattia Scardamaglia.

THE RESULTS AND EXPECTED IMPACT

With the help of the beam scientist, we solved the problem of magnetic effect on the XPS measurement. During the APXPS measurement (at 17 mbar), there is still an aqueous adlayer on the sample surface, so the information obtained represent the whole passive film including the hydroxide part. Moreover, we could analyse the passive film immediately after anodic polarization, yielding valuable information of the chemical composition and stability of the passive film.

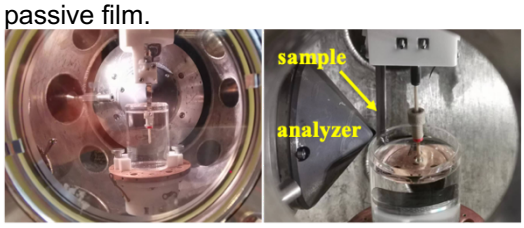

Figure. Left: the electrochemical cell in APXPS chamber. Right: sample in pull position for XPS measurement.

This experiment demonstrates the unique possibility to analyse, in-situ, the passive films formed on the surface of metallic materials including martensitic tooling alloys, and the information obtained are helpful for the optimization of the tooling alloy and heat treatment. We also extended the collaboration with other academic and industrial partners. The technique has already been included in other projects.

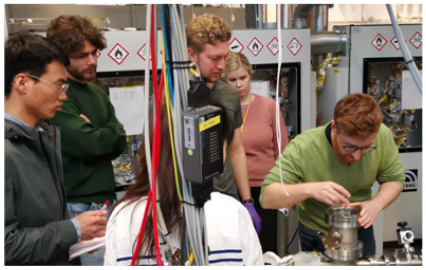

Figure. Beam scientist, Mattia Scardamaglia, shows how to remove dissolved gas from the corrosive solution.

"This project has demonstrated the unique possibility for us to do detailed and relevant surface analysis of our tooling alloys, which is of great importance in our R&D work" ***/Krishnan Anantha, Uddeholm.***

UDDEHOLM
a voestalpine company

KTH VETENSKAP OCH KONST

Contacts: Krishnan H. Anantha – Uddeholms AB, kriha@uddeholm.com
Jinshan Pan – KTH Royal Institute of Technology, jinshanp@kth.se

Vinnova's project No: 2020-03778 **Duration:** November 2020 -- May 2022

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

In-situ SAXS and WAXS synchrotron Xray diffraction investigations of additive manufacturing parts for Ni-base superalloys.

THE INDUSTRIAL CHALLENGE

Gas turbine components produced by powder bed fusion laser beam (PBF-LB) additive manufacturing are gaining large industrial interest. Component and turbine performance can be significantly improved if features such as lattice structures, surface modifications and novel cooling design concepts can be produced. However, final properties of Ni-based superalloys required to produce these parts rely on post processing steps. For Ni-based superalloy components produced by PBF-LB technique, heat treatment (HT) steps in terms of hot isostatic pressing, solution heat treatment and ageing are required to tailor the mechanical properties. The group of superalloys which meet the mechanical properties, i.e., precipitation hardened superalloys, are difficult to process by powder bed fusion techniques. Gamma-prime precipitation and built-in residual strain and stresses play roles in a phenomenon called strain-age cracking. It is already known that high temperature exposure during post-processing will trigger strain age cracking. Improving alloy processability requires investigation of these phenomenon at relevant conditions in situ.

WHY USING A LARGE-SCALE FACILITY?

Home lab Xray diffraction (XRD) measurements have shown residual tensile stresses exceeding 900 MPa in alloy 247. This indicates the importance of reducing stresses in the HT cycle. The phase transformations during heating can be observed from dilatometry experiments, where various temperatures, heating and cooling rates can be applied. However, only the high brilliance and beam penetration at high energy synchrotron source coupled with in-situ dilatometry capabilities would allow for monitoring the introduction of residual stresses or the secondary phase precipitation in-situ, at a time scale necessary.

HOW THE WORK WAS DONE

Material of the Ni-base alloy 247 type was PBF-LB printed at Siemens Energy AB in Finspång. From these prints hollow dilatometer samples (OD 4mm / ID 2mm x 10mm) were prepared by electric discharge machining. Samples were produced as hollow in order to improve the signal response. Combined wide angle (WAXS) and small angle (SAXS) scattering were performed at the P07 (High Energy Materials Science) Beamline of Helmholtz-Zentrum Hereon and DESY at Petra III, Hamburg. P07 also allows in-situ thermal cycles (programmed to mimic the industrial processes used in the tempering steps of the PBF-LB production step). Seven thermal cycles of the material were done in order to study precipitation and growth of γ', dissolution of the precipitates together with the associated stress development. during heating and cooling.

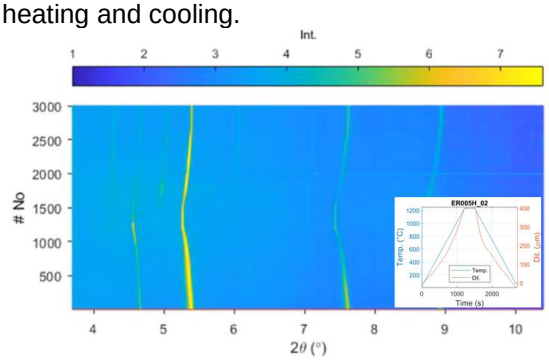

Figure. During the thermal cycling diffraction patterns were recorded and analyzed based on precipitation and micro stress evolution.

THE RESULTS AND EXPECTED IMPACT

The performed in-situ experiments gave valuable insights in the response of thermal cycling on stress development. The measurement results are, however, not conclusive and separate test series for SAXS and WAXS would improve data quality for a better determination of the development of stresses. Especially continued WAXS experiments are needed for a better understanding of how the thermal treatments can be optimized for improved component quality.

