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DOI:

[10.1007/s11340-019-00528-w](https://doi.org/10.1007/s11340-019-00528-w)

Document Version

Accepted author manuscript

[Link to publication record in Manchester Research Explorer](#)

Citation for published version (APA):

Smith, A. D., Donoghue, J., Garner, A., Winiarski, B., Bousser, E., Carr, J., Behnsen, J., Burnett, T., Wheeler, R. W., Wilford, K., Withers, P., & Preuss, M. (2019). On the application of Xe⁺ plasma FIB for micro-fabrication of small-scale tensile specimens. *Experimental Mechanics*. <https://doi.org/10.1007/s11340-019-00528-w>

Published in:

Experimental Mechanics

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On the application of Xe⁺ plasma FIB for micro-fabrication of small-scale tensile specimens

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Keywords: Xe⁺ Plasma FIB-SEM Dual Beam Microscope, In-situ Mechanical Testing, Digital Image Correlation, X-Ray Computed Tomography, Finite Element Analysis, SA508-4N steel

Abstract

In order to perform site-specific mechanical studies to examine the contribution or interaction of a material constituent, a reliable methodology for the production of small-scale samples is required. With the high milling rates achievable with the newly developed Xe⁺ plasma FIB (PFIB), it is now possible to manufacture specimens approaching the smallest representative volume (SRV). In this study, a methodology has been developed that allows for the manufacture of mesoscale specimens approaching SRV to be tested in tension. It is demonstrated that yield and tensile strength measured in specimens of this scale are representative of bulk behaviour, of the properties measured the strain hardening exponent was found to be dependent on scale.

Introduction

Small-scale mechanical testing of limited volume specimens can provide unique insight into a wide range of different material investigations, including film/coatings [1,2], testing of phase/grain boundary interactions [3], ion irradiated layers [4–6] and radioactive materials [7]. For site specific applications, conventional mechanical tests with large samples typically

incorporate the region of interest with other regions and separating the relevant component from the global response can be difficult. Such approaches become impractical when the region of interest becomes minimal in size relative to the bulk, with the situation complicated further by property gradients and interactions between constituent parts [8]. A more practical option is to reduce the size of the samples so that they only contain the region or regions of interest. Unfortunately, testing at the sub-micron to $\sim 2\ \mu\text{m}$ length scale introduces a number of challenges, including a well-documented size effects in metals, where an increase in hardening is found to be inversely proportional to specimen diameter [9]. This is understood to occur due to the limited number of dislocation sources as the length scale is reduced, causing the material to approach its theoretical strength [10–14]. A comprehensive review of these size dependant mechanical properties has been performed by Greer and Hosson [15]. Beyond the threshold for size dependant hardening a specimen will typically comprise of a limited number of grains due to the constraints of available preparation techniques. It has been suggested that the grain size has to be sufficiently fine in small-scale specimens during plastic deformation to achieve the smallest representative volume (SRV) that effectively emulates the bulk [16], although this threshold is currently unknown. This severely limits the materials that can be reliably investigated using traditional sample preparation techniques.

Perhaps the greatest challenge of testing at these small scales is the reliable fabrication and testing of specimens. Previous studies have used Ga^+ focussed ion beam (FIB) to machine specimens from regions of interest and utilised micro- and nano-indentation or piezo-actuated loading to perform tests on miniaturised specimens in bending [17], compression [4,5,18,19] and tension [6,16,20]. However, the limited milling rate of Ga^+ FIBs restricts the maximum practically achievable cross section of tensile specimens to $\sim 10 \times 10\ \mu\text{m}^2$. For the large majority of engineering materials, the sampled volume would be limited to only a single or a few grains

and therefore the SRV for bulk material properties is unlikely to be met. These types of samples are therefore better suited to the study of single and bi-crystal properties [6,20]. A further issue with Ga^+ FIBs for micro fabrication is the implantation and subsequent diffusion of Ga^+ ions that can lead to damage such as phase changes and segregation to grain boundaries that can affect mechanical properties of sensitive materials [21].

Both of these limitations can be mitigated by the use of the Xe^+ plasma FIB (PFIB). In Ga^+ FIBs the divergent nature of the point source results in a spherical aberrated non-gaussian beam profile, limiting practical currents to ~ 65 nA [22,23]. Conversely, whilst exhibiting a larger virtual source the more collimated beam of the PFIB means that a focussed spot can be retained at milling currents in excess of 2500 nA [22]. The higher usable current, combined with an increased sputter efficiency for most metals, has a dramatic effect on milling rates which are increased by up to two orders of magnitude compared to a Ga^+ FIB. This greatly improves the practically achievable scale of specimens [24,25]. In addition, by using an inert species, the Xe^+ FIB does not promote phase changes due to micro-alloying as seen with Ga^+ FIB [21]. Furthermore, it has been clearly demonstrated that the use of Xe^+ ions results in a reduced depth of amorphisation when compared to Ga^+ FIB [26,27]. The efficient sputtering rate and relatively high operating currents make the Xe^+ PFIB an ideal candidate for the fabrication of mesoscale tensile specimens. By employing PFIB milling for the preparation of uniaxial tension mechanical test-samples, it is possible to achieve sample cross sections an order of magnitude greater in size than with conventional Ga^+ FIB. With this increased specimen size, materials can approach the SRV, provided they have sufficiently fine grains, which will allow these small-scale samples to give representative mechanical properties to bulk [16].

In this study, a sample preparation methodology is presented showing how PFIB technology can be used to prepare site-specific mesoscale tensile samples to contrast against the standard uniaxial flow curves gathered using a technique set out in ASTM E8/E8M [28]. SA508 Grade 4-N steel with martensite lath size of $\sim 5\text{ }\mu\text{m}$ was studied using this approach. This material was chosen due to the fine microstructure, whilst also having industrial relevance due to its proposed use in the nuclear industry as a candidate reactor pressure vessel material. Ion irradiations with low penetration depths and high damage rates are being increasingly used to simulate the effects of neutron irradiation and therefore this type of small scale mechanical testing is likely to be of great interest to the nuclear community. Specimens have been prepared both with and without overtill to assess the effect of the well documented sidewall tapering inherent to top down FIB milling [29].

Experimental Methods

Material and preparation

The material used in this study was forged SA508-4N in the quenched and tempered condition supplied by Rolls-Royce plc. Rectangular specimens were first extracted by electrical discharge machining (EDM) with the forging transverse direction parallel to the face normal of the specimen (TD//33). Coupons of dimensions $27 \times 3 \times 1\text{ mm}^3$ were ground mechanically to remove the EDM recast layer from all faces using 400 grit silicon carbide abrasive paper. Further mechanical grinding was performed with 400 grit silicon carbide paper to remove approximately $700\text{ }\mu\text{m}$ of material. Successive grinding steps through to 4000 grit, took the remaining coupon from $\sim 300\text{ }\mu\text{m}$ to a final thickness of $60 - 100\text{ }\mu\text{m}$. The resulting thin foil was polished with diamond paste, with a final polishing step using diluted colloidal silica. A standard TEM sample punch was used to prepare the foil into a 3 mm diameter disk (Figure

1a). Burrs were carefully removed with 4000 grit abrasive paper. The foil disk was mounted in wax between two glass slides, ground into a 'half-moon' and attached to a brass sample mount using cyanoacrylate adhesive (Figure 1b).

Plasma focussed ion beam milling

A schematic of the PFIB preparation procedure is illustrated in Figure 1, whereby a region of the ground disc is locally thinned to produce a uniform region, from which the tensile sample geometry is subsequently milled. All milling was performed at 30 kV using an FEI Helios G3 PFIB DualBeam™ equipped with a Xe⁺ plasma ion beam column. The sample mount was fixed to a 45° pre-tilt holder using a grub screw and a 7° stage tilt was used so that the ion beam (at 52°) would be incident normal to the specimen surface. All alignments were made relative to the sample mount, since the sample mount attaches directly into a housing in the mechanical testing equipment, this ensured that the tensile specimens were well aligned along the loading axis. An initial cut was made to mill away the mechanically deformed layer on the top edge of the disk, normal to the X-Y plane, and to ensure the sample longitudinal axis ran parallel to the mount. This rough milling was performed using a cleaning cross section, at the highest beam current of 1.3 μA, whereby the cut is made up of a series of successive line scans progressing from one side of the specified area to the other (i.e. top to bottom), rather than a dynamic multidirectional scan. The z-depth of each line cut was set at 150 μm, where the PFIBs unit of depth is defined in terms of fluence required to mill through that depth of Si. The ratio of milling efficiency in steel to Si was found to be approximately 1:1 so no depth correction was required. Following the high current milling, a further cut was made at 0.18 μA with the same z-depth. The lower current polishing step was used to reduce the curtaining (striations caused by local differential mill rates of microstructural features) associated with high current milling and avoid the propagation of those curtains in subsequent preparation steps.

The specimen was rotated by 180° on the 45° pretilted holder, which allowed thