EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

ESRF	**Experiment title:** **In-situ observations of pitting and intergranular stress corrosion crack nucleation and growth**	**Experiment number**: ME577
Beamline: ID19	**Date Of Experiment**: From: 16 April 03 To: 19 April 03	**Date of report**: Dec 03
Shifts:9	**Local contact(s)**: Dr Elodie BOLLER (e-mail: boller@esrf.fr)	*Received at ESRF:*

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Report:

Abstract

High resolution, synchrotron, X-ray tomography has been demonstrated as a tool for observing pitting, intergranular corrosion and intergranular cracking. Exemplar studies have investigated pitting and intergranular cracking in 5083 aluminium alloy and intergranular cracks in sensitised 302 stainless steel. High resolution X-ray tomography has potential applications for the in-situ study of pitting and the transition from pitting to cracking in stainless steels, in particular the effects of near-surface residual stress.

Aims of the experiment and scientific background

Aim: The first in-situ tomographic observations of pitting and intergranular stress corrosion crack nucleation and growth.

Objective: To perform tomographic observations of stress corrosion cracking for a model system and preliminary observations for 304 stainless steel.

Background: Stress corrosion cracking and corrosion fatigue cause failures in the safety critical aerospace and nuclear industries. Cracking frequently nucleates from corrosion pits, and depending on the material and environment may be transgranular or intergranular. The incubation period prior the development of a critical crack nucleus depends on several factors. These include the rate of pit formation and growth, the transition from pit to crack, and propagation of a **short crack**, with a size comparable to the microstructure scale. Although models for these processes have been developed, the prediction of incubation periods remains uncertain, due to the complex interaction between microstructure, environment and the mechanisms of pitting and cracking.

Resistance to intergranular corrosion and cracking can be affected by the distribution of grain boundary crystallographic character. The grain boundary character may also indirectly affect intergranular failure via solute concentrations and precipitation. The geometry of the grain boundaries junctions may also impede crack propagation. **Grain boundary engineering** aims to improve the resistance to intergranular stress corrosion cracking by disrupting easy paths through the microstructure. The interactions between the crack and the microstructure are expected to be most significant when the crack is a short crack. However, very little is known about the behaviour of short stress corrosion cracks due to experimental difficulties in monitoring their nucleation and growth.

This project aimed to make the **first in-situ tomographic observations of intergranular stress corrosion crack nucleation and growth**. These unique observations will make a significant contribution to our understanding of intergranular stress corrosion cracking. This work will ultimately lead to more reliable predictions of stress corrosion crack incubation periods, and improvements in cracking resistance through grain boundary engineering.

Experimental method

Two materials were selected for investigation. These were aluminium alloy 5083, sensitised by ageing at 150ºC for 7 days and 302 stainless steel, solution treated at 1050ºC for 30 minutes and sensitised for 1 hour at 650ºC. The aluminium samples were machined from 5 mm plate after sensitisation. The tensile specimens had a 3 mm diameter, reduced to 1 mm in the gauge length. The specimens were lacquered, except for a gauge length of approximately 0.7 mm, resulting in an exposed area of approximately 0.02 cm^2. The stainless steel samples were in the form of 0.4 mm diameter wire. The wire was spot-welded to 3mm diameter steel grub screws to form a tensile specimen, which was then lacquered to expose a gauge length of approximately 0.5 mm. The specimens were tested under load in an environmental cell on the ID19 stage (Figure 1). The experiments produced approximately 200 Gb of raw data. A full analysis has not yet been completed. Preliminary observations are reported below.

Aluminium Alloy 5083

The sensitised aluminium alloy 5083 was selected for the study of intergranular corrosion. The specimens were loaded in tension in 3.5% NaCl solution. The open circuit potential (OCP) was approximately 770mV SCE. In-situ observations of intergranular corrosion, pitting and cracking were performed at an initial stress of approximately 220 MPa. A gauge length of less than 0.7 mm, with diameter 1 mm was exposed to the test solution. Tomographic data was recorded after intervals of exposure to the pitting potential, which was maintained at –740mV SCE, monitored using the miniature Ag/AgCl reference electrode in the environmental cell. During the tomographic observations, the electrochemical potential and the tensile stress were reduced to suspend pitting and cracking. Typically, the potential was reduced to below -1200mV SCE, with the tensile stress below 100 MPa.

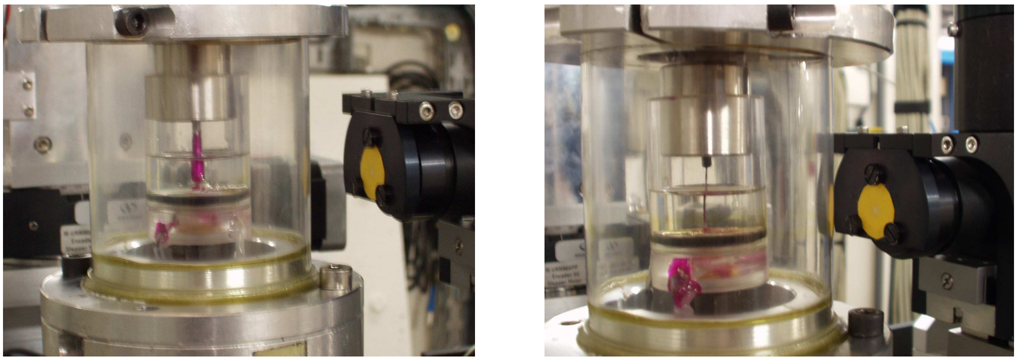

Figure 1: Aluminium alloy (left) and unloaded stainless steel wire sample (right) in the environmental cell. The detector is to the right of the image.

The analysis of the aluminium tomographic data has not been completed yet due to the large quanitity of data. The data includes a sequence of observations to observe the gradual development of corrosion damage and cracking. Preliminary results are shown in Figure 2, which shows a two-dimensional axial section of the data set after 4 hours exposure at –740mV SCE. Pitting can be observed to propagate from the exterior surface. Reconstruction of the data (Figure 3) to show the development of intergranular corrosion and cracking. Further analysis of the data is in progress to quantify the development and coalescence of pitting, and to evaluate the rate of crack development. The preliminary analysis of data from these experiments confirms that high resolution synchrotron tomography can be used to observed the development of pitting and cracking in aluminium alloys.

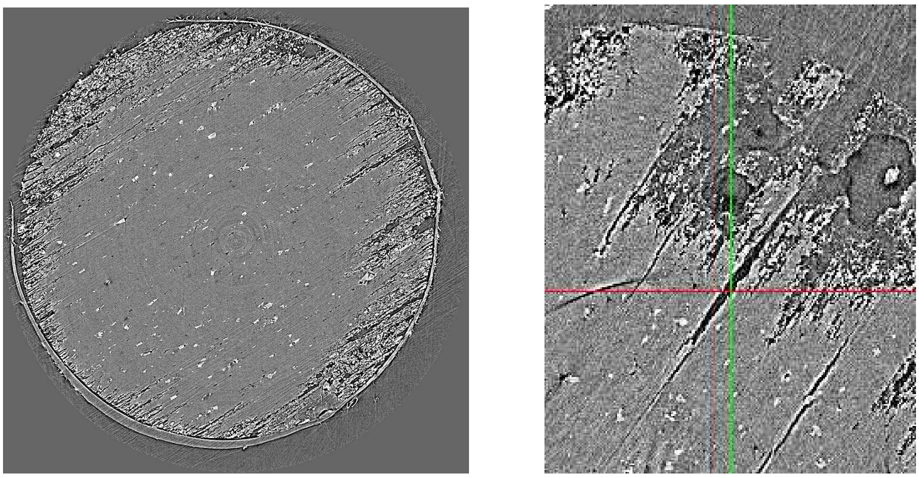

Figure 2: Section of tomographic data for aluminium sample. The image to the right shows crack propagation, nucleated at intergranular corrosion pits. Note the precipitates in the alloy are imaged. The sample diameter is 1mm.

Stainless Steel

The feasibility of using X-ray tomography to study pitting and stress corrosion crack development in an austenitic stainless steel was also investigated. Preliminary experiments established suitable test conditions for intergranular corrosion and intergranular stress corrosion cracking. Intergranular corrosion and cracking can be induced in 0.15 M potassium thionate ($K_2S_4O_6$), pH 2.5 at low stress levels in 302 stainless steel. Isolated regions of ductile failure were observed, which may correspond to crack resistant low angle grain boundaries (Figure 4).

Cracking was initiated by coupling the sample to a platinum working electrode for a few seconds to polarise the stainless steel wire anodically. Preliminary experiments demonstrated that cracking would then occur under open circuit conditions, and could be suspended by simultaneously reducing the load and coupling the stainless steel wire to a pure aluminium electrode to impose a cathodic polarisation. Cracking re-initiated when open circuit conditions were restored.

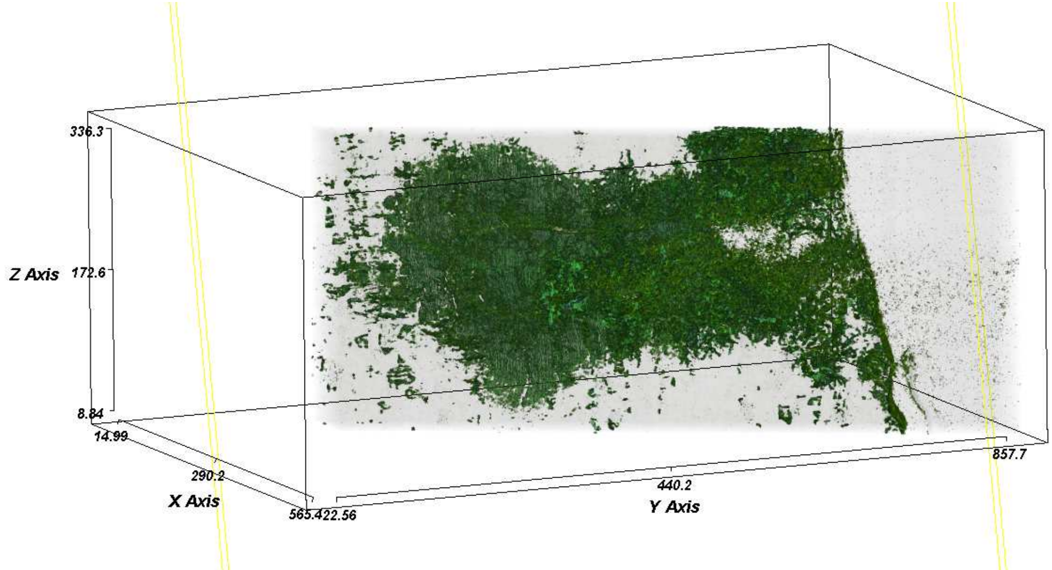

Figure 3: Three dimensional reconstruction of a part of the corrosion/cracking of the aluminium sample, observed in Figure 2. An intergranular crack has nucleated from corrosion pitting . The intergranular corrosion/cracking propagates from surface (right) to left right. The depth of cracking is approximately 200µm.

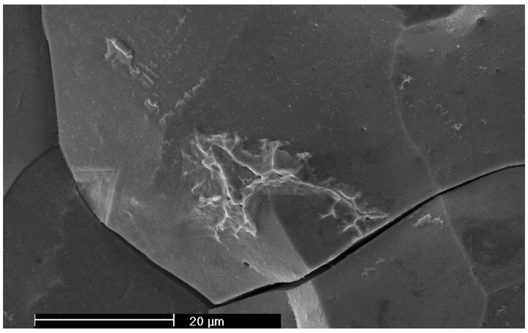

Figure 4: Isolated region of ductile failure in sensitised 302 stainless steel wire, tested at 200 MPa

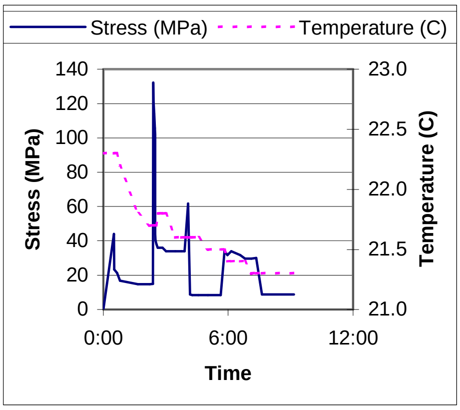

Figure 5: Variation of applied stress and temperature during stress corrosion experiment with stainless steel wire.

Tomographic observations were made with the load reduced and the sample coupled to the aluminium electrode to prevent cracking during the observations. Cracking occurred rapidly on re-application of the stress, which was progressively reduced as cracking was observed (Figure 5). The initiation of cracking and specimen failure due to coalescence was recorded in samples sensitised for 1 hour at 650ºC (Figure 6, Figure 7).

Full analysis of the data is currently in progress. Preliminary analysis confirms that the cracks may be observed using tomography. Figure 8 shows the coalescence of cracks in the latter stages of sample life.

Analysis of two-dimensional sections of the data implies that the crack is discontinuous. However, three-dimensional visualisation of the same region shows that the crack is, in fact, continuous (Figure 9). The apparent discontinuities are observed as “holes” in the crack (Figure 10), which implies that crack bridging occurs. Post-mortem analysis of the fracture surface is in progress to correlate the bridging ligaments with ductile regions (e.g. Figure 4). Detailed analysis of the data will investigate the development of bridging ligaments.

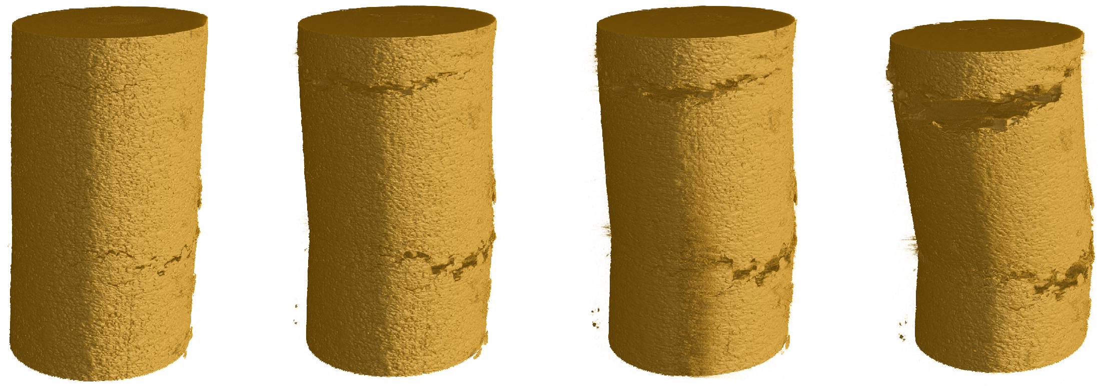

Figure 6: Tomographic reconstructions of the sample, showing failure of 302 stainless steel wire (diameter 0.4mm, sensitised for 1 hour) due to initiation and coalescence of intergranular stress corrosion cracks. Wire diameter, 0.4mm.

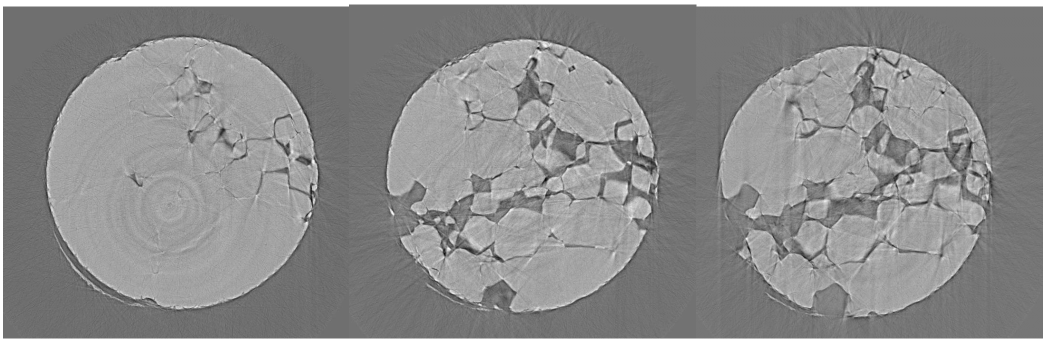

Figure 7: Sequence of axial sections at the same location in the wire, demonstrating the growth and coalescence of intergranular cracking (302 stainless steel, sensitised). Wire diameter 0.4 mm.

Further Work

Full analysis of the data obtained is in progress. In particular, the development, propagation and coalescence of pitting/intergranular corrosion in aluminium and cracking in stainless steel will be studied. The results reported demonstrate that tomography is a suitable tool for observations in both aluminium and stainless steel.

Summary

- High resolution, synchrotron, X-ray tomography has been demonstrated as a tool for observing pitting, intergranular corrosion and intergranular cracking.
- In-situ observations of pitting and intergranular cracking in 5083 aluminium alloy have been obtained. These show the progressive development and coalescence of intergranular corrosion cracks within the bulk of the sample.

- In-situ observations of intergranular cracks in sensitised 302 stainless steel have been obtained. These provide evidence for crack bridging ligaments, which may be caused by low angle grain boundaries.
- Further analysis of the data (>200Gb) is in progress.
- High resolution X-ray tomography has potential applications for the in-situ study of pitting and the transition from pitting to cracking in stainless steels, in particular the effects of near-surface residual stress.

Figure 8: Segmented image of intergranular cracking in the 302 sensitised stainless steel wire (diameter 0.4 mm).

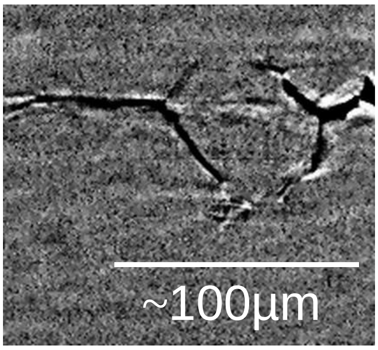

Figure 9: Intergranular cracking observed within the specimen.

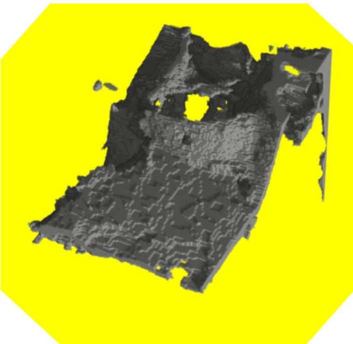

Figure 10: Three-dimensional reconstruction of the region in Figure 9. The "hole" in the centre of the crack implies that crack bridging occurs.