"The use of LSI in-situ techniques will help industry R&D to optimize process parameters in the AM field."/ Håkan Brodin, Siemens Energy AB

Contacts: Håkan Brodin, Siemens Energy AB hakan.brodin@siemens-energy.com
Pelle Mellin, Swerim AB pelle.mellin@swerim.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Spatially resolved nano-Xray diffraction of advanced metals: machining induced white layers of Ti- and Ni-based alloys

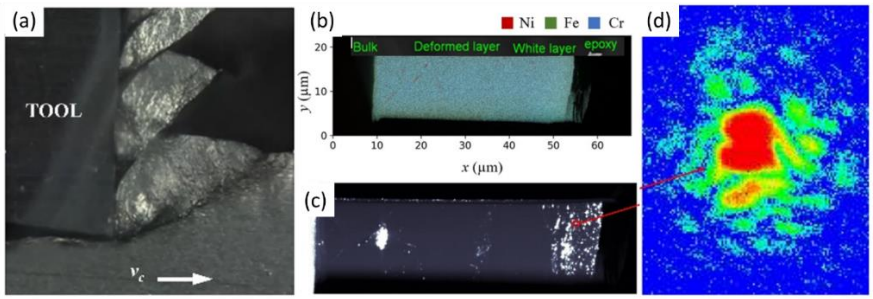

Figure. 1: (a) Photo of the machining process showing the tool and evidencing the formation of the white layer (WL) in machined Inconel 718. (b) High resolution X-ray fluorescence (XRF) map showing the distribution of Ni, Fe and Cr along the lamella. (c) Diffraction map of the lamellae. Bright spots indicate nanocrystalline grains aligned in Bragg condition. (d) High resolution Bragg spot from a selected grain in the WL, evidencing fringes related to its shape and internal strain.

THE INDUSTRIAL CHALLENGE

Recent development of Near net shape manufacturing technologies (e.g. additive manufacturing) offers promising alternatives to conventional processes for the manufacture of titanium and nickel based alloy components. Under conditions of high-speed finishing, the cutting tool is subjected to high temperatures leading to excessive wear thus increasing the risk of causing the subsurface damage called white layer (WL). The formation and properties of WLs are still not fully understood.

WHY USING A LARGE SCALE FACILITY

Studying deformation mechanisms at nano scale and the formation of WL is providing extremely valuable information for the science community and metal industry. Characterization using scanning nano-focused X-ray diffraction (nano-XRD) is necessary to determine, elastic strain, residual stress and phase in the WL and surrounding material. Nano-XRD permits this by allowing a, for example, greater field of view than transmission electron microscope (TEM). Synchrotrons also allow for simultaneous / complementary chemical analyses with X-ray fluorescence, (XRF). Most importantly, however, Bragg Coherent Diffraction Imaging (BCDI) measurements can be performed to investigate the internal strain of individual grains in the WL, something no other technique can do.

HOW THE WORK WAS DONE

High-speed cutting experiments on Inconel 718 specimens were conducted at Seco Tools R&D facility in Fagersta, Sweden. Lamella samples with approximate thicknesses of 1 μm, containing both WL and bulk structure, were prepared using focused ion beam at Lund University. The synchrotron diffraction experiments were conducted at both the P10 beamline (Petra III – Hamburg) and the NanoMAX beamline (MAX IV – Lund). Both beamlines have similar capabilities with the latter offering a much better resolution for nano-XRD and XRF analysis. The thin lamella was studied in transmission geometry and the strain distribution was investigated by tracking the (111) Bragg reflection of the *γ* phase while the beam was scanned with the sample. Rocking curves were performed on selected grains in the WL, allowing for coherent imaging via phase retrieval algorithms.

THE RESULTS AND EXPECTED IMPACT

The nano-XRD results seen in Figure 1 b-d are providing us with a better understanding of some characteristic features of the WL for example by revealing an, until now, unidentified strain structure in the top surface white layer. The analysis of individual grains of the WL with BCDI suggests it is possible to use this method for the reconstruction of individual grains and map their internal strain field. Replicating such measurement across multiple grains would allow us in the future to obtain relevant information about fine structured strain gradients inside typical WL grains, and grain statistic across the WL phase. Such detailed strain fields are currently not achievable with a lab-based TEM.

Contacts: Rachid M'Saoubi – Seco Tools, rachid.msaoubi@secotools.com
Filip Lenrick – Lund University, filip.lenrick@iprod.lth.se

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INDUSTRIAL PILOT PROJECT

Precipitate characterization in electrical steels using a combination of SANS and VSANS

THE INDUSTRIAL CHALLENGE

Surahammars Bruks AB, part of Tata steel, produces electrical steels that are used in magnetically active parts of motors. The use of electric vehicles has been increasing enormously, resulting in great demand for the continuous development of electrical steel. Nano size precipitates (typically 10-400 nm) can strongly influence the magnetic properties of the steels, either directly by interference in domain wall motion, or indirectly by grain growth inhibition.

WHY USING A LARGE SCALE FACILITY

Although electron microscopy techniques are powerful for the analysis of small precipitates, they only give local information from extremely small volumes. Obtaining statistical information on nano-size particles for sample sizes, that also are measurable during magnetic and mechanical tests, requires small-angle scattering (SAS) methods using a photon or neutron beam. Neutrons (SANS) provide two great advantages: good contrast for metallic precipitates within the metallic matrix; and magnetic contrast for non-magnetic precipitates within a ferritic matrix.

HOW THE WORK WAS DONE

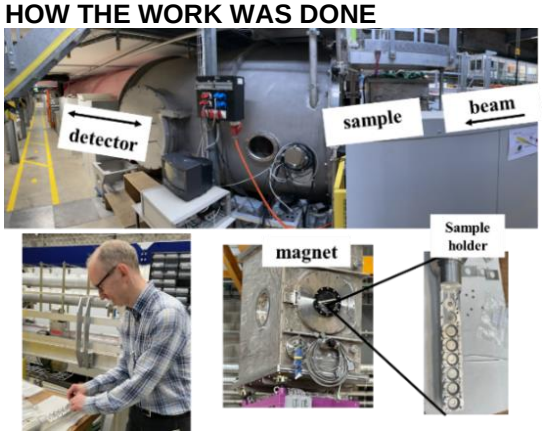


Figure 1. Top: The experimental setup, the detector could move in distances between 3 and 18 m from the sample to cover a wide range of particle size. Bottom: Arvid Broddefalk preparing samples; magnet mounted on the sample stage and the sample holder.

The experiment was performed on SANS-1 instrument at the Swiss Spallation Neutron Source, SINQ, at PSI, Switzerland. In one set of materials, small particles (<15nm) were added deliberately to increase the strength of the material, while another group of materials contained precipitates and particles that were detrimental to the magnetic properties. Each measurement was performed by stacking ten 0.3 mm thick samples and illuminating a 10 mm beam on the stack.

THE RESULTS AND EXPECTED IMPACT

The outcome provided statistical results about precipitates' size and concentration within the bulk. Such results can be directly correlated to the production parameters, as well as to the magnetic and mechanical properties of the final products. As a non-destructive method SANS has great potential for quality control and comparative study of samples. This data analysis resulted in the number density of different types of precipitates. Figure 2 shows the size distribution of precipitates in three samples with different magnetic response.

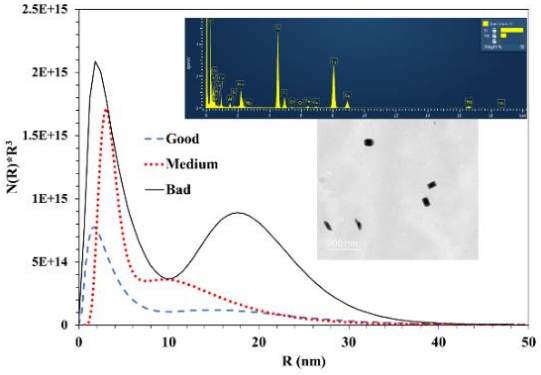


Figure 2. Size distribution of particles for samples that show good, medium, and bad magnetic properties. The inserted TEM results show the type of larger precipitates found with different fractions in all samples.

"To optimize electrical steels for high performance, it is important to understand the parameters that influence the magnetic and mechanical properties. Through excellent collaboration with Swerim, PSI and Anaxam we had interesting results on the size distributions of precipitates in selected samples. The SANS results together with SEM and TEM should enable us to better understand the underlying causes of the physical properties of our materials."
//Arvid Broddefalk, Surahammars Bruks AB

Contacts: Arvid Broddefalk – Surahammars Bruks AB, arvid.broddefalk@sura.se
Shirin Nouhi – Swerim AB, shirin.nouhi@swerim.se

Vinnova's project No: 2020-03786 **Duration:** November 2020 – October 2022

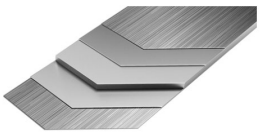
Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

In-situ investigation of grain growth and texture evolution of multilayer Al-alloys during simulated brazing using 3DXRD

THE INDUSTRIAL CHALLENGE

Heat exchanger parts in automotive applications can be exposed to a very corrosive environment that can result in tube leakage. Gränges has patented a unique product to migrate corrosion issues by engineering multilayer alloys that modifies themselves during the brazing process.



WHY USING A LARGE SCALE FACILITY

The multilayer products are rolled to a total thickness of ~ 0.3 mm or less. Cross-section electron back-scatter diffraction (EBSD) can provide ex situ 2D information of the grains in each layer. However, an in-depth understanding of recrystallization and grain growth for each layer and/or at the interfaces during brazing processes needs diffraction studies performed at very high time resolution and fine beam size. The fine beam size with high energy of synchrotron Xray diffraction allows probing each layer separately, but still penetrating through a certain height of ~ mm thick rod. The time resolution of synchrotron also allows an in-situ experiment for such a fast recrystallization process.

HOW THE WORK WAS DONE

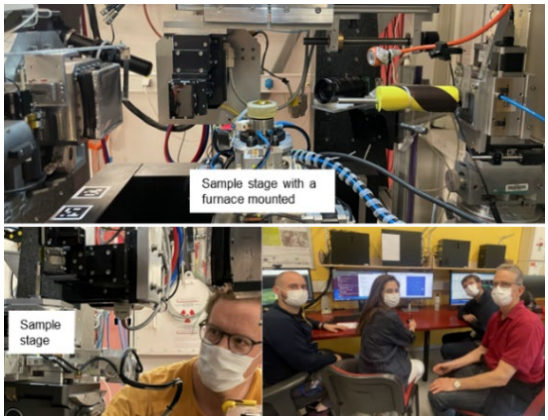


Figure 1. top: 3DXRD setup with a furnace on sample stage, bottom left: Adjustment of the sample setup for DCT measurement and bottom right: and controlling and following the experiment together with ESRF instrument scientists.

