Developing a DIC capability for measuring thermal and irradiation creep at multiple length scales on an ion beamline

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Abstract. The current work focusses on the development of an in-situ ion irradiation-thermal-mechanical materials testing capabilities and novel scientific experimental methodologies for quantifying the deformation and damage at multiple length scales. The goal of these experiments is to examine irradiation-enhanced creep, a long-term degradation phenomenon of which further understanding is required of for timely qualification of high temperature structural materials for fusion. Here, we present the proposed methodologies for measuring the strain using both in-situ optical digital image corelation (DIC) and post-mortem scanning electron microscope-based high-resolution DIC. We discuss DIC set ups, calibrations and patterning methodologies employed to facilitate this full-field strain mapping at multiple length scales under fusion relevant conditions.

Introduction

There is a significant lack of knowledge on the impact that harsh fusion environments will have on the structural integrity of candidate materials. Previous research in the field of irradiation damage has focussed on materials performance under a single or dual aspect of these complex conditions: e.g., thermal-irradiation, thermal-mechanical or thermal-irradiation followed by thermal-mechanical testing. However, these effects are non-linear and examining them in isolation fails to predict the synergistic effect on material performance. Being able to test a material when subjected to these conditions synergistically provides us with a realistic assessment of the expected in-service performance, reducing over-conservatism in component design.

It is essential that detailed characterisation of the material response under irradiation-thermal-mechanical testing is carried out at multiple length scales to investigate conditions that may lead to critical failure of the component. Current techniques that have been used extensively to investigate irradiation damage are typically used post-irradiation. Here, we propose a method of using DIC to provide a live measure of the local strain during testing. Compared with conventional approaches for optical DIC, setting up such a system on an ion beam accelerator end station has the added complication of being unable to place the camera in the direct line of sight of the sample, requiring non-standard optics in a multiple mirror and port set up. Additionally, ion beam irradiation has a relatively shallow penetration depth. Sample thicknesses of less than 200 μm are therefore required for a through-thickness uniform irradiation damage profile.

A complementary technique has been developed for measuring the changes due to thermal or irradiation creep across multiple grains at sub-micron resolution. This technique is referred to as high resolution digital image correlation (HRDIC) and is predominantly used to investigate plastic deformation in metallic materials [1], but has been optimised to resolve the small changes a material is likely to undergo due to irradiation damage and/or creep. Typically, a fine nanoscale speckle pattern is applied to the surface with the sample then deformed at room temperature and imaged at regular intervals. Here, the suitability of a gold nanoscale speckle pattern for measuring the local deformation when a sample is subjected to irradiation and/or temperature is demonstrated for future use in measuring irradiation and thermal creep.

Optical DIC system on an ion accelerator beamline. The development of in-beamline mechanical tests has been undertaken as a collaboration between UKAEA, the University of Manchester, and testing equipment manufacturer NewTec Scientific, using the University of Manchester's Dalton Cumbrian Facility as the ion accelerator. The rig is a modified MT1000, funded by Henry Royce Institute, with an incorporated optical microscope and pyrometer system to provide accurate measurement of strain and temperature during the test. Figure 1 shows the location of the port housing the optical and pyrometer system relative to the mechanical stage and sample surface to enable the ion beam a direct line to sample. The optical image will be collected using a three-mirror system for which a detailed calibration procedure is being developed, due to the non-standard optics, prior to irradiation, to ensure confidence in the strain measurement.

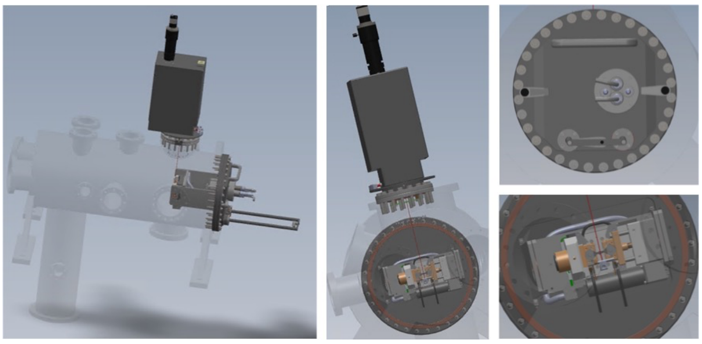

Figure 1: CAD drawing of the end station showing the relative positions of the MT1000 loading stage and the optical microscope and pyrometer systems.

High resolution strain mapping. Further adaption of the HRDIC approach for investigating the changes induced by irradiation damage/temperature is proposed. Firstly, a nanoscale gold speckle pattern is applied to the surface of the specimen using the gold remodelling technique. The pattern is then stabilised at the test temperature by further remodelling in an argon furnace. Then, a set of images of the region of interest are taken using a high-resolution scanning electron microscope. The local changes in strain at the microstructural level post irradiation/temperature are then determined using HRDIC by comparing the images taken before and after the experiment. The robustness of the pattern to these conditions was of particular concern, as there was the possibility of continued pattern development due to the temperature and sputtering on the sample surface due to irradiation.

The approach was validated by thermal-mechanical and thermal-irradiation testing, where pattern development was only observed when the test temperature exceeded the stabilisation temperature and the pattern was resilient to irradiation damage for doses up to 2 displacements per atom. From thermal-mechanical testing on a ferritic-martensitic steel, it was possible to observe a local change in the slip characteristics (Figure 2). Thermal-irradiation testing on a ceramic material showed significant expansion within individual grains and subsequent cracking at grain boundaries [2]. These studies open the possibility for future synergistic irradiation investigations where the local strains induced by loading during irradiation can be determined at the nanoscale.

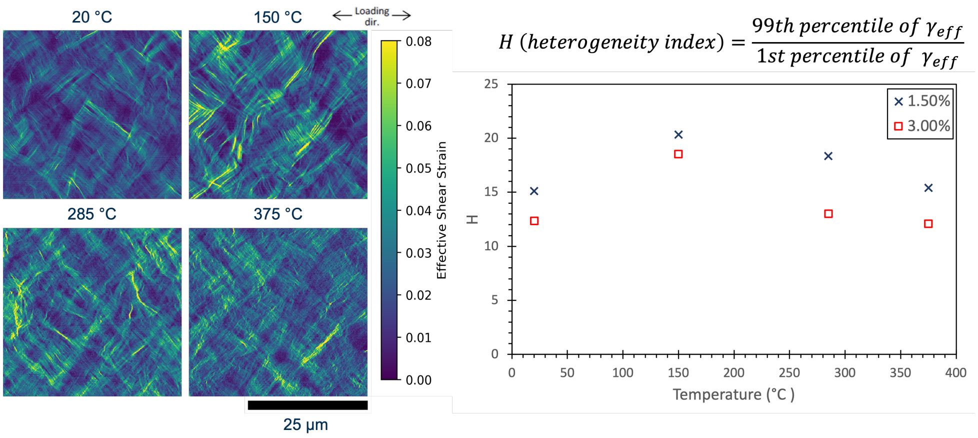

Figure 2: Maps of effective shear strain, at 1.5% applied strain, in a ferritic-martensitic steel showing the change in slip characteristic with temperature by comparing the max. to min. strain ratio

Conclusion. The current work has focussed on developing technologies to be able to produce a live strain measurement during thermal-mechanical testing on an ion irradiation beamline using optical DIC, along with complimentary HRDIC post irradiation strain measurements for understanding the deformation within single grains. The application of these techniques will allow for significant improvements to our knowledge of deformation behaviour of structural materials under conditions relevant to the fusion operating environment.

References

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