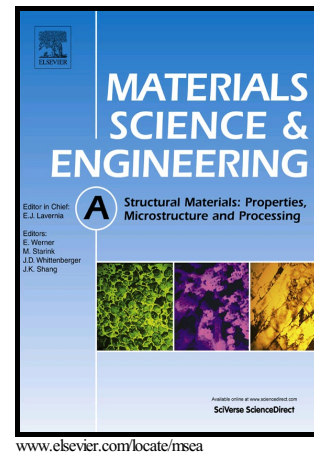


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Experimental study and multiscale modelling of the high temperature deformation of tempered martensite under multiaxial loading

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ABSTRACT

The microstructural deformation of ex-service 9Cr-1Mo steel, with a tempered martensitic microstructure, has been examined in this study, through the combined use of electron backscatter diffraction (EBSD) and multiscale modelling techniques. Both the experimental and predicted deformation of the material at a notch root on a range of scales from the specimen level down to the microstructural block level are compared. A tension loaded notch specimen of the material which was extracted from an ex-service power plant pipe was used for this analysis. The deformation at the specimen level was quantified by analysis of the load displacement curves and notch opening displacement, which showed excellent agreement with the predicted results from the experimentally calibrated elastic-plastic finite-element model of the specimen geometry. The microstructural deformation was experimentally measured through the use of EBSD carried out at the notch root before and after high temperature mechanical testing. The initial orientation of the microstructure as well as the displacement around the boundary of the area of interest in the macroscale model were applied to a representative volume element (RVE) and a slip based crystal plasticity modelling framework was implemented to model the in-elastic deformation of the material under high temperature loading.

KEYWORDS: *Ex-service material, 9Cr-1Mo steel (P91), Elevated temperature, EBSD, multiscale modelling, Plastic strain, Mean orientation difference (MOD)*

1 INTRODUCTION

1.1 Background

In recent years there has been an increased emphasis on the thermal efficiency of conventional (fossil fuel) power plant operating under flexible loading conditions (Bugge et al., 2006). A key route to improving efficiency is by increasing the steam temperature and pressure entering the turbine. The development of materials capable of withstanding these conditions under flexible (cyclic) loading is therefore needed, coupled with an understanding of how the material microstructure changes when subjected to conditions

representative of the operating temperatures and pressures of conventional power plants. In the current study a novel experimental methodology has been developed to allow for the examination of the high temperature deformation, at multiple length scales following testing in a high temperature atmospheric environment representative of plant operating conditions. This method involves a combination of mechanical testing, electron backscatter diffraction (EBSD), and finite element (FE) analysis. The developed methodology has been implemented to examine the microstructural deformation of a modified 9Cr-1Mo steel (P91) at a notch during testing at 500 °C. A similar approach taken in previous work in this area has examined the deformation behaviour of this material at room temperature (Golden et al., 2016, 2014), but as these materials operate in a high temperature environment, it is critical to understand how these materials behave under high temperature loading, which has been examined in the current work.

1.2 Material microstructure

P91 is a martensitic steel, which contains 9 %wt Cr and 1 %wt Mo with the balance being primarily Fe (Czyrska-Filemonowicz et al., 2006; Fournier et al., 2009; Thomas Paul et al., 2008). The microstructure is a hierarchical one comprising of prior austenite grains (PAG) (of width approx. 20–25 μm) which, following rapid cooling during heat treatment, transform to a martensitic structure, containing packets, which are further subdivided into blocks (approx. 7–10 μm in size). The lowest length scale in the microstructure is the martensitic laths (approx. 2 μm in width) which form within the block (Dudko et al., 2013; Li and Mitchell, 2013; Thomas Paul et al., 2008), but these features will not be discussed further as they are not currently accounted for in the microstructural model. An etched micrograph of a P91 sample with a PAG boundary outlined in red is illustrated in Figure 1(a). Packets and blocks within the PAG can be identified through a misorientation analysis of neighbouring pixels in an EBSD map, as shown in Figure 1(b). The black lines in this figure represent block or packet boundaries. A schematic of the hierarchical microstructure is shown in Figure 1(c) to illustrate the different microstructural features present in the material.

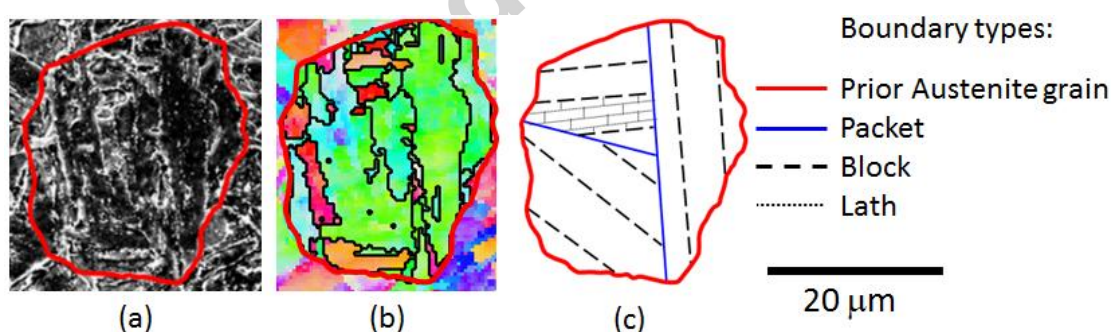


Fig 1: (a) Etched SEM image of the microstructure of P91, with a PAG boundary outlined in red, (b) EBSD map of the PAG with block boundaries highlighted in black, (c) Schematic of the hierarchical microstructure of P91

In this work electron back scattered diffraction (EBSD) is used to obtain crystallographic information over the relevant length scales of the material. The various boundaries present in the microstructure can be identified through misorientation analysis of the EBSD maps where block boundary misorientation is $> 15^\circ$ and lath boundaries have a misorientation of $< 2^\circ$ (Dudko et al., 2013; Panait et al., 2010). In order to distinguish between PAG, packet and block boundaries directly from the EBSD scan a martensitic variant analysis of the orientations obtained from EBSD is required, as these boundaries all have similar

misorientations. This method has been previously used to identify these boundaries in P91 in Sun et al., 2018. Another key microstructural feature of these materials is the presence of precipitates, the most common of which are $M_{23}C_6$ ($M = Cr, Fe, Mo$) which typically form on the lath/block boundaries and contribute to the strength and toughness of the material by retarding sub-grain growth during the tempering process. The typical diameter of $M_{23}C_6$ precipitates found in P91 is 110 nm. Fine MX type ($M = V, Nb$, and $X = C, N$) precipitates are typically found within laths and have an approximate diameter of 30 nm. These precipitates play a fundamental role in the microstructure by inhibiting the movement of dislocation leading to the materials superior creep resistant properties, which make this material suitable for high temperature power plant applications (Czyrska-Filemonowicz et al., 2006; Dudko et al., 2013; Panait et al., 2010)

