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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **In-situ diffraction and phase-contrast X-ray tomography to investigate the fatigue mechanisms for a nickel base superalloy**	**Experiment number**: in813
Beamline: I11	**Date of experiment**: from: 22/11/2012, 8:00 to: 22/11/2012 24:00	**Date of report**: May 2, 2013
Shifts: 2	**Local contact(s)**: Andrew KING	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Olivier MESSE, Department of Materials Science, University of Cambridge, UK

Joel LACHAMBRE, MATEIS, INSA Lyon, FRANCE

Jean-Yves BUFFIERE, MATEIS, INSA Lyon, FRANCE

Cathie RAE, Department of Materials Science, University of Cambridge, UK

Report:

The aim of the experiment was to investigate fatigue crack propagation mechanism in RR1000, a nickel base superalloy used for turbine disc applications and exihibting a high twin density through in situ X-ray tomography.

In this alloy, the short crack growth sensitivity to the microstructure necessitate the alloy to be fully characterise in 3D. To the end, diffraction contrast tomography (DCT) technique have been used before starting the fatigue experiment in order to characterise the initial 3D microstructure in the vicinity of the notched area where the crack should initiate. The specimen was then placed into the machine to be fatigued and phase constrast tomographs (PCT) have been recorded at regular intervals where the crack visibly advanced.

Dog-bone shaped test specimen have been fabricated from coarse grained RR1000. The central 4mm of the sample was electropolish to produce a rounded cuboidal gage volume with a maximum transversal length of ~800µm which enables us to image the entire volume at 60keV. During the experiment, we recorded two volumes at the notch location and just below, composed of ~5000 grains (including twinned grains). Following the initial characterisation of the microstructure, the specimen was then fatigued at room temperature. The test was carried out at 830MPa maximum stress under constant stress amplitude with a ~0.1 stress ratio and a frequency of 20Hz. The fatigue test was interrupted 18 times where a tomograph was recorded under load (650 MPa) to ensure the maximum contrast for the tomographic imaging. The specimen was fatigued to failure.

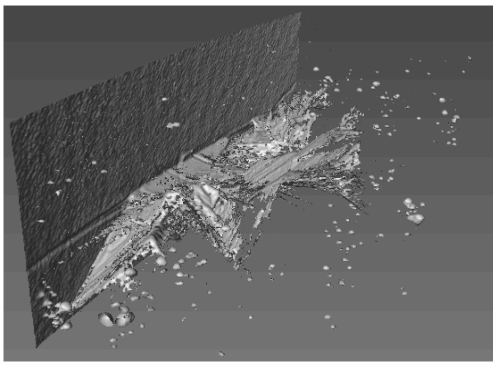

Figure 1: Fatigue crack from the notch after 195864 cycles. Pores are also visible in this subvolume.

All the PCT tomographs have been reconstructed and allow the evalution from the crack originated from the notch as well as the presence of numerous pores inside the volume associated with the powder processing route employed to fabricated this specimen (Figure 1). DCT volumes

from the two dataset have also been reconstructed and are consistent with EBSD maps taken at the surface of the fracture specimen.

Post-mortem analysis revealed that the specimen did not failed from the notch, but instead failed from a well oriented set of grains. The primary crack was located inside the electropolished gauge volume, but outside of the field of view of the tomographs. The primary crack shows an analogous fracture mechanism to the crack initiated from the notch, i.e. highly faceted crack path highlighting the sensitivity to the microstructure (Figure 2).

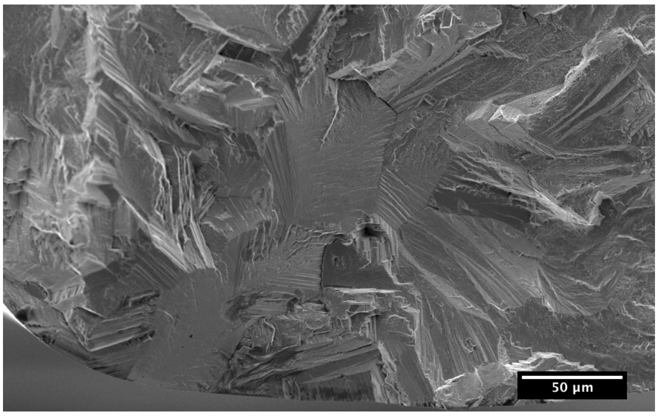

Figure 2: Primary fatigue crack. The initiation site is located at the lower left of the SE image.

EBSD analysis revealed that the primary crack has initiated from a boundary between two grains. The grain boundary type is very close to a twin boundary by failed to be exactly equal to 60° due to the fact that the EBSD data were collected post mortem after the fatigue test, although they evidently share a common (111) plane – A1 and $A1_{twin}$.

The presence of the twin boundaries was futher infered by the result obtained from a FIB-TEM specimen sampled from this location. The boundary possess diffraction pattern characteristic of a Σ3-twin boundary.
This evidence together with the EBSD and SEM images show a clear evidence that the specimen crack initiation site occurred by cleavage of the grain along its twin boundary.

The imperfections visible on the faceted grains is due presence of ledges within the twin boudary plane (not mobile at r.t.) together with the presence of carbides which locally disrupted the cleavage.
Serrated crack growth has also been observed at the surface and is due to the activation of two slip systems close to 45° to the loading direction, but their combination enable the crack to preserve a global growth perpendicular to the loading direction where the release stress rate is maximized.

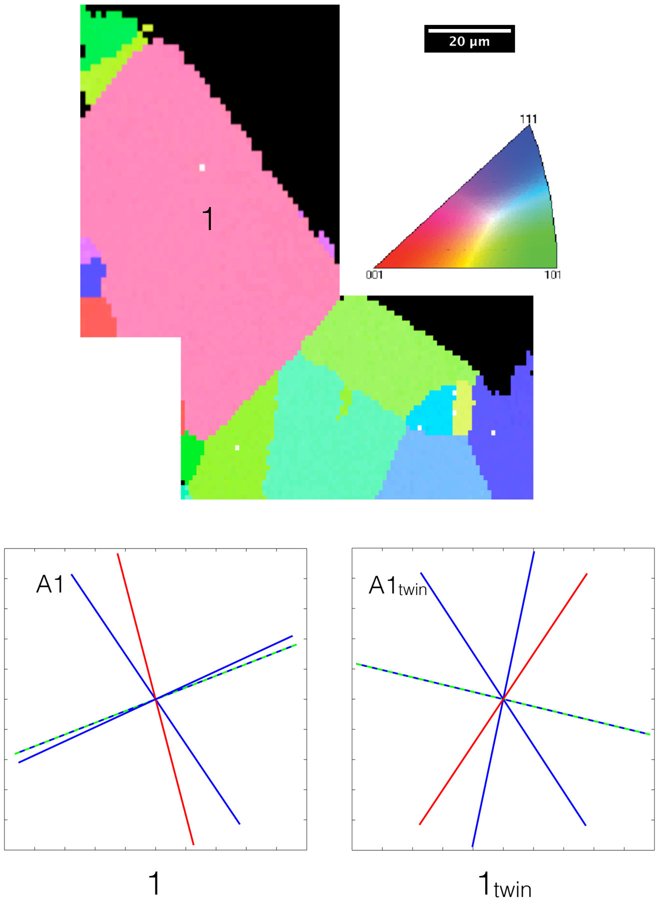

Figure 3: EBSD maps from the side of the initiation site (Figure2), and associated slip traces from grain 1 and twin on the other side. The red line identify the slip trace with the highest Schmidt factor, while the dash green line; the slip system with the highest resolved shear stress

Work is currently underway to improved the crack resolution in order to accurately evaluate its characterisation relative to the loading direction and grain orientation.

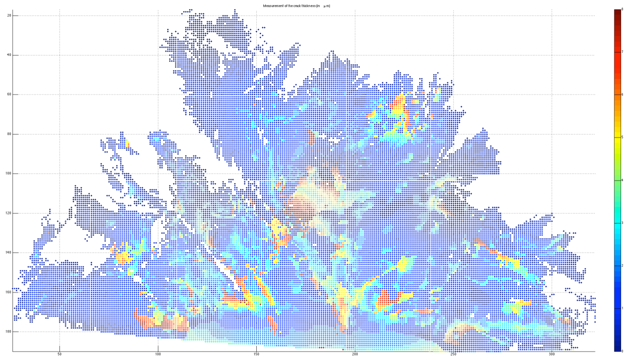

Figure 4: Measurement of the crack thickness along Z-direction

Additionally, the DCT volumes will be used to evaluate the fatigue crack initiation in high cycle fatigue in a realistic microstructure.