The experiments were performed on the ID11 instrument at the ESRF facility in Grenoble, France. The sample was heated during the 3D X-ray diffraction (3DXRD) experiment and measurements were performed at different heights with a 15 µm beam size to probe different layers of the sandwiched material. At each height, a full rotation of the sample was measured to access all texture components present in each layer. At the end of the recrystallization process, also diffraction contrast tomography (DCT) measurement was performed on the recrystallized sample to obtain absolute grain size and shape. Experiment was performed on two materials with slightly different compositions for different layers.

THE RESULTS AND EXPECTED IMPACT

The results showed the effect of layer composition on dominant texture components that is corelated directly with the mechanical and corrosivity properties of the material.

They also provided a detailed understanding of the texture evolution of individual layers that together are responsible for the performance of the product. Such understanding is crucial for the design and further improvement of these products. 3DXRD and DCT are both new techniques within large scale research infrastructure methods with great potential for different metallic materials.

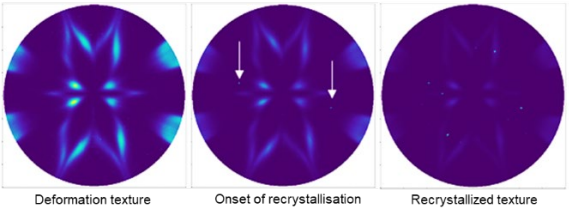


Figure 2. {111} pole figures at different temperatures show the texture evolution during recrystallisation.

"This work and technique used verifies a model for texture control and related properties very important for Gränges"
/Anders Oskarsson Gränges Finspång AB

GRÄNGES

SWERIM

Contacts: Anders Oskarsson – Gränges AB, anders.oskarsson@granges.com
Shirin Nouhi – Swerim AB, shirin.nouhi@swerim.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

Renewable packaging materials-impregnation depth measurements for pulping industry using synchrotron XRF

THE INDUSTRIAL CHALLENGE

The environmental impact of plastic packaging is a growing concern but using wood fibre-based materials as a replacement could significantly reduce the problem. High Yield Pulps (HYP), such as Chemithermomechanical pulp (CTMP) will be a major component of sustainable packaging materials in future. The major challenge for CTMP is to achieve the even distribution of sulphite (SO_3^{2-}) ions in the inner-and outer parts of the wood chips to preserve in homogenous wood fibre properties. However, the sulphonation degree and softening of each fibre in the chip refiner before being defibrated is largely unknown at a micro-scale level. There is currently no easy method to measure the distribution of sulphonate groups either in wood chips or the individual fibres.

WHY USING A LARGE SCALE FACILITY

Synchrotron XRF imaging technology is a suitable way to achieve high resolution imaging measurements of sulphur distribution between fibres and also within individual fibres, since the image resolution achieved is 1 µm.

HOW THE WORK WAS DONE

Thin paper sheets with diluted sulphonated fibre content were required for investigation at the synchrotron. Since kraft pulp is sulphur free due to thoroughly wash, Valmet pilot CTMP pulp sample diluted with different percentages of SCA kraft pulp assured individual sulphonated fibre observation in 20 g/m^2 paper sheets that were produced.

Two beamtime applications were approved from two synchrotrons:

1. The Phoenix beamline of the Swiss Light Source (SLS) in Switzerland.
2. The 2-ID-D beamline of the Advanced Photon Source (APS) in USA.

The samples were measured by remote access due to pandemic restrictions, but in live contact with beamline scientists Camelia Nicoleta Borca at SLS/PSI and Barry Lai at APS.

THE RESULTS AND EXPECTED IMPACT

PSI measured in vacuum, which enabled to see a both sodium (Na) and sulphur (S), Figure 1. Sulphur shows good accuracy and is used further in the project.

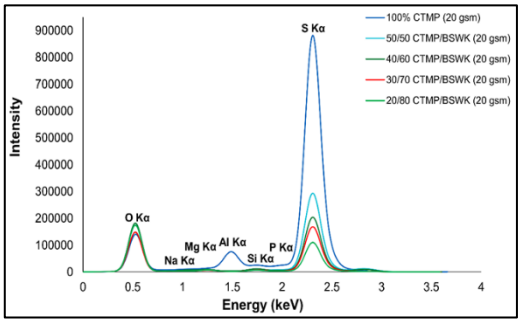


Figure1. Sample spectra from PSI using 0.5 × 0.8 mm spot size for area averaging.

APS focused on imaging with 1 µm spatial resolution in air. Figure 2 clearly reveals significant variation in sulphonation.

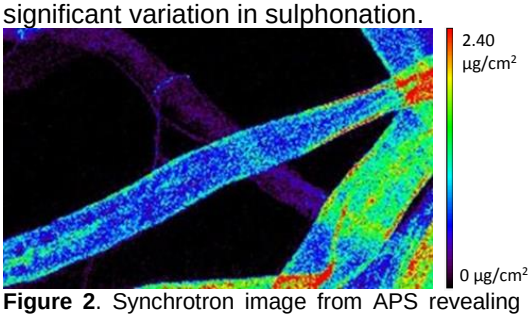


Figure 2. Synchrotron image from APS revealing uneven sulphur homogeneity on individual fibre level in CTMP pulp

The conclusion of this project is:

1. The sulphur content of fibres in the sample varies. Within fibres, the sulphonation seems uneven, with higher concentration at the fibre surface.
2. The resolution needed for homogeneity measurements must be able to resolve sulphur within individual fibres.
3. Such measurements can assist in optimizing process parameters to reach even impregnation for the manufacture of advanced fibre materials.

The final goal is increased pulp yield with lower shives providing energy saving and minimizing sulphite doses.

BILLERUDKORSNÄS

Valmet FORWARD

Mittuniversitetet MID SWEDEN UNIVERSITY

Contacts: Henrik Edlund – BillerudKorsnäs Frövi, henrik.edlund@billerudkorsnas.com
Fredrik Lundström – Valmet AB, fredrik.lundstrom@valmet.com
Börje Norlin – Mid Sweden University, borje.norlin@miun.se

Vinnova's project No: 2020-03791 **Duration:** November 2020 -- May 2022

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

In-situ AP-XPS study of corrosion in AM steels

THE INDUSTRIAL CHALLENGE

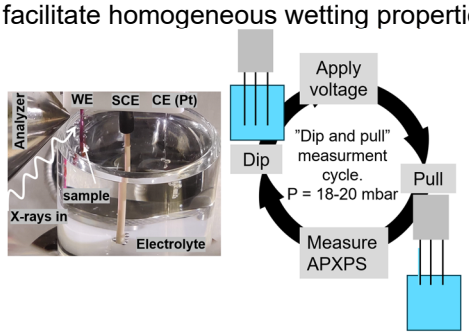
The replacement of conventional stainless steel (SS) elements with sophisticated geometry by additively manufactured (AM) components is of strategical importance for Alfa Laval, which specializes in production of heat exchangers. Quintus Technologies specializes in HIP processing of AM components. Both companies are looking for new methods able to provide a direct insight into the chemical processes accompanying corrosion in wet environments, to be able to use them for optimisation of post-processing and printing of AM materials.

WHY USING A LARGE SCALE FACILITY

The aim was to test if ambient pressure XPS (APXPS) combined with electrochemical cell can be used to complement the laboratory corrosion tests. The high brightness of the synchrotron allows to perform APXPS measurements for which the signal to the background ratio is very small. This would allow direct studies of the chemical composition of the surface in presence of thin liquid electrolyte layer.

HOW THE WORK WAS DONE

The experiment was performed at HIPPIE beamline of MAX IV in Lund. Samples were cut from AM 316L SS columns and polished to achieve surface roughness of 3 µm to facilitate homogeneous wetting properties.



The sample holder has slots for the sample, which also acts as working electrode, as well as for the SCE reference- and Pt counter electrodes. The sample holder was placed on the manipulator in the vacuum chamber which was kept at 18-20 mbar. To run the electrochemical reaction, the electrodes were dipped in the beaker with electrolyte (1M NaCl or 0.1M HCl) and the potential was applied. After a fixed time, the electrodes were pulled from the beaker and APXPS spectra were measured through the thin electrolyte film. The manipulator in the dipped position is shown in the figure together with the diagram illustrating "dip and pull" measurement cycle.

THE RESULTS AND EXPECTED IMPACT

The results revealed that APXPS can be used to follow changes in the oxide, even though the anodic potential was not applied exactly during the APXPS measurements and only thin layer of electrolyte was present on the sample surface.

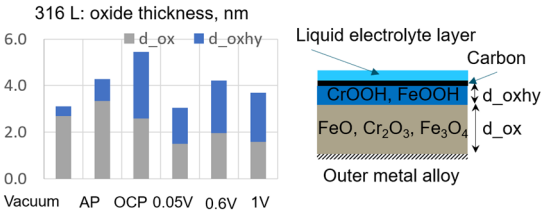


Figure above shows how the thickness of oxide changes for a sample kept at vacuum, at ambient pressure, and after contact with 0.1M HCl electrolyte at OCP followed by stepwise increase of anodic potential. The concentration of chemical species in the corresponding oxide layers was calculated. This information allows to directly relate the corrosion current to the changes in passive film composition, which is highly relevant for the industry and can be used for in-depth corrosion investigations.

"It has been a great opportunity to participate in exploring the APXPS technique, which can lead to improved understanding of oxide properties and corrosion behaviour." */D. Klint, Alfa Laval*

"Quintus Technologies looks forward to follow-up this work using HIPed material." */J. Shipley, Quintus Technologies*

Contacts: D. Klint – Alfa Laval Lund, daniel.klint@alfalaval.com
J. Shipley – Quintus Technologies, james.shipley@quintusteam.com
K. Simonov – Swerim AB, konstantin.simonov@swerim.se

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Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

In-situ study of corrosion in high strength steels by AP-XPS

THE INDUSTRIAL CHALLENGE

Traditional high strength steels (HSS) used by SSAB in waste recycling equipment are exposed to aggressive environments causing corrosion. By developing new products with different surface oxides to reduce the corrosion, the lifetime of the equipment can be significantly extended. For SSAB it is important to understand the phenomena of the passive layer of the existing and improved product during corrosion so that the composition and thickness of the surface layer can be optimized and utilized most effectively at the customer.

WHY USING A LARGE SCALE FACILITY

The high brightness of the synchrotron allows to perform ambient pressure XPS (APXPS) measurements even if the photoelectron signal is attenuated by liquid electrolyte layer and scattering on residual gas molecules. The APXPS setup with electrochemical cell enables direct study of the chemical composition of the surface in presence of thin liquid electrolyte layer directly after electrochemical reaction.

HOW THE WORK WAS DONE

The experiment was performed at HIPPIE beamline (MAX IV, Lund). Samples were cut from HX450 and HXHiAce plates and polished to achieve surface roughness of 3 µm to facilitate homogeneous wetting properties.

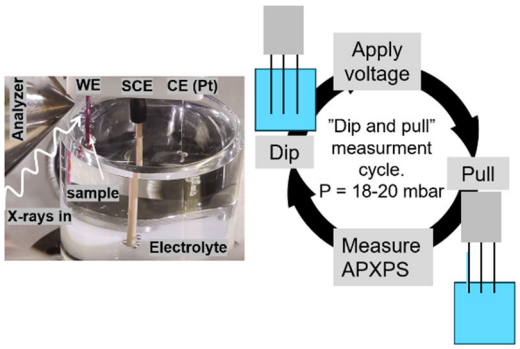
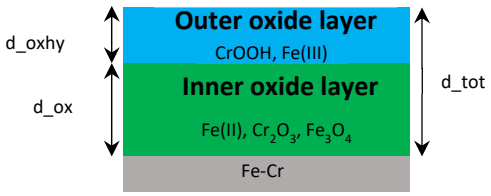


Figure. Left: The manipulator in the dipped position, Right: the "dip and pull" measurement cycle.