1.3 Aims and objectives

There have been significant advances made in experimental microstructural deformation measurement, such as the Wilkinson method (Wilkinson, 1996) which determines strain and orientation changes from small shifts in the Kikuchi patterns in an EBSD measurement using cross correlation functions, allowing for strain measurements as low as $\pm 10^{-4}$ and rotations of $\pm 0.006^\circ$. Furthermore, advances in the field of computational modelling of microstructural deformations through crystal plasticity based models for polycrystalline materials (Dunne et al., 2012, 2007; Erinoshio et al., 2013; Zhang et al., 2014) have significantly improved the capability for accurately predicting deformation at microstructurally relevant length scales. However, there have been few attempts to compare microstructural model predictions of deformation to direct experimental measurements of initial and deformed microstructures. The aim of this research is to investigate experimentally inelastic deformation occurring under complex loading at a notch in an ex-service P91 material, subjected to high temperature monotonic loading. These results will be used to validate the microstructural model used to represent the response of this material, at the micro-scale. Wilkinson has proposed a method to determine plastic strain (Wilkinson et al., 2006; Wilkinson and Dingley, 1991) through the measurement of Kikuchi pattern contrast reduction brought about by lattice distortion close to dislocations within the diffraction volume of the electron beam. To carry out this analysis a calibration curve must first be established by comparing Kikuchi patterns from samples with a known induced strain, which can then be used to determine the level of plastic strain present in the sample. However Kamaya et al., 2005, found that it is difficult to distinguish between pattern quality reduction caused by plastic strain and that brought about by the electron beam parameters such as beam focus, current and energy, surface quality and sometimes even crystal orientation when comparing successive measurements. Due to these difficulties, coupled with the fact that in order to compare Kikuchi patterns before and after deformation in this study, both patterns would have to be obtained from a region of the similar orientation (Britton and Hickey, 2018) in this case from within the same martensitic lath which would be extremely difficult to achieve in practice, this approach has not been pursued in this study. Instead lattice orientation measurements, obtained from EBSD over a relatively large region of the microstructure, (approx. $120 \mu m \times 200 \mu m$) containing a number of PAGs, packets and blocks are compared directly before and after deformation and the change in mean orientation difference (MOD) of the different regions is quantified. This value of MOD is defined as the average difference between the orientation of each pixel in an area of interest and the average orientation of that area. This value is similar to

the crystal deformation (C_d) described in Kamaya et al., 2005. The measured deformation is then compared with the prediction of plastic strain and block level deformations using a multiscale modelling approach. A similar approach has been used by Golden et al., 2016, 2014 to model the microstructural deformation in P91 at room temperature. However, to our knowledge, no similar experiments have been reported to date of EBSD measurements on tested notched specimens at high temperatures.

2 EXPERIMENTAL PROCEDURE

2.1 Specimen geometry

The specimen geometry used in this study was a notched specimen extracted from a pipe of ex-service P91 material, which had seen a service period of approximately 63,000 hr at a temperature range of 450–550 °C. The specimen geometry can be seen in Figure 2(a); the specimen thickness is 12.5 mm and the notch width is 0.7 mm. The compact size of the specimen allows it to fit inside a Hitachi SU-70 SEM for imaging and EBSD analysis and the presence of the notch allows for large local strains to be generated simulating a stress concentration which may be present in a power plant component.

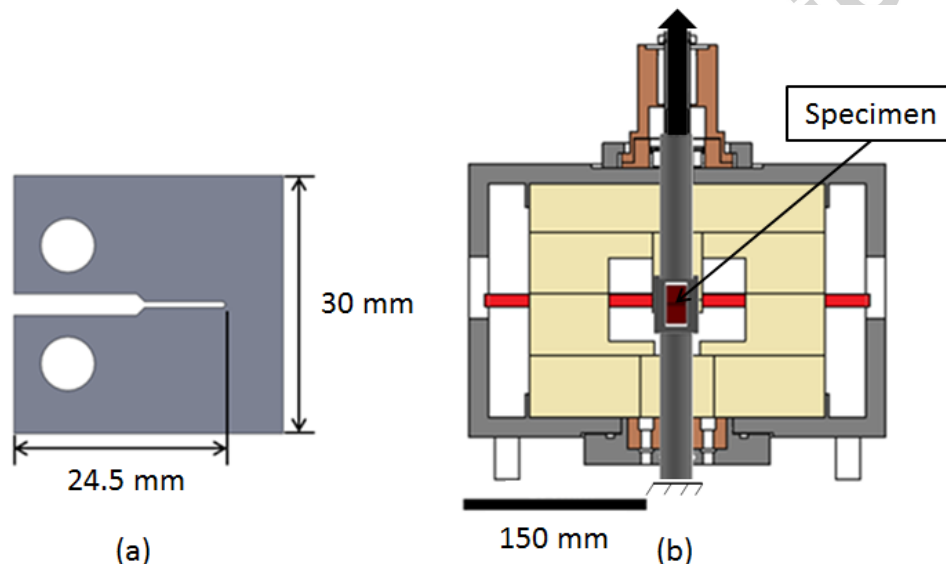


Fig 2: Experimental set-up for high temperature mechanical testing (a) specimen geometry (notch width 0.7 mm), (b) schematic of the mechanical test rig used to carry out high temperature testing by the application of a temperature controlled furnace

2.2 Specimen preparation

Prior to testing the specimen surface is polished to a high quality mirror finish which is a requirement for EBSD analysis. This process involved grinding the surface with papers of reducing grit size (P240–P1200) to remove damage from the sample surface. The surface is then polished using diamond solution with varying particle sizes (1–0.3 μm), before final polishing in a colloidal silica solution. Following this preparation procedure an EBSD scan of 126 $\mu\text{m} \times 196 \mu\text{m}$ was taken at a position 75 μm from the notch root. The scan is not positioned directly adjacent to the notch in order to avoid excessive damage within the measured region during the high temperature test. The EBSD scans in this study were taken in the Hitachi SU-70 SEM system using the HKL Channel 5 software with a step size of 0.7 μm and a tilt angle of 70°. After the initial EBSD scan had been obtained, the specimen was

tested in a specially adapted Tinius Olsen tensile test rig, shown in Figure 2(b). This test rig is capable of testing materials at temperatures up to 1000 °C; for this study a temperature of 500 °C was chosen as it lies within the material's operating temperature range of 450–550 °C. Prior to the test the specimen was left to thermally soak at the test temperature for 2 hours to ensure uniform temperature throughout the sample, as determined by thermocouples attached to the specimen.

Following testing in an atmospheric environment a thin (approx. 0.5 μm) oxide layer is formed on the surface. This layer must be removed prior to obtaining the post-test EBSD scan as the electron beam is not capable of penetrating the oxide to obtain the crystallographic information from the underlying material. Therefore, a secondary polishing step is required after the mechanical testing stage. This step is carried out in a low energy ion beam source within a vacuum chamber. This process slowly removes the oxide layer from the sample but can be stopped before any base material which was scanned in the initial EBSD scan is removed. Due to the out-of-plane deformation of the specimen during mechanical testing a local depression (necked region) is produced on the surface at the notch root, resulting in an angular offset of the area of interest to the horizontal plane. To account for this offset the post-test surface topography was measured using a laser profilometer which produced a surface profile of the specimen near the notch. This profile allowed for the measurement of the angle between the specimen surface and the horizontal plane within the region of interest for the EBSD scan. This angle can then be corrected for when carrying out the post-test EBSD scan by reducing the specimen tilt angle so that the surface to be scanned is at 70° to the horizontal plane, as required for an EBSD measurement. The EBSD scan before and after testing is used to analyse the block orientation change during high temperature deformation of the notched specimen.

3 MULTISCALE CONSTITUTIVE MODELING FRAMEWORK

Following the approach of Golden et al., 2016, a multiscale modelling strategy is adopted. A macroscale model provides a prediction of the global force-displacement response of the material and determines the displacements to be applied to the boundary of the microscale model which will be used to predict the microscale deformation of the material.

3.1 Macroscale modelling

To predict the global response of the notched specimen during the high temperature test a standard elastic-plastic finite-element (FE) model of the specimen geometry was been created in Abaqus (Dassault Systemes Simulia Corp, 2014). This is an isotropic Mises plasticity model with linear piecewise strain hardening. The elastic plastic behaviour of the material was obtained from tensile specimens extracted from the same pipe as the notched specimens. The Young's modulus and yield strength obtained from these tests were 177 GPa and 300 MPa respectively, the experimental error was ± 3.5 MPa, based on three measurements. The response of a single element model in uniform tension was compared to the experimental tensile response of the material. This comparison showed an exact fit, allowing for the elastic-plastic behaviour of a notch specimen to be modelled.

A 3-D model of the test specimen geometry, shown in Figure 3, was created in Abaqus. The specimen is loaded by applying a displacement to a rigid loading pin, connecting the nodes in the pin to a reference point through an equation constraint, as shown in Figure 3(a). The displacement is obtained from the model at the location where the extensometer was positioned in the experiment. The extraction of these values allowed for direct comparison

of the predicted force-displacement response with that of the experiment. Symmetry boundary conditions are applied as shown in Figure 3(b). Following a mesh convergence study, a mesh of 4661 elements of type C3D20H was used to represent the geometry. A rectangular region of high density elements was created close to the notch root, representing the region considered in the microscale model, which is discussed in Section 3.2.

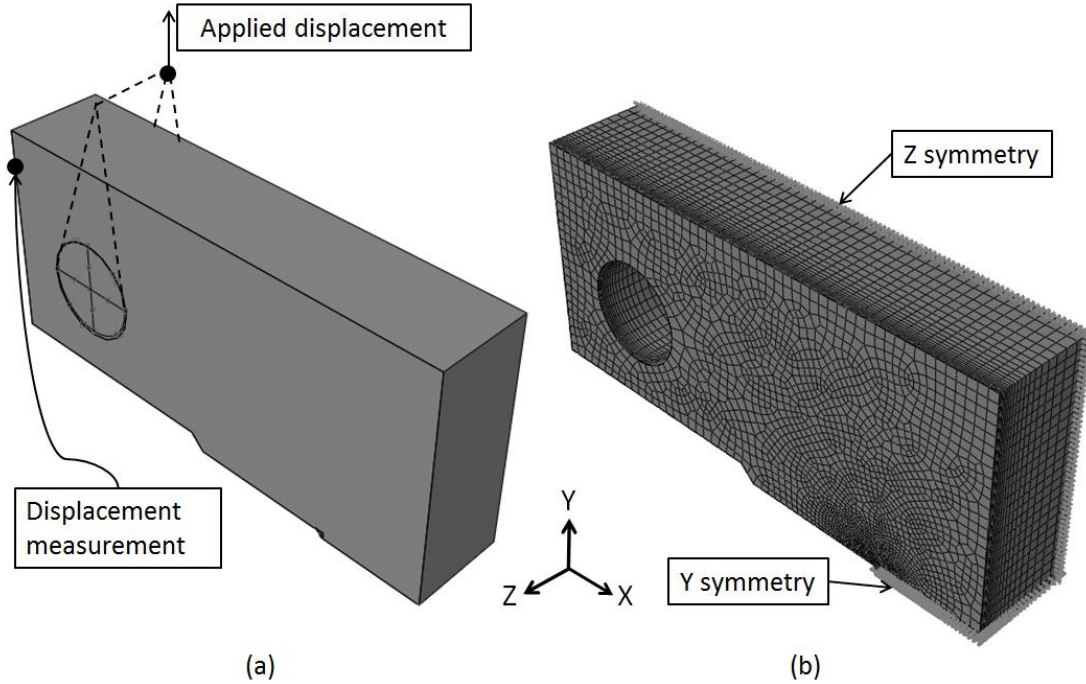


Fig 3: 3D FE model set up: (a) loading condition applied to the loading pin through a single reference point and the location of the displacement measurement node to be compared with extensometer data (b) 3D mesh applied to the model with symmetry planes indicated.

3.2 Multiscale model setup

The macroscale model was constructed so that information could be easily extracted and applied directly to the microscale model. A rectangular region with uniform element size was constructed close to the notch root in the macroscale model, which is in the same position ($75 \mu\text{m}$ from the notch root) and has the same size ($98 \mu\text{m} \times 126 \mu\text{m}$) as the region scanned by EBSD in the experimental analysis (see Figure 4). The element size in the microscale model was chosen to match the step size in the EBSD scan ($0.7 \mu\text{m}$). Therefore, each element in the microscale model represents one pixel in the EBSD scan, allowing for direct comparison of the measurement with the finite element model. To simplify the interpolation of the displacement field from the micro- and macro-scale models, the element size in the macroscale model is divisible by an integer value of the element size of the microscale model. In this case the size of the macroscale element is $14 \mu\text{m} \times 14 \mu\text{m}$ corresponding to the microscale element size of $0.7 \mu\text{m} \times 0.7 \mu\text{m}$, i.e. there are 400 (20×20) microscale elements within each macroscale element in the region of interest.

The size of the macroscale elements close to the notch are on the order of the microstructural block size. Therefore, a macroscale element may contain two or more blocks with differing orientations. A crystal plasticity modelling framework could be introduced for these elements in order to obtain a more accurate representation of the displacement field.

However, the mesh remote from the notch is much coarser (see Figure 4a) and each finite-element contains multiple material blocks. Therefore incorporating a crystal plasticity model in the vicinity of the notch would result in a model which transitions from an anisotropic material near the notch to an isotropic material remote from the notch, which would be undesirable. Therefore in the macroscale model the material is assumed to be a polycrystal with a random orientation and the material is treated as isotropic.

The displacements from the boundary of the rectangular region in Figure 4(b) were applied as boundary conditions to the microscale model. The macroscale model was deformed such that the inelastic strain close to the notch root was approximately 2%.

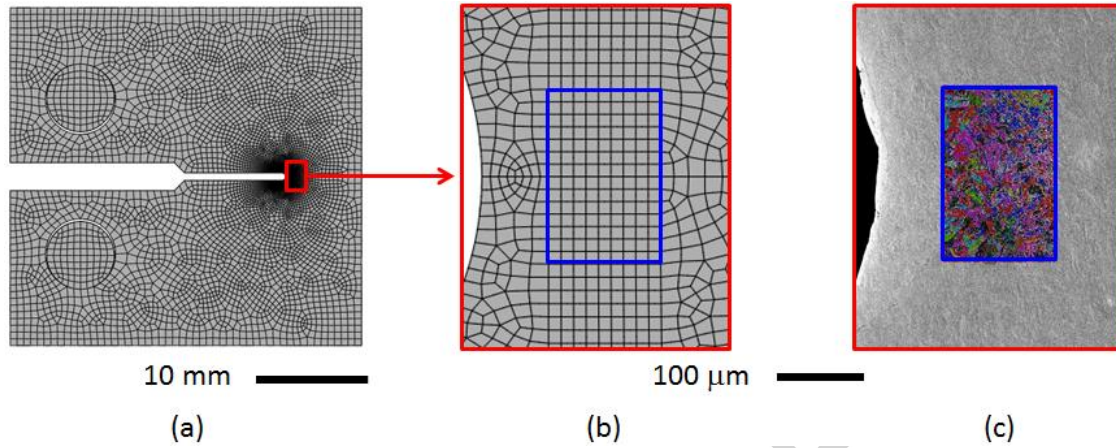


Fig 4: Macroscale mesh design: (a) full mesh (2D view mirrored to show full specimen geometry) (b) Magnified view of highlighted region in (a) showing rectangular region of square elements, (c) SEM image with superimposed EBSD scan showing the size and position of the EBSD scan.

The nodal displacements on the boundary of the region of interest in the macroscale model were extracted, as illustrated in Figure 5, and applied to each boundary node in the microscale model by linear interpolation, as shown in Figure 5(c). The Euler angles of each pixel measured in the initial EBSD scan of the microstructure are applied to the corresponding microscale elements.

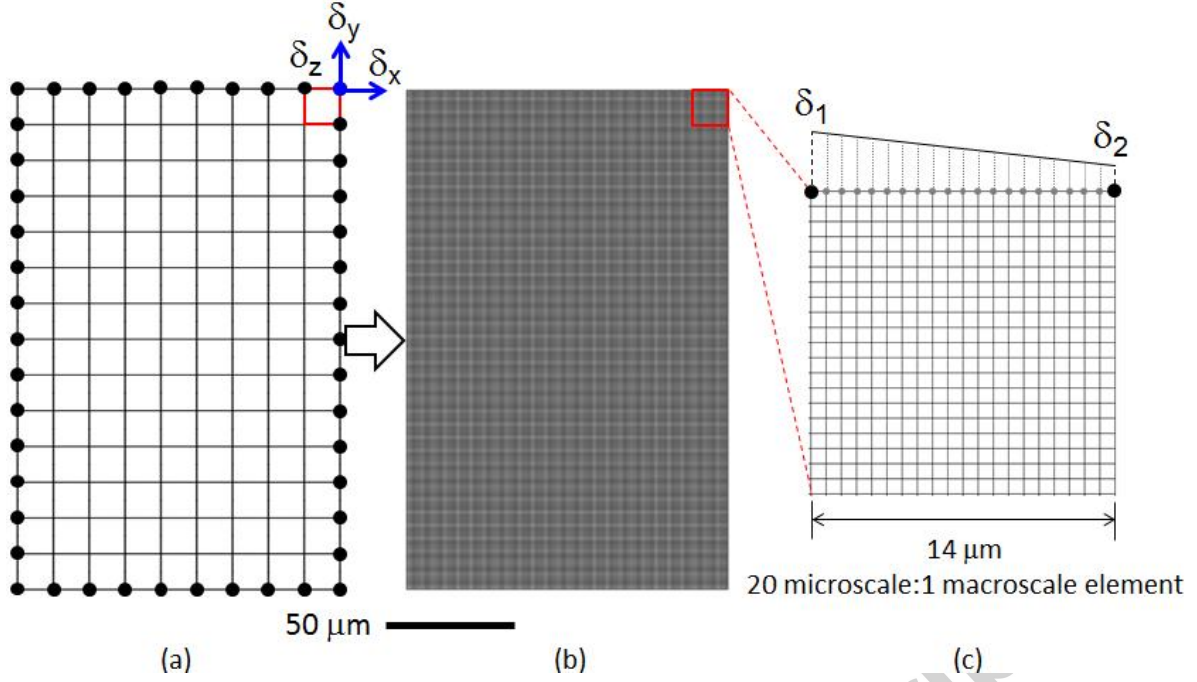


Fig 5: Application of boundary conditions to the microscale model, (a) macroscale mesh in region of interest, (b) microscale mesh, (c) a segment of the microscale mesh representing 1 macroscale element, showing the application of the nodal displacements.

3.3 Microscale modelling

A crystal plasticity model is used to represent the constitutive behaviour at the microscale, using the slip system based modelling framework of Asaro and Rice, 1977, implemented via an Abaqus UMAT (Cheong and Busso, 2006; Golden et al., 2016, 2015, 2014, Li et al., 2014a, 2014b, 2011; Li and O'Dowd, 2011). The crystal plasticity model described below is based on a slip system (Schmidt rule) model, to predict the plastic deformation of crystalline materials. Equation (1) describes the plastic velocity gradient, $\mathbf{L}^p$, which is linearly dependent on crystallographic slip rate,

$$\mathbf{L}^p = \dot{\mathbf{F}}^p (\mathbf{F}^p)^{-1} = \sum_{\alpha=1}^N \dot{\gamma}^{\alpha} \mathbf{m}^{\alpha} \otimes \mathbf{n}^{\alpha}, \quad (1)$$

where $\mathbf{F}^p$ denotes the plastic deformation gradient; $\dot{\gamma}^{\alpha}$, $\mathbf{m}^{\alpha}$ and $\mathbf{n}^{\alpha}$ describe the slip rate, slip direction and slip plane normal to the slip system, α , respectively. The total number of potentially active slip systems is denoted by N , the symbol $\otimes$ indicates a dyadic product. The equivalent plastic strain, ε_{eq} associated with this plastic velocity gradient is defined in Equation (2):

$$\varepsilon_{ep} = \int_0^t \left(\frac{2}{3} \mathbf{D}^p : \mathbf{D}^p \right)^{\frac{1}{2}} d\tau. \quad (2)$$

In Equation 2, t is the current analysis time and $\mathbf{D}^p$ is the plastic rate of deformation tensor defined in Equation 3.

$$\mathbf{D}^p = \frac{1}{2} \sum_{\alpha=1}^N \dot{\gamma}^{\alpha} \left[\mathbf{F}^e \mathbf{m}^{\alpha} \otimes \mathbf{n}^{\alpha} (\mathbf{F}^e)^{-1} + (\mathbf{F}^e)^{-T} \mathbf{n}^{\alpha} \otimes \mathbf{m}^{\alpha} (\mathbf{F}^e)^T \right], \quad (3)$$

where $\mathbf{F}^e$ denotes the elastic deformation gradient. A linear elastic relationship is defined in Equation 4 between second Piola-Kirchhoff stress, $\mathbf{S}$, and elastic Green tensor, $\mathbf{E}^e$.

$$\mathbf{S} = \mathbf{C} : \mathbf{E}^e \quad (4)$$

where $\mathbf{C}$ is the anisotropic elastic stiffness tensor. The P91 crystallographic model is based on a BCC crystal structure which has a total of 48 potentially active slip systems. Slip on these systems is controlled by a thermally activated flow rule and slip resistance (Li et al., 2013) shown by Equations (5) and (6), respectively.

$$\dot{\gamma}^{\alpha} = \dot{\gamma}_0 \exp \left(-\frac{F_0}{kT} \left(1 - \left(\frac{|\tau^{\alpha}| - S^{\alpha}}{\tau_0} \right)^p \right)^q \right), \quad (5)$$

where F_0 is the total energy to overcome lattice resistance, T is the absolute temperature, k is the Boltzmann constant, τ_0 is the critical shear stress, and p , q and $\dot{\gamma}_0$ are the exponential and pre-exponential constants. The slip resistance evolution on slip system α ($\dot{S}^{\alpha}$) is determined by Equation (6),

$$\dot{S}^{\alpha} = \sum_{\beta=1}^N h^s \left(\frac{S_{sat} - S^{\beta}}{S_{sat} - S_0} \right) |\dot{\gamma}^{\beta}| \quad (6)$$

Where h^s is the hardening constant and S_0 and S_{sat} are the initial and saturated values of slip resistance, respectively. The use of a single hardening constant rather than a hardening matrix $h^{\alpha\beta}$ implies Taylor hardening, with all active slip system interacting equally during strain hardening (Li et al., 2013).

This material model has been implemented for P91 through a user material subroutine (UMAT) within Abaqus. Although there are 48 potentially active slip systems in this material of the type; $\langle 111 \rangle \{110\}$, $\langle 111 \rangle \{211\}$ and $\langle 111 \rangle \{123\}$, for computational efficiency only 12 slip systems are considered in this model of the type $\langle 111 \rangle \{110\}$. These slip systems are the primary systems in BCC structures according to Du et al., 2018 and Rogne & Thaulow, 2015, where it has been shown that under unconstrained loading (micro-tensile and micropillar compression tests, respectively) primary slip in BCC structures occurs mainly on these slip systems. Du et al., 2018 show that under constrained loading primary or secondary slip can occur on the $\langle 111 \rangle \{211\}$ systems, with no evidence found of slip occurring on the $\langle 111 \rangle \{123\}$ system. Furthermore, Batista et al., 2015 showed that the majority of slip in P91 occurs on the $\{110\}$ family of planes. Therefore, we believe that the incorporation of the $\langle 111 \rangle \{211\}$ systems, on which secondary slip may occur (Du et al., 2018), would not significantly improve the accuracy of the model while increasing the computational time.

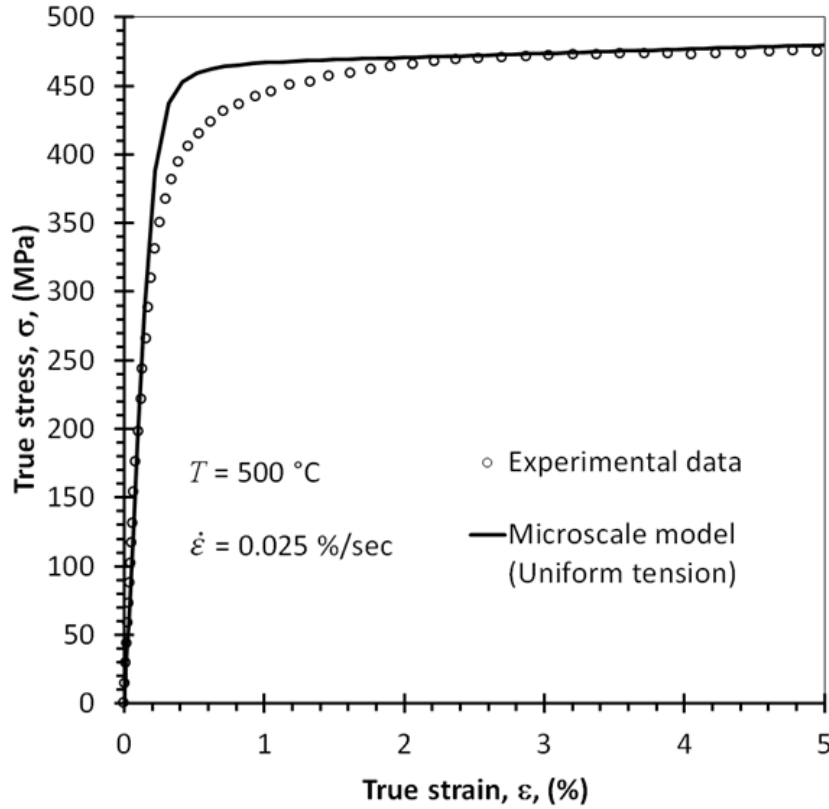


Fig 6: Microscale model calibration when loaded in uniform tension, compared to high temperature tensile data obtained from the material.

To model the microstructural deformation of the material the framework described in this section was applied to a microscale representative volume element (RVE), as described in Section 3.2. The Abaqus UMAT was then used to predict the microscale deformation at the notch root in conjunction with the RVE.

The material parameters in Equations (5) and (6) were obtained by calibrating the microscale model response, using an RVE under uniaxial tension, against the global experimental tensile response of the material at the relevant temperature. The results of this calibration are presented in Figure 6. There is reasonably good agreement between the experimental tensile data obtained from the high temperature mechanical test of the material and the data obtained from the monotonically loaded microscale model. Although there is some discrepancy close to the yield point, the stress/strain response of the model and experiment match at the strain levels (2%–3%) of interest in this study. The parameters used to obtain the fit shown in Figure 6 can be seen in Table 1.

Table 1: Microscale model parameters

T °C	τ_0 MPa	S_0 MPa	S_{sat} MPa	h_0 MPa	$\dot{\gamma}_0$ s ⁻¹
500	532	38	250	70	450
F_0 ($\times 10^{-18}$) J	p	q	E_{100} GPa	G_{100} GPa	G_0 GPa
0.43	0.5	1.25	97	121	140

4 RESULTS AND DISCUSSION

This section presents the results obtained from the experimental analysis and compares these results to those obtained from the computational simulations at the different length scales.

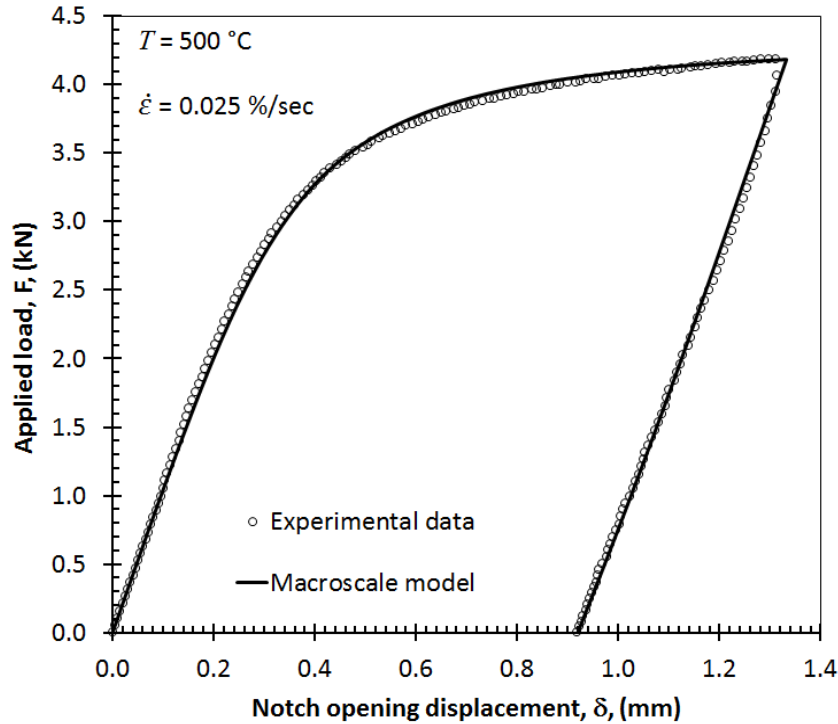


Fig 7: Global force displacement response from notched specimen compared with macroscale FE model (error bars are omitted as the error between the two experimental tests is negligible).

4.1 Macroscale results

The global response of the material during the high temperature test is compared with the experimentally calibrated macroscale model prediction in Figure 7. As can be seen there is excellent agreement between the experiment and FE simulation at this scale. This test has been repeated with the data showing minimal variability between tests.

As an additional validation of the model prediction at this length scale, the notch root opening at incremental distances (0.5 mm) from the notch root was measured using a shadowgraph before and after testing. The results of this analysis are presented in Figure 8.

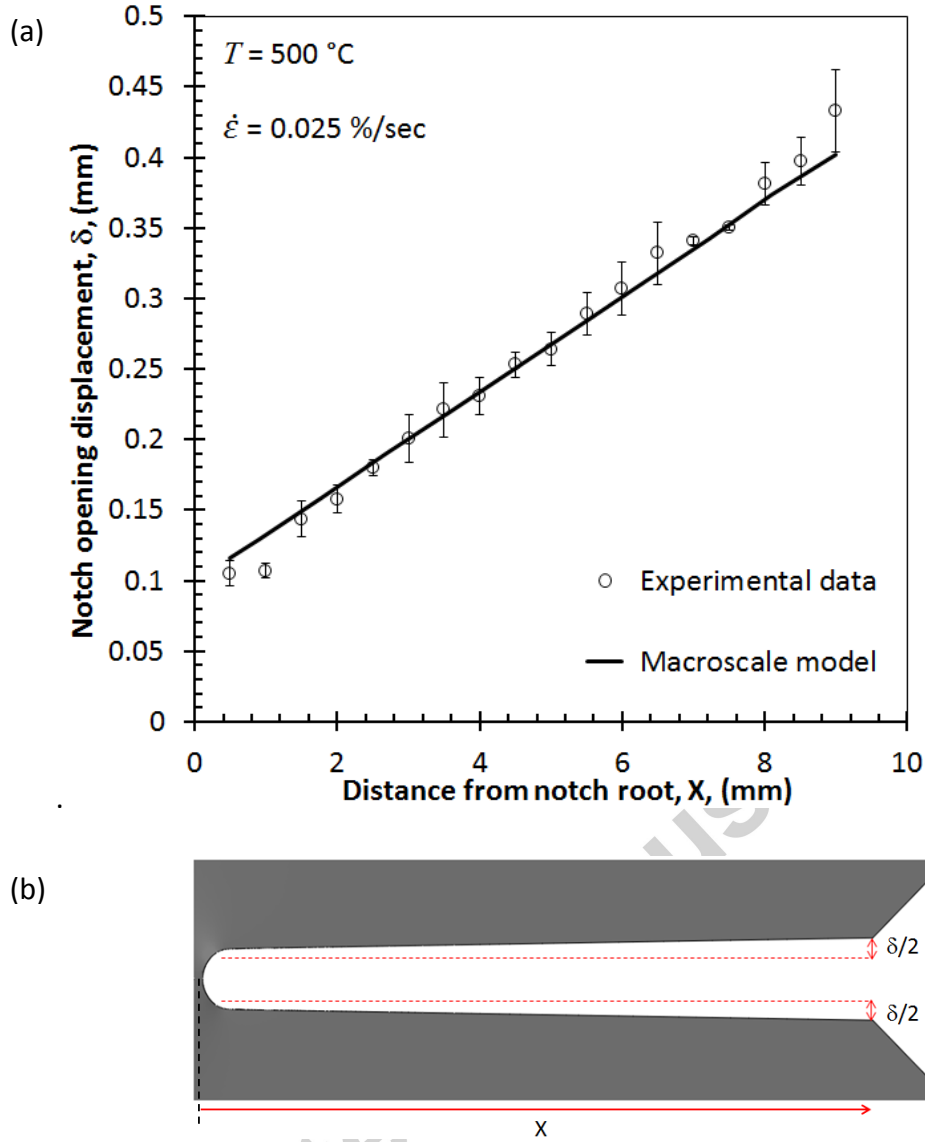


Fig 8: (a) Measured and predicted notch opening displacements at incremental distances from the notch root. The experimental scatter is based on two measurements. (b) Schematic showing the location of the notch opening measurements.

4.2 Microscale model results

This section presents the microscale results obtained from the experimental EBSD analysis of the area of interest and from the crystal plasticity FE model. The initial microstructure is determined from EBSD and the deformation is controlled by the displacements obtained from the macroscale FE model.

In Figure 9 the average centroidal stress and strain in the loading direction within the area of interest for both the macro- and micro-scale models are compared. An initial reduction in stress is noted in the microscale model after yielding, compared to that predicted from the macroscale model. However, overall it is seen that there is good agreement between the stress/strain responses for the macro- and micro-models in the region of interest.

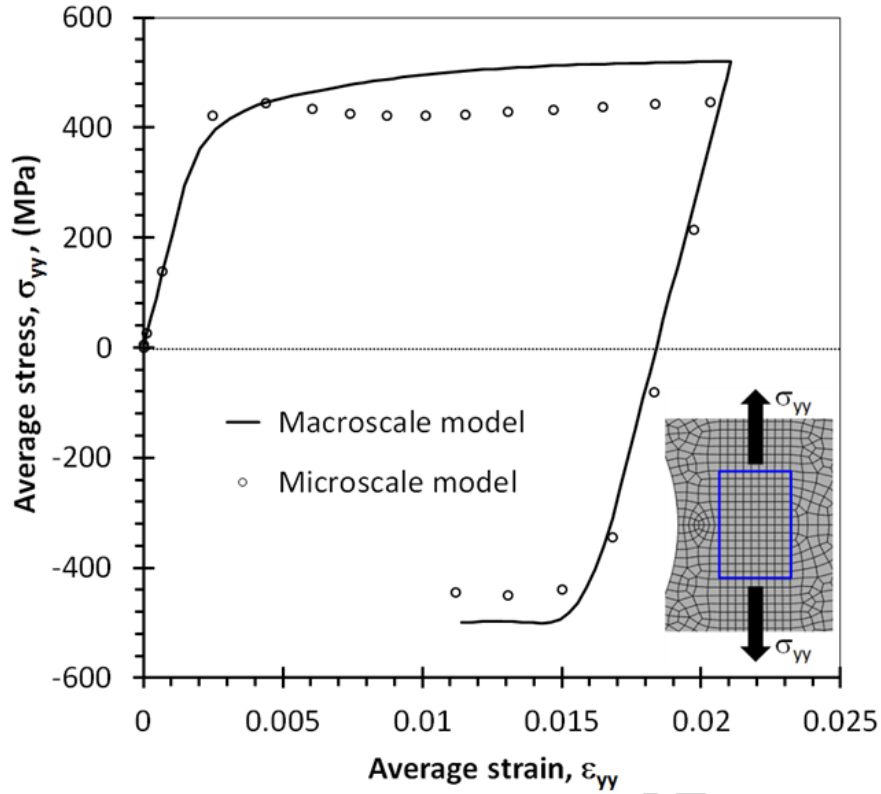


Fig 9: Comparison of average stress/strain response in the loading direction obtained from the macro-and microscale models within the area of interest.

Experimental validation of the microscale model was examined by comparing the EBSD scans before and after deformation, which may be considered a type of digital image correlation (Hild and Roux, 2006; McCormick and Lord, 2010). The EBSD scans obtained from the experiment before and after loading are divided into 10 equally spaced sections as shown in Figure 10. In each of these sections the distance between two points in the scan is measured. Each point is chosen at the intersection of three colours in the EBSD scan which corresponds to a point where three microstructural blocks meet. The initial and deformed positions for each point can be obtained directly from the EBSD image and the average strain across the region of interest can be established. An identical measure can be extracted from the results of the finite-element analysis at the micro-scale. At the macro-scale, comparison can be made with the FE results based on the total strain ahead of the notch root.

The results of this analysis are presented in Figure 11, which shows the strain measurement results across the area of interest at incremental distances from the notch root. Both the macro- and microscale models provide similar predictions and closely match the trend of the experimental measurement. Both models under predict the strain measured from the EBSD scan by approximately 0.5% strain. The error bars on the experimental results are based on three separate measurements of the same scans at different positions.

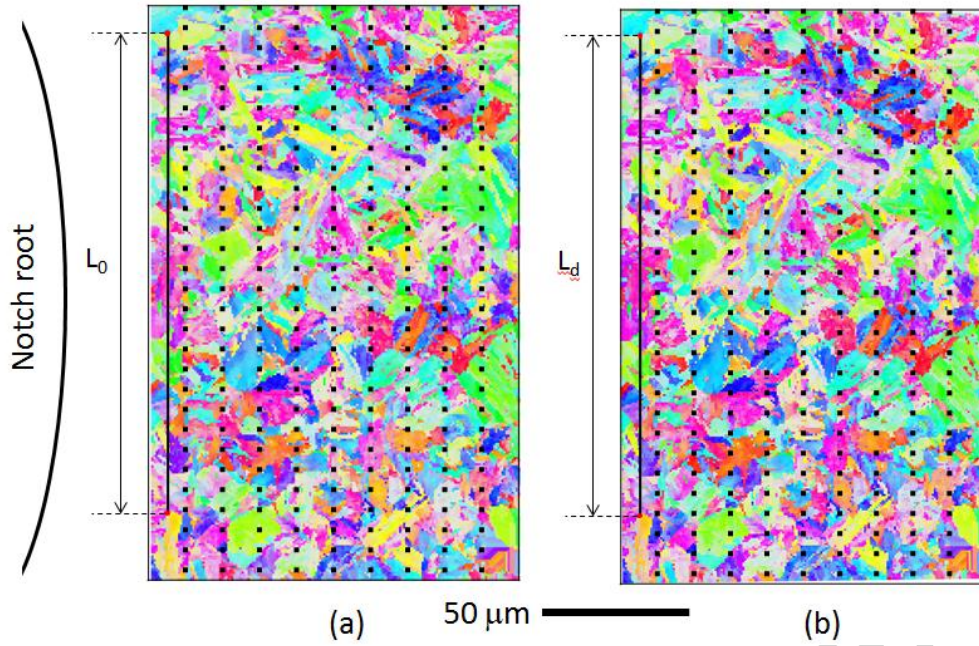


Fig 10: Schematic illustrating method of strain measurement to determine the strain within the region of interest by comparing the (a) initial and (b) deformed microstructures obtained from EBSD.

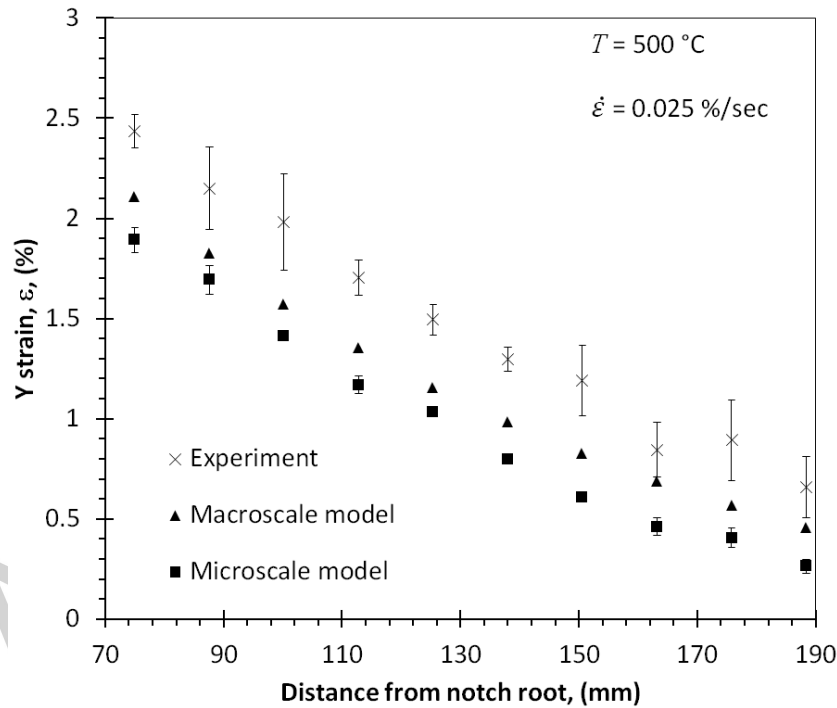


Fig 11: Microscale strain measurement within the area of interest: comparing the experimental, macroscale and microscale model results

While the macro and microscale models provide a similar prediction in Figure 11, the crystal plasticity model provides a more detailed representation of the microstructural behaviour of the material within the region of interest. This is illustrated by the plastic strain contour plot in Figure 12. As can be seen, by including the material microstructure and the crystal plasticity constitutive description, the microscale model shows regions close to the notch root with plastic strain levels as high as 10% (red regions) which are not predicted by

the macroscale model. An accurate prediction of damage and failure at the notch root requires the identification of the sites where these local strain inhomogeneities are present which would not be identified from the macroscale level model.

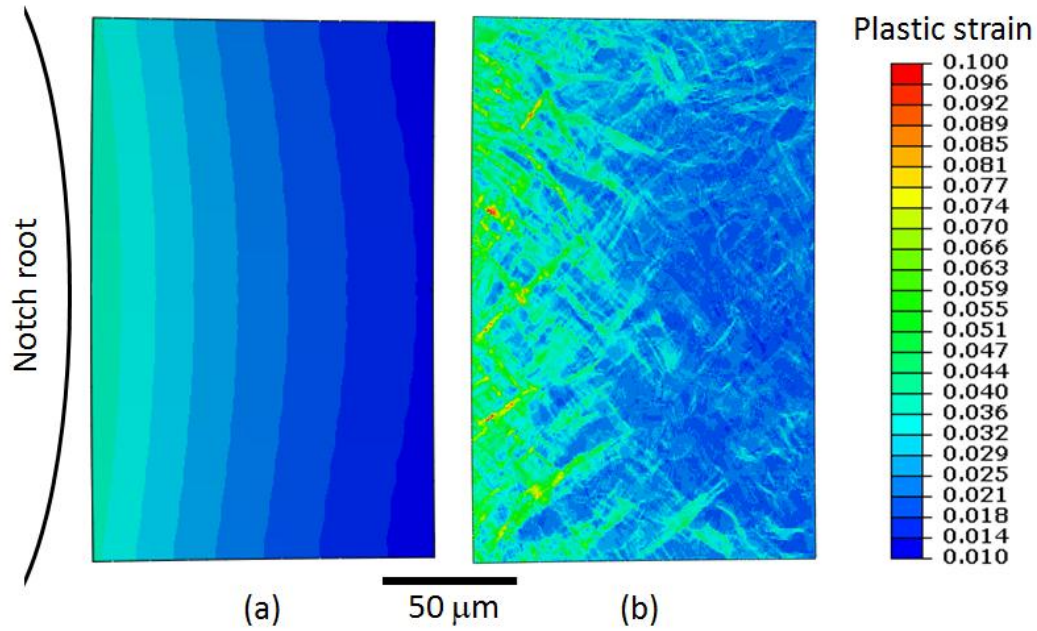


Fig 12: Plastic strain contour plots of the region of interest at the notch root: (a) macroscale model, (b) microscale model

The microscale model can also be used to predict directly the orientation change of blocks within the region of interest, as a further validation of the model. The orientation change due to deformation can be visually represented in an inverse pole figure for both the experimental measurement and the model prediction, as shown in Figure 13 for a selected region of the scan. In the inverse pole figure, shown in Figure 13(a), the initial orientations of the material points in the region shown are represented by the blue data points. The red data points in Figure 13(b) and (c) represent the orientation of the same region after deformation, obtained from the experiment and model, respectively. It may be noted that the direction of the change in orientation from the original to the deformed condition is similar in the experiment and model, though there are some differences in the magnitude of the change in orientations within the blocks, with the model (Figure 13c) predicting a wider spread in orientations following deformation. Figure 13(d)–(f) provides orientation distribution functions (ODFs) of the region shown. By comparing the initial ODF in Figure 13(d) with those obtained from the experiment and model shown in Figure 13(e) and (f), respectively, it is clear that there is a similar change in orientation density in the experiment and model due to the deformation. However there is a slightly lower peak density of orientation in the model (smaller red region near [101] in Figure 13f, compared with the same area in Figure 13e).

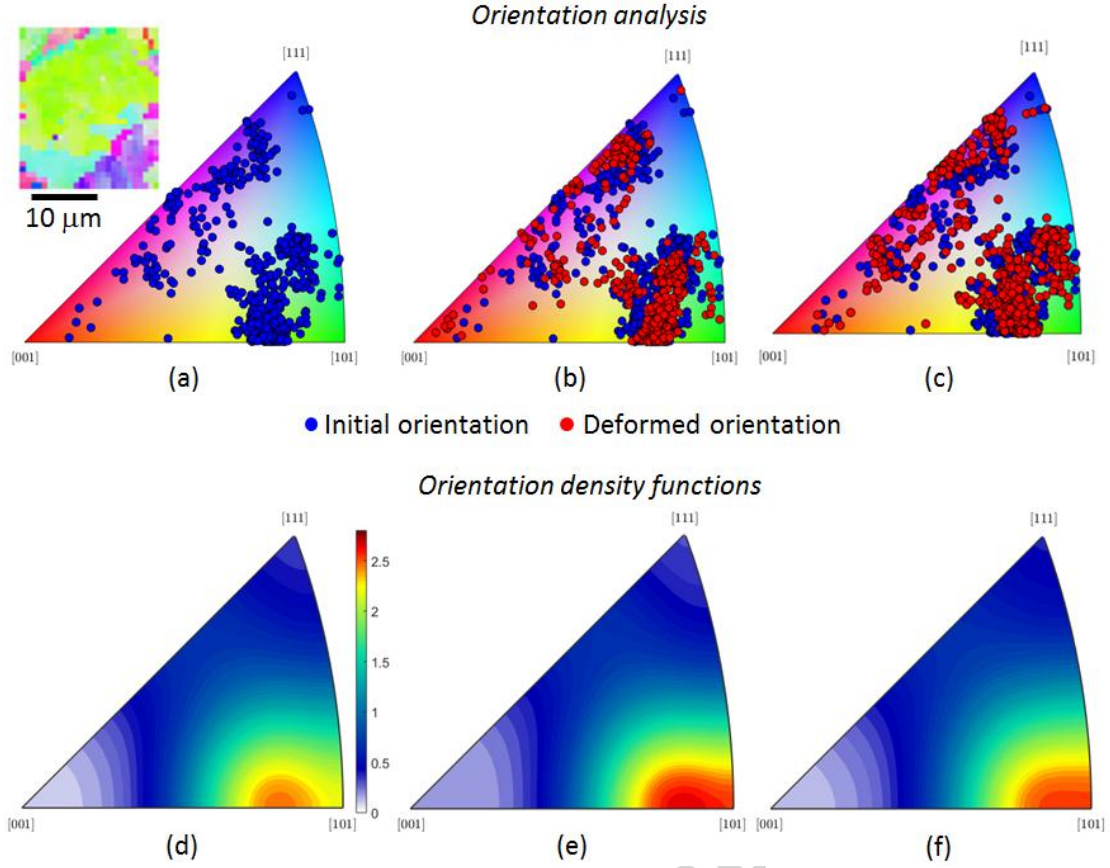


Fig 13: Inverse pole figures for initial and deformed region (a)–(c) orientation of each pixel within the region (a) initial state (b) experimental measurement, (c) model prediction; (d)–(f) orientation density functions (ODF) of the region (d) initial state (b) experimental measurement, (c) model prediction

