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Experiment Report Form

ESRF	**Experiment title: Three-dimensional characterisation of the networks of grain boundary planes in austenitic stainless steel**	**Experiment number**: MA830
Beamline: ID11	**Date Of Experiment**: From: 09 June 2010 To: 14 June 2010	**Date of report**: Sept 11
Shifts: 15	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

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Report:

Background

The strain path in the thermo-mechanical history can have a significant effect on the frequency of grain boundary planes with special characteristics in austenitic stainless steels. The clustering and connectivity of these boundaries are also affected by thermo-mechanical processing, and this has a direct effect on the local resistance of the microstructure to stress corrosion cracking.

This heterogeneity is because the re-arrangement of grain boundary structures during re-crystallisation is driven by the local distribution of stored plastic strain energy, which in turn is influenced by the anisotropy of slip and the local orientations of grains and grain boundaries. This heterogeneity is important, as it can determines the local likelihood of crack nucleation and resistance to crack propagation. These have a direct effect on lifetime of materials in aggressive environments. Designing new materials or processes for fabricating components with improved resistance requires knowledge of the sensitivity of critical aspects of the microstructure to factors such as the strain paths during thermo-mechanical processing.

Two-dimensional analysis (i.e. EBSD- Electron BackScatter Diffraction) and stereology cannot provide information on the clustering behaviour of grain boundary plane structures, and this can only be achieved by three-dimensional studies. Although serial sectioning and 3D reconstruction by focussed ion beam milling provide the necessary resolution to characterise grain boundary structure, it is inadequate for the large numbers of grains that are needed to describe grain boundary cluster characteristics. More traditional metallographic sectioning methods have insufficient resolution. Diffraction Contrast Tomography is ideal, as it allows very large numbers of grains to be studied with sufficient resolution to describe grain boundary structures. The objective of this experiment was to use Diffraction Contrast Tomography to characterise the grain boundary plane distributions in an austenitic stainless steel, as a function of the strain path and annealing time.

Experiments Performed

A total of 24 scans were performed, each collecting DCT data in a volume of approximately 0.3 x 0.3 x 0.5 mm (Table 1), from 3 microstructures of similar grain size, in which prior EBSD characterisation had been conducted. Attention was focussed on samples H2 and 13, which differ in their extent of multiple twinning for similar grain size, to provide a sufficiently large number of grains for statistical analysis. Other microstructures (e.g. “4_”) exhibited too much mosaicity for characterisation.

Example results are shown in Figure 1, which illustrates the successful mapping of microstructure, and the characterisation of grain boundary types, using the coincidence site lattice model.

Table 1: Summary of samples and scans obtained

Name	Processing	EBSD results	Remarks
B2_	5% strain/1050° C/1 hour	37 μm grain size (41%Σ3, 18%Σ9&Σ27)	4 scans
H3_	5% strain/1050° C/24 hours	45 μm grain size (32%Σ3, 8%Σ9&Σ27)	10 scans
13_	20% strain/1050° C/1 hour	40-50 μm grain size (46%Σ3, 2%Σ9&Σ27)	10 scans
4_	10% strain/950°C /1 hour	70 μm grain size (56%Σ3, 1%Σ9&Σ27)	Too much deformation for standard DCT

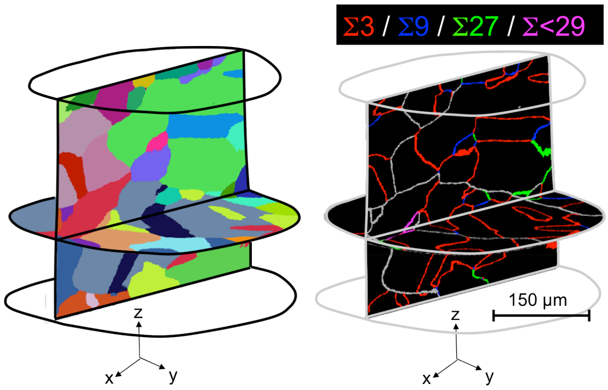

Figure 1: Reconstruction of grains and grain boundaries by DCT, (from sample B2)

Current Status of Analysis

The experiment has been successful, with the collection of data with a significant number of grains (estimated to be around 1000 grains in each of H3 and 13), which will be sufficient for statistical analysis og microstructure. However, the data are only partially analysed due to shortage of personel. The principal investigator (JM) has moved from Manchester to Oxford and currently has no researcher available to complete the data analysis. A visiting scientist at Oxford is expected to complete the analysis, in collaboration with AK and LW at ESRF, in the summer of 2012.

Twinned microstructures present specific challenges to all graintracking type techniques (DCT, 3DXRD). Because of the symmetry relations between a twin and its parent grain, some diffraction vectors are identical, while some are different. This means that diffraction spots may correspond to (a) only the parent grain, (b) only the twin, or (c) both combined. Further difficulties may also arise from the plate-like morphology of typical twins. Extensively twinned microstructures have been reconstructed with the current DCT algorithms, as shown in figure 1. However, post-test characterisation of samples, using FIB-milling and EBSD needs to be done to validate the results achievable.

Further development of techniques for reconstructing such microstructures will be done as part of the PhD project of Laura Nervo (jointly funded by Manchester and ESRF).

A paper will be prepared, presenting the comparison with the EBSD characterisation of grain boundary character distribution, and describing the population of grain boundary planes.