ESRF	**Experiment title:** Investigation into iodine-induced stress corrosion cracking in zirconium metal	**Experiment number**: MA2274
Beamline: ID11	**Date of experiment:** from: 23/1/2015 to: 29/9/2015	**Date of report:** December 2016
Shifts: 12	**Local contact(s):** Nicholas Harker	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
Dr Philipp Frankel* (University of Manchester)
Dr Alistair Garner* (University of Manchester)
Dr Imran Bhamji* (University of Manchester)
Conor Gillen* (University of Manchester

Report

With the need to reduce the restrictive operating procedures imposed on nuclear reactors in order to allow for improved power maneuverability associated with the increased use of renewable energy, a better understanding of the mechanism of stress corrosion cracking (SCC) of Zr alloys, used as cladding material in nuclear reactors, is required. This aim of this experiment was to characterise the microstructure of a commercial zirconium alloy (Zircaloy-4) in 3D using diffraction contrast tomography (DCT). After characterisation, a series of small, notched tensile samples were to be loaded in-situ using a tensile rig corrosive iodine environment in order to induce a stress corrosion crack. The crack initiation and propagation could then be tracked in situ through the internal structure using phase contrast tomography. The correlation of the PCT and DCT datasets would allow the interaction of the crack with the microstructure to be studied in 3D, which has not previously been performed on Zr alloys. This would allow for new insight into the mechanism of stress corrosion cracking of Zr alloys in nuclear reactors.

Due to experimental constraints, it was only possible to perform the DCT characterisation part of the experiment during our 12 allocated shifts. Due to the volatile nature of iodine gas, it was unfeasible to have it in contact with the tensile rig, as it would attack the components. However, the successful acquisition of DCT datasets was achieved for 9 samples in total, which used up all 12 allocated shifts. Therefore, the allocated beam time was fully utilised despite the fact that it was not possible to complete the original experiment as planned. Analysis of the data has been a slow process but we have now achieved a successful reconstruction of a 300 μm^3 volume (Figure 1). In order to verify the grain shapes and orientations, verification is now underway using 3D EBSD. The use of a plasma focused ion beam at the university of Manchester allows for destructive 3D characterisation of large volumes of material (Figure 2). Once the data analysis method has been verified, the procedure can then be used to complete the analysis of the remaining samples.

Experiments are also underway to investigate the possibility of using methanol-iodine solution in order to induce a stress corrosion crack. The use of such a solution would allow the in-situ loading rig at ID11 to be used with the use of a custom-designed environmental cell, and many of the problems associated with the use of volatile iodine gas would be overcome. This also opens up the possibility of completing the experiment using a laboratory-based tomography set up. Although the data analysis process has been fairly slow, the procedure has now been optimised so that analysis of as 3D reconstructions using DCT have not previously been verified using 3D-EBSD. In addition, the results of the experiment will be used to provide 3D crystallographic information for subsequent in-situ tomography experiments, either using synchrotron or laboratory sources.

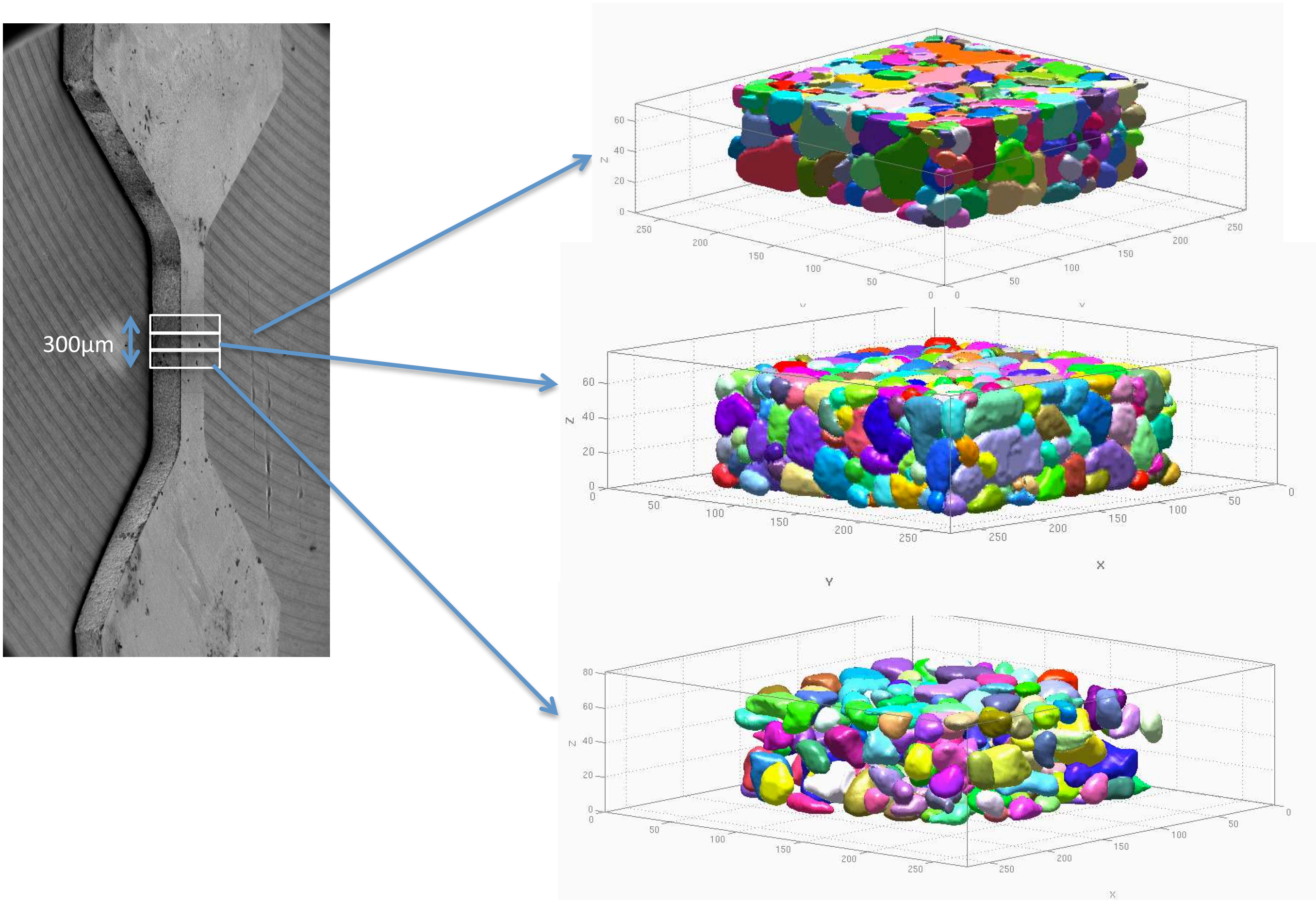

Figure 1: Successfully reconstructed DCT datasets showing equiaxed grain morphology in Zircaloy-4.

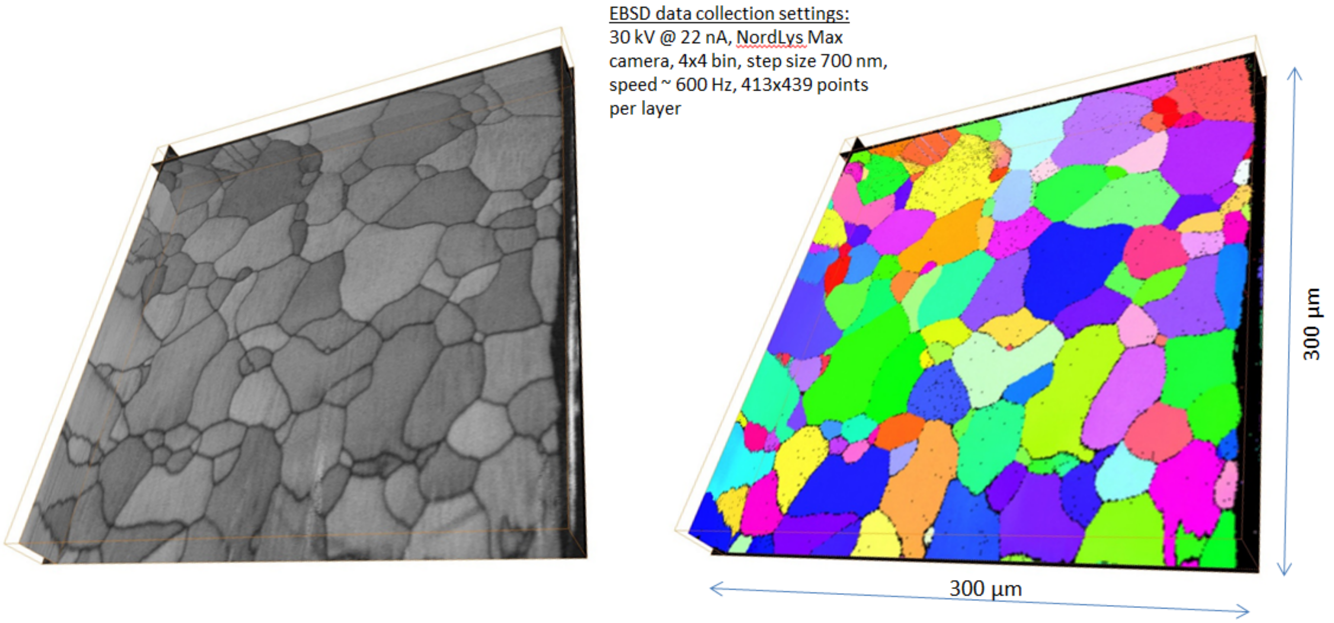

Figure 2: 3D microstructure of Zircaloy-4 using 3D-EBSD for verification of DCT dataset.