In order to quantify this difference between the model and experiment the mean orientation (reference orientation) of a number of blocks (see Figure 14) was calculated before and after deformation in both the experiment and model. This analysis was carried out using the MATLAB based code MTEX (Bachmann et al., 2010a, 2010b) to determine the reference orientation of blocks/grains within a microstructure. The mean orientation difference (MOD) with respect to this reference orientation is obtained from Equation (7).

$$MOD = \frac{1}{n} \sum_{i=1}^n \theta_{mis}^i \quad (7)$$

In Equation 7, θ_{mis} is the misorientation between a certain pixel within the block and the reference orientation and n is the number of pixels within the block or region of interest. The MOD describes the average misorientation within a block/region with respect to a reference orientation. For martensitic steel, before deformation this is expected to be on the order of 2° , the average lath misorientation. A large change in this value due to deformation would indicate a significant level of non-uniform deformation within a block. The change in MOD before and after deformation obtained from these blocks for the experiment and model can be seen in Table 2. There is some discrepancy seen in Table 2 between the experimental results and those obtained from the microscale model. In all cases, except Region 2, the model predicts a small increase in MOD while the experiment predicts a decrease.

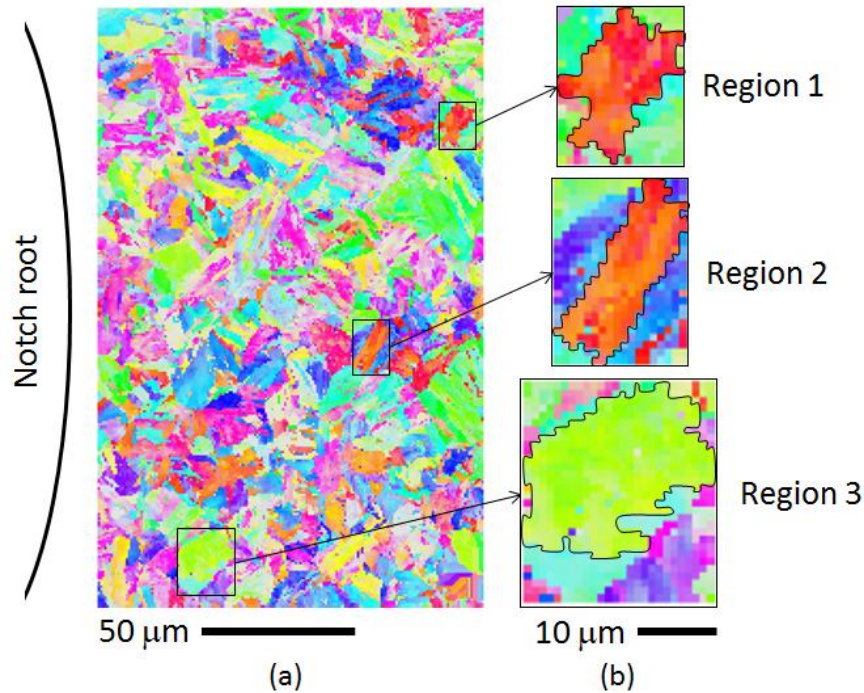


Fig 14: (a) EBSD map of the initial microstructure within the region of interest, (b) Three regions containing one full block (outlined in black in each case)

From the inverse pole figures presented in Figure 13 it is clear that the model predicts the correct direction of overall block rotation. This is evident from Figure 13(a)–(c) where the clusters of points, which represent blocks, move in a similar direction in both the model and experiment. The orientation density functions (ODF) in Figure 13(d)–(f) also show that the peak density of orientation changes in a similar direction in the experiment (Figure 13e) and the model (Figure 13f). However, as discussed the intensity of the orientation density is slightly lower in the model than in the experiment. This is due to the fact that the MOD of the region has decreased in the experiment after deformation, meaning the orientations of the individual pixels have reoriented closer to the reference orientation for the MOD measurement. The model predicts that the MOD increases, meaning that the orientations of the pixels have changed to an orientation further from the reference orientation. This causes the peak intensity of the predicted ODF to be lower than the measured value. Thus, the model correctly predicts the overall rotation of the block after deformation, but there is some discrepancy in the level of deformation being predicted within the block measured using MOD as seen in Table 2. However it may be noted that these changes are very small (a change in MOD of 0.2° corresponds to a shear strain of approx. 0.3%) and could be considered to be outside the resolution of the standard EBSD technique of approximately 0.5° in misorientation measurements (Brough et al., 2006; Humphreys, 2001; Maitland and Sitzman, 2007). Therefore the possibility that MOD is not a suitable measure to quantify the level of deformation at low levels of strain must be considered, in this particular case the change in orientation may be too low to be accurately measured using this technique. Therefore it is difficult to make a definitive judgement with regard to model validation using MOD based on these values.

Table 2: *The change in MOD (in degrees) for the whole region of interest and three smaller regions containing one full block, as outlined in Figure 14*

Area of interest	Initial MOD (degrees)	Source	Final MOD (degrees)	Change in MOD (degrees)
Overall region	2.58	Experiment	2.35	-0.23
		Model	2.69	0.11
Region 1	3.88	Experiment	3.24	-0.64
		Model	3.92	0.04
Region 2	3.62	Experiment	3.66	0.04
		Model	3.63	0.01
Region 3	3.52	Experiment	3.35	-0.17
		Model	3.80	0.28

At higher deformation levels, it is expected that experimental uncertainties will have a reduced effect of the results. This is illustrated by the results in Table 3, which provides the predicted change in MOD at higher levels of strain, determined from the FE microscale model. In this case, with an average microscale strain of 20%, the changes in MOD are greater than 3° (shear strain approx. 5%). It may be noted that for similar levels of deformation at room temperature, Golden et al., 2016a measured orientation change values of more than 4° (note that in Golden et al., 2016a, the measure of orientation change was that of crystal deformation C_d , Kamaya et al., 2005, rather than mean orientation difference (MOD)).

Table 3: *Microscale model case study results: Predicted change in MOD (in degrees) for average strain level of 20 %*

Area of interest	Initial MOD (degrees)	Final MOD (degrees)	Change in MOD (degrees)
Overall region	2.58	8.63	6.05
Region 1	2.91	5.93	3.02
Region 2	2.60	5.57	2.97
Region 3	2.14	8.63	6.49

CONCLUSIONS AND FUTURE WORK

As P91 is a widely used material for the power generation industry it is imperative that the behaviour of this material is understood at multiple length scales at temperatures representative of in-service conditions. In the current study a multiscale experimentally based modelling approach has been developed to simulate the high temperature deformation of P91. This modelling approach has been experimentally validated through the use of high temperature mechanical measurements and electron backscatter diffraction (EBSD) analysis. The approach presented consists of two finite element (FE) based models. The first is a specimen level model to predict the macroscale response of the material when subjected to high temperature loading. This experimentally calibrated FE model of the test specimen geometry shows excellent agreement in the force/displacement response of the material and the notch opening measurements obtained from the experiment at this length scale.

The second of these models is a crystal plasticity slip system based FE model to simulate the microstructural deformation of the material. This model has also been calibrated against uniaxial tensile data obtained from the material and the initial microstructural information is obtained through EBSD measurements of the experimental sample prior to deformation. The microscale model is driven by the nodal displacements of the macroscale model providing a link between scales and establishing a multiscale modelling approach. This model allows for the comparison of detailed microstructural deformation measurements with those obtained from the experimental EBSD measurements of the microstructure before and after deformation. An average strain can be determined by direct analysis of the EBSD maps generated by the experiment and model and close correspondence was obtained. The use of the mean orientation difference (MOD) within a block or collection of blocks to quantify the comparison between experiment and model was also examined. However, it was found that the measured and predicted changes in MOD are very small, close to the resolution limit of standard EBSD measurements. Future experimental work in this area will examine higher levels of deformation (up to 20 % strain) in order to introduce greater changes in MOD.

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DATA AVAILABILITY

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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