

# Enabling microscale full-field strain mapping in fusion relevant materials

B. Poole<sup>1a</sup>, D. Lunt<sup>1,2</sup>, A. Marsh<sup>1</sup>, C. Hardie<sup>1</sup>, C. Hamelin<sup>1</sup> and A. Harte<sup>1</sup>

<sup>1</sup>United Kingdom Atomic Energy Authority, Culham Science Centre, Abingdon, OX14 3DB, United Kingdom

<sup>2</sup>Department of Materials, University of Manchester, M13 9PL, United Kingdom

<sup>a</sup>ben.poole@ukaea.uk

**Abstract.** The untestable fusion environment necessitates the use of predictive modelling to qualify component material selection and design prior to manufacture and service. However, these predictive models require a thorough understanding of deformation mechanisms under relevant conditions. Deleterious deformation effects are often underpinned by microstructurally controlled phenomena and, as such, full-field strain mapping is necessary at the microstructural length scale. Here we present methodologies for examining these phenomena using high resolution digital image correlation, understanding the related uncertainties, and provide examples of the application of this technique in fusion relevant materials.

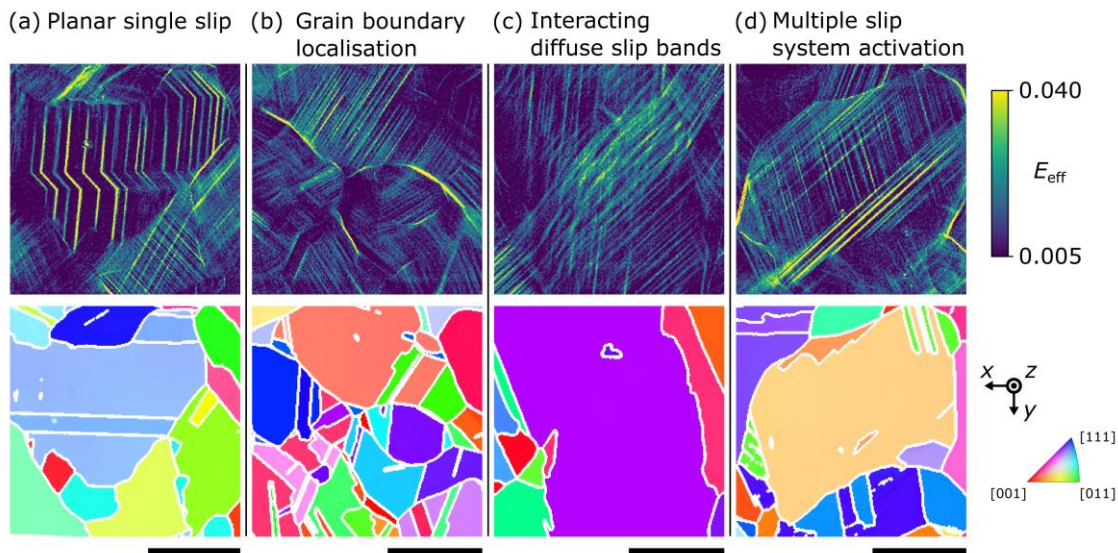
**Introduction.** Significant progress has been made internationally in the development of fusion as viable energy source. However, there remain outstanding engineering challenges in the delivery of a fusion power station, delivering net useful electricity output. One of these is the qualification of materials and components in the fusion environment. Components must withstand a unique combination of extremes in temperature (~1000°C for plasma facing components to cryogenic temperatures for magnet materials), high heat fluxes, strong magnetic fields, a high energy neutron irradiation spectrum and the presence of tritium [1]. This combination of conditions cannot be replicated prior to the completion of full-scale fusion devices and, as such, predictive modelling is required to qualify components.

Full-field strain mapping using SEM-based high resolution digital image correlation (HRDIC) provides a wealth of information and insight into microstructural deformation phenomena. When combined with orientation mapping using electron backscatter diffraction (EBSD), these quantitative displacement and strain data are ideal for the calibration and validation of explicitly representative crystal plasticity models [2]. These models can then be used to build representative volume elements which can inform component design. Applying this workflow to fusion relevant materials allows us to begin to probe materials performance under conditions relevant to operation.

**Patterning for sensitive materials.** State of the art HRDIC patterning techniques found in the literature generally apply heat [3] or aqueous environments [4] to remodel thin gold films, forming well dispersed, nanometre sized islands. These techniques are incompatible with many fusion materials where the application of heat can alter the microstructure, for example precipitation hardened Cu-base alloys [5], or change defect structures in materials which have been irradiated. Aqueous environments can corrode delicate specimen surfaces, producing surface layers which mask the underlying deformation of the substrate [6].

We introduce a technique which modifies that proposed by Montgomery et al. [7] to facilitate non-aqueous, chemical remodelling of silver films at room temperature to produce high quality speckle patterns, comparable to those achieved with gold remodelling. We have found that this pattern is suitably transparent to backscattered electrons that it does not impede EBSD measurements, allowing the use of EBSD without the need for pattern removal. We also successfully perform cross-correlation-based high-angular resolution EBSD (HREBSD) strain and geometrically necessary dislocation (GND) density measurements through the pattern.

**Full-field strain mapping experiments.** We demonstrate the usage of this speckle pattern on fusion relevant Cu-base alloys at the microstructural length scale. Full-field strain mapping, with a spatial resolution of ~140 nm, is linked to EBSD orientation measurements to relate deformation character to grain orientation and morphology. In oxygen-free high conductivity (OFHC) copper, a fusion heatsink material, we observe a diverse range of deformation behaviour (Figure 1), including widely dispersed planar slip, fine diffuse slip, slip system interaction, grain segmentation and grain boundary localisation. We attempt to relate these microstructural phenomena to macroscopic behaviour, for example linking grain boundary localisation to poor creep performance of OFHC-Cu.



**Figure 1: Detail of various slip behaviours observed in OFHC-Cu under 1.3% global applied tensile strain. Strain maps (top row) show the maximum in plane shear strain ( $E_{eff}$ ). The loading direction is horizontal. Orientation maps (bottom row) show crystallographic orientations parallel to the horizontal direction. The scale bar for each column is 20  $\mu$ m.**

**Exploring uncertainty in SEM-based HRDIC.** We explore the uncertainty in HRDIC displacement and strain measurement, an area thoroughly examined in the optical DIC community but often neglected for SEM-based DIC. If SEM-derived quantitative full-field displacement data are to be used to calibrate crystal plasticity models, an understanding of experimental measurement uncertainty is required. Instrument parameters which measurements are most sensitive to are identified using systematic studies as well as the impact of experimental procedure, for example reloading specimens for ex-situ loading, stage position automation during in-situ testing and electron beam focussing strategies. We provide some best practice recommendations for the use of SEMs for strain mapping.

**Conclusion.** We demonstrate the successful application of SEM-based HRDIC to fusion relevant materials, applying a patterning process which eliminates deleterious artifacts introduced by conventional patterning methodologies. The nature of this patterning method facilitates the use of HRDIC during post-irradiation examination and testing. Since the pattern is compatible with EBSD, we see a future of multimodal strain mapping experiments with suitable in-situ testing setups, combining elastic strain and GND density (HREBSD) mapping and total strain (DIC) mapping. Enabling these experiments, with a quantitative understanding of experimental uncertainty, on fusion relevant materials provides a wealth of data to produce calibrated and physically informed crystal plasticity models to allow predictive modelling under the extreme fusion operating environment.

## References

- [1] A. Quadling, W.E. Lee, J. Astbury, Materials challenges for successful roll-out of commercial fusion reactors, *J. Phys. Energy*. 4 (2022) 030401. <https://doi.org/10.1088/2515-7655/ac73b2>.
- [2] B. Poole, F.P.E. Dunne, Slip band interactions and GND latent hardening in a galling resistant stainless steel, *Mater. Sci. Eng. A*. 813 (2021) 141176. <https://doi.org/10.1016/j.msea.2021.141176>.
- [3] A. Orozco-Caballero, D. Lunt, J.D. Robson, J. Quinta da Fonseca, How magnesium accommodates local deformation incompatibility: A high-resolution digital image correlation study, *Acta Mater.* 133 (2017) 367–379. <https://doi.org/10.1016/j.actamat.2017.05.040>.
- [4] F. Di Gioacchino, J. Quinta da Fonseca, Plastic Strain Mapping with Sub-micron Resolution Using Digital Image Correlation, *Exp. Mech.* 53 (2013) 743–754. <https://doi.org/10.1007/s11340-012-9685-2>.
- [5] G.M. Kalinin, A.D. Ivanov, A.N. Obushev, B.S. Rodchenkov, M.E. Rodin, Y.S. Strebkov, Ageing effect on the properties of CuCrZr alloy used for the ITER HHF components, *J. Nucl. Mater.* 367–370 (2007) 920–924. <https://doi.org/10.1016/j.jnucmat.2007.03.256>.
- [6] D. Lunt, A. Orozco-Caballero, R. Thomas, P. Honniball, P. Frankel, M. Preuss, J. Quinta da Fonseca, Enabling high resolution strain mapping in zirconium alloys, *Mater. Charact.* 139 (2018) 355–363. <https://doi.org/10.1016/j.matchar.2018.03.014>.
- [7] C.B. Montgomery, B. Koohbor, N.R. Sottos, A Robust Patterning Technique for Electron Microscopy-Based Digital Image Correlation at Sub-Micron Resolutions, *Exp. Mech.* 59 (2019) 1063–1073. <https://doi.org/10.1007/s11340-019-00487-2>.