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.

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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.

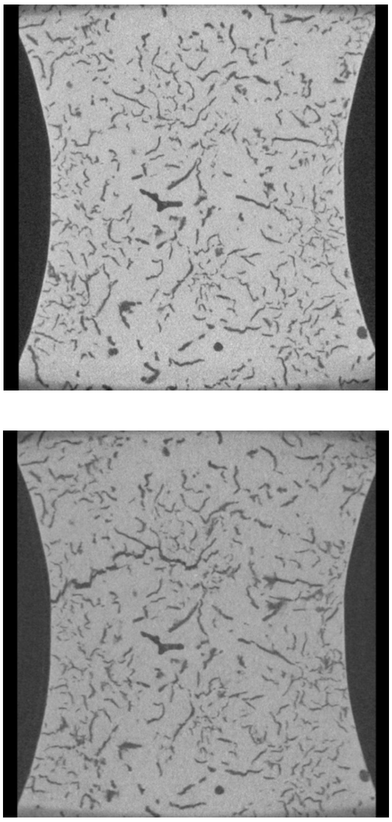

Figure. 2D slices extracted from the 3D tomography Images of the same sample with (lower) and without (upper) an initiated fatigue crack.

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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 EMEA 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.

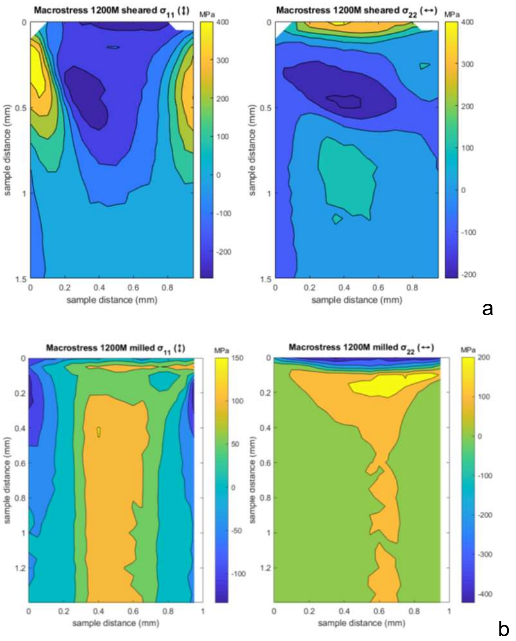

Figure. The constructed macro strain maps calculated by the determined cell parameters in the north (σ11) and east (σ22) direction for the a) sheared sample and b) sheared and milled sample.

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***

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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.

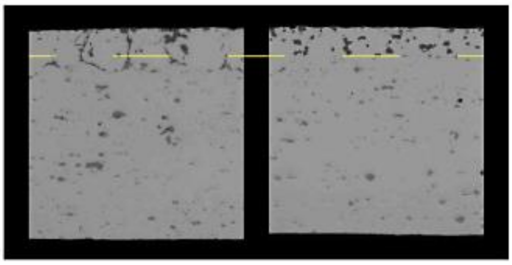

Figure 1. Aluminium sheet with a braze clad before brazing (right) and after brazing (left). The dashed yellow line highlights the original boundary between the aluminium core and the braze clad.

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.

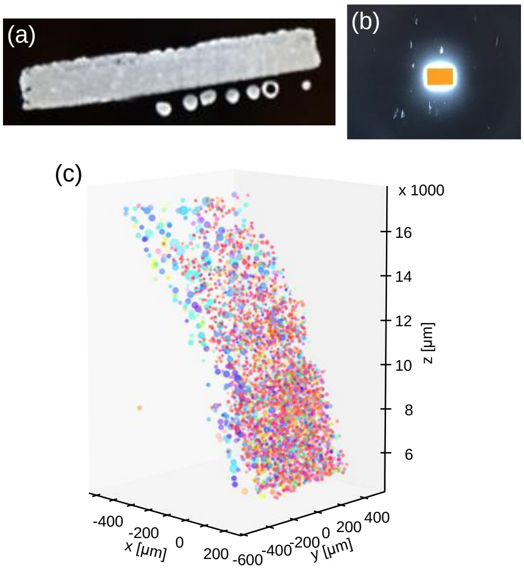

Figure 2. (a) a slice from the tomography imaging at SPring-8. The balls under the aluminium sample are glass beads that make it easier to identify positions in the samples. (b) the DCT data where every bright spot around the centre is a diffraction-projected grain image with a certain orientation. (c) 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***

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