The sample holder has slots for the sample, which also acts as working electrode, as well as for the SCE reference- and Pt counter electrode. It was placed on the manipulator in the vacuum chamber kept at 18-20 mbar. To run the electrochemical reaction electrodes were dipped in the beaker with degassed electrolyte and the potential was applied by a potentiostat connected to manipulator. The CH_3COOH (pH 4.5) and 0.01M NaCl electrolytes were used for tests. After fixed time electrodes were pulled from the beaker and APXPS spectra were measured through the residual thin electrolyte film, while sample was at OCP conditions.

THE RESULTS AND EXPECTED IMPACT



The results revealed that the stability and composition of the oxide layer is different when the HSS surface is corroded by the weak acetic acid solution as compared to the salt solution. The surface oxide of the HSS in the pH 4.5 electrolyte increased in oxide thickness. At increasing corrosion currents, Fe_3O_4 increases as rust appears in the HX450 whereas the Cr_2O_3 and Cr hydroxide are depleted in the HXHiAce, unable to repassivate the surface. In the salt solution, both materials saw an increased thickness of the oxide layer; however, for the HXHiAce the outer oxide did not form a continuous layer and the Cr was not replenished. The results contribute to the strategic efforts of Swedish metal industry and Swerim to develop more sustainable products with longer useful lifetimes.

"Being at MAX IV provides a great opportunity to learn and use new techniques to study corrosion behaviour on surfaces. Being able to induce corrosion and study the steel surfaces in-situ is a huge leap forward."
/D. Orrling, SSAB Special Steels

Contacts: D. Orrling – SSAB Special Steels, diana.orrling@ssab.com
S.-E. Hörnström – SSAB EMEA AB, sven-erik.hornstrom@ssab.com
K. Simonov – Swerim AB, konstantin.simonov@swerim.se

Vinnova's project No: 2020-03793 **Duration:** November 2020 -- May 2022

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

HAXPES characterization of biocorrosion of additively manufactured Mg components

THE INDUSTRIAL CHALLENGE

OssDsign produces implants for improved healing of cranial defects (fig 1), consisting of a bioresorbable ceramic and an inert Ti64 mesh produced by additive manufacturing (AM). However, the full regeneration of the bone is hindered by the Ti64, which is remining permanently in the body. By replacing the Ti64 with a Mg alloy, the implant would become fully degradable, and thus lead to a complete healing of the bone. Powder extruded (PE) orthopedic screws of Mg are already clinically used, but the degradation rate of Mg processed by AM is too high. This is a problem as it can lead to premature loss of mechanical strength of the implant. To solve this problem, new routines are needed for the characterization of biological corrosion mechanisms of Mg alloys processed by AM.

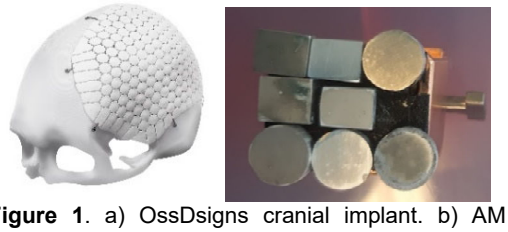


Figure 1. a) OssDsigns cranial implant. b) AM samples (circular) and PE reference samples (square) immersed in DPBS.

WHY USING A LARGE-SCALE FACILITY

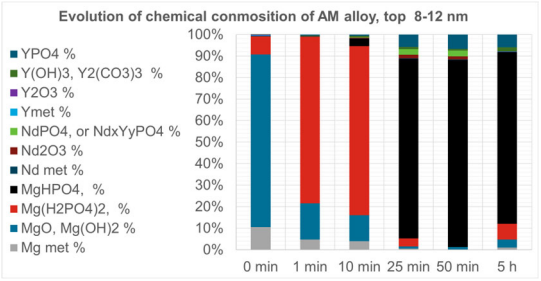
The synchrotron based hard X-ray photoelectron spectroscopy (HAXPES) offers great capability for studying the chemical species forming as a results of corrosion reaction between the alloying elements (Mg, Y, Nd) and the media used. Thanks to the high photon energy corrosion layers up to 30 nm can be analysed. Due to the very high brightness a good signal to noise ratio can be achieved in short time.

HOW THE WORK WAS DONE

The HAXPES measurements were carried out ex-situ at beamline P22 at Petra III (Hamburg, Germany) using two different energies (2.5 keV and 7.5 keV. Orthopedic screws of powder extruded (PE) Mg alloy WE43 are already used clinically and served as reference samples. 16 samples produced by AM and PE were prepared by grinding down to 2500 grit (fig 1) and cleaning with ethanol and acetone. To mimic the salt concentrations of the body they were immersed between 1 min and 24 hours in Dulbecco's Phosphate Buffered Saline (DPBS) solution, with salt concentrations corresponding to the ones found in the body.

THE RESULTS AND EXPECTED IMPACT

Quantitative analysis of the HAXPES spectra clearly show reduction of material in its metallic state on the surface of both the AM and PE samples. Contact with DPBS for less than 10 min leads to the formation of oxides and hydroxides of Mg, Y and, to less extent, also Nd. After 20 min we obtained rapid growth of an inhomogeneous compound layer with thickness of more than 30 nm composed of mainly Mg phosphates and Y phosphates.



The composition of compound layer and dynamics of its formation were slightly different for AM and PE materials. Such observations became possible only thanks to the variable surface and high chemical sensitivity of HAXPES and opens up to study the evolution of the surface of more metallic materials used in implants.

"HAXPES provides an impressive level of detail in the evaluation of surface chemistry. Information that is very important to understand how implant materials will interact with the body."
/Jonas Åberg, Regulatory Affairs Manager, OssDsign

OSSDSIGN®

SWERIM

Contacts: Jonas Åberg – OssDsign, ja@ossdsign.com
Konstantin Simonov – LSI Expert at Swerim AB, konstantin.simonov@swerim.se
Hanna Nilsson Åhman – Swerim AB/Uppsala University, hanna.nilsson-ahman@swerim.se

Vinnova's project No: 2020-03794 **Duration:** May 2020 – September 2022

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.

INDUSTRIAL PILOT PROJECT

In situ X-ray diffraction during heat treatments to guide machine learning modelling of phase transitions in steels

THE INDUSTRIAL CHALLENGE

A recent development in steel modelling is machine learning (ML), a sub-branch of artificial intelligence (AI). ML is an important complement to physical based modelling methods, and some steel phenomena can only be modelled using a data-driven approach like ML. Ferritico develops ML models and software to predict phase transformations during heat treatments, so-called continuous cooling transformations (CCT), which is very difficult to predict accurately using any physical model. The modelling of CCT is critical input data for thermomechanical FEM simulations in process and product development, i.e. for case hardening and tool steels.

WHY USING A LARGE SCALE FACILITY

ML is a datadriven method and thus the CCT data must be of excellent quality. This can only be ensured through in situ measurements during real heat treatments. Traditionally this is performed indirectly by dilatometry, but the only viable measurement for direct in situ measurements is time-resolved synchrotron x-ray diffraction (SXRD). Such measurements would add key data for the modelling of CCT and provide excellent validation of the modelling.

HOW THE WORK WAS DONE

The experiments were conducted at the P07 beamline of Petra III, Hamburg. The samples were heated to austenitization temperatures, to fully transform the steel to the austenite phase, followed by continuous cooling at different rates and at different stages to investigate e.g. the effect of a rate change at low temperatures for the martensitic transformation. Considering the rapid heating and cooling applied, we needed to ensure time resolutions in the order of milli-seconds, about 50 ms, which means that the Pilatus detector was used. The evolution of the carbon diffusion was investigated by studying the peak shifts for the fcc and bcc phases. In addition, dislocation densities were possible to eliminate by the use of the Williamson-Hall and warren Averbach methods.

THE RESULTS AND EXPECTED IMPACT

The project successfully monitored and measured the volume fraction evolution of the ferrite, martensite as well as precipitate phases. The extremely precise in situ SXRD measurements provided high quality data to further refine the precision of the steel heat treatment simulation ML models. The data also enabled validation to calibrate the current version of the models and gave insights to where to focus future work of Ferritico ML model development.

Phase transformation temperatures

The aggregated average error for simulation of the temperatures on which transformation of the phases Ferrite, Pearlite, Bainite and Martensite are starting and finishing respectively. The benchmark is based on empirically measured CCT diagrams for 14 steel grades (some chemical compositions listed below) that are not included in the machine learning database and where benchmarking CCT data is available for 8-12 cooling rates. In the table, _s and _f refer to the start and finish temperature of the transformation of a phase, e.g. Fe_s being Ferrite start temperature.

	Ferritico error	Competitor error
Fe_s	34.9 °C	49.0 °C
Fe_f	45.7 °C	77.5 °C
P_s	28.4 °C	68.0 °C
P_f	55.2 °C	117.1 °C
B_s	53.6 °C	74.3 °C
B_f	40.1 °C	93.4 °C
M_s	39.6 °C	47.8 °C

The so far unprecedented model performance for CCT predictions has been confirmed with the high quality SXRD data that has been compared with the ML model simulation.

"The project and the generated SXRD data will have significant impact on steel simulation software performance and support industry transition from trial-and-error process and product development to a data-driven development process" /Claes Holmström, Ferritico

ferritico

RI.SE Research Institutes of Sweden

Contacts: Claes Holmström – Ferritico claes.holmstrom@ferritico.com
Albin Stormvinter – RISE, albin.stormvinter@ri.se

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INDUSTRIAL PILOT PROJECT

Facilitating improved hole-transporting interlayers in organic solar cells using synchrotron radiation techniques

THE INDUSTRIAL CHALLENGE

Epishine AB is a young company (founded 2016) and is currently producing light-energy harvesting modules that harvest indoor light aimed at supporting low power devices. The long-term plan is to expand the product portfolio into many other solar cell applications.

Organic solar cells have undergone tremendous improvement during the last four years and now reach power conversion efficiency values of over 19% for single cells (AM1.5) and over 26% at indoor lighting conditions. However, continued advances are still needed to improve cell stability. For this improving the selective charge transporting interface layers between the light absorbing organic semiconductor blend layer and the electrodes is important. This project aims to facilitate the development of improved hole-transporting interlayers.

WHY USING A LARGE SCALE FACILITY

Synchrotron facilities enables the use of x-ray photoelectron spectroscopy (XPS) over a wide range of photon energies, not available at a typical local lab, and can also offer such experiments combined with oxygen/air exposure. This enables non-destructive depth profiling of the hole-transporting interlayers and the mapping out of vertical concentration gradients of the molecular components. In addition, the chemical modification of the interlayer upon exposure to air can be determined.

HOW THE WORK WAS DONE

We used the HIPPIE beamline at MAX IV that enables XPS over a wide range of photon energies as well as measurements under controlled exposure to gases such as air. We measured pristine interlayers on flexible foils from the production line (cut to ~1x1 cm^2 samples) as well as interlayers exposed to thermal stress, and tracked the vertical concentration gradient of the molecular components using elemental core level spectra taken at different photon energies. To study the possible effects of air exposure, we studied pristine never-exposed-to-air interlayer films inserted using a glove bag, as well as films that were exposed to air for different durations. Chemical and concentration changes were tracked by core level spectra.

THE RESULTS AND EXPECTED IMPACT

The results showed that molecular concentration gradients in the hole-transporting interlayers exist already before exposure to thermal stress. The concentration gradients, however, change upon thermal stress as well as after long-term storage at room temperature.

During short-term exposure to air (in situ), no detectable chemical changes occur, while new chemical bonds with oxygen can be detected after long-term exposure (ex situ).

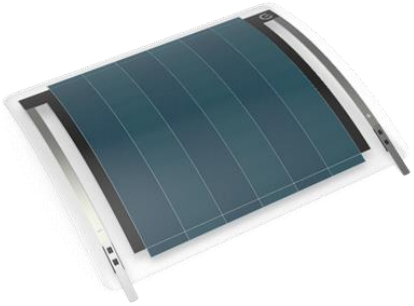

Figure. Examples of a curved solar cell device by Epishine AB.

"It is important for us to understand how our material is built up and how interact with the environment – HIPPIE provides the possibility to investigate this"
/Thomas Österberg, Epishine AB

epishine harvesting light

LINKÖPINGS UNIVERSITET

Contacts: Thomas Österberg – Epishine AB, thomas.osterberg@epishine.se , www.epishine.com
Mats Fahlman – Linkoping University, mats.fahlman@liu.se

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INDUSTRIAL PILOT PROJECT

In operando studies of Ru(III) and Ti(III) oxidation at calcination of electrode coatings

THE INDUSTRIAL CHALLENGE

Ruthenium dioxide (RuO_2) or mixtures of RuO_2 and other metal oxides (TiO_2, IrO_2, etc.) have already for decades successfully been used as catalytic and durable electrode coatings in different industrial processes (such as chlorine, oxygen, or hydrogen gas production). The RuO_2-based electrode coating is deposited on a titanium (Ti) or nickel (Ni) support and consists of ~20 nm nanoparticles formed in a calcination process where precursor solutions of Ru(III) oxidizes in air to Ru(IV). The knowledge about the oxidation process is however very limited, especially in the view of large-scale industrial production, which prohibit R&I regarding further optimization of the calcination process in the production of efficient and energy saving electrodes. This is mainly because of experimental difficulties to study the oxidation process *in operando*.

WHY USING A LARGE SCALE FACILITY

X-ray photoelectron spectroscopy (XPS) is an element sensitive technique that can provide chemical-specific information about the probed elements in a sample. It is, thus, possible to follow the oxidation of Ru during the calcination process while it occurs. The formation of RuO_2 require oxygen gas and performing experiment in an O_2-gas mixture in turn require a high X-ray intensity that can only be obtained in a synchrotron radiation (SR) facility. In addition, the high X-ray intensity facilitates fast data acquisition necessary for in operando measurements. It is also possible to obtain the resolution needed to observe the small binding energy shifts in the XPS spectra during the calcination process. Hence, this study would be impossible using conventional XPS.

HOW THE WORK WAS DONE

An initial study was performed using a conventional XPS system, which provided information needed to select suitable samples prepared at the Permascand research lab. The *in operando* study was part performed at the ambient pressure XPS beamline HIPPIE at MAX IV in Lund.

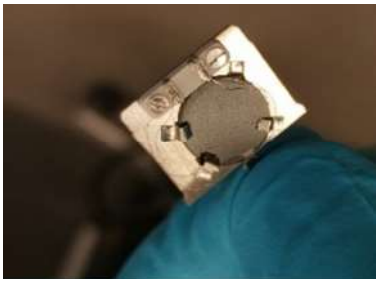

Figure 1. A circular coated electrode mounted in a sample holder, ready for SR-XPS at MAX IV.

THE RESULTS AND EXPECTED IMPACT

As displayed in Figure 2, RuO_2 is formed at 370 °C. However, a temperature of 475 °C was needed to obtain an electrocatalytic active coating. The experience has provided new perspectives on the calcination process and valuable inputs for the optimization process.

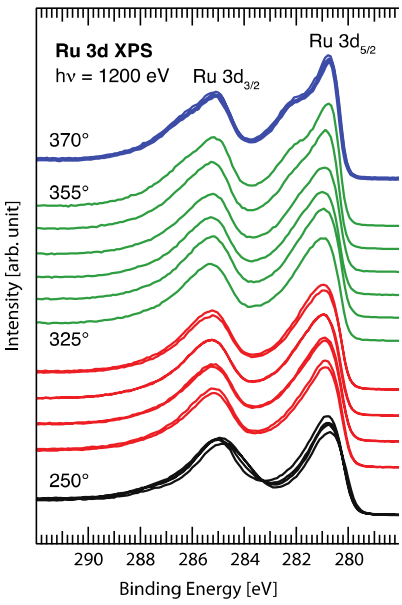

Figure 2. Ru 3d XPS of the transition between Ru(III) and Ru(IV) forming RuO_2-based coating at 325-355 °C (green spectra).

"We see a great value in the results for our future product development"
/Fredrik Herlitz, CTO, Permascand AB

Uppsala Synchrotronix AB

PERMASCAND

Contacts: Susanne Holmin – Permascand AB, susanne.holmin@permascand.com
Robert Moberg – Uppsala Synchrotronix AB, robert@synchrotronix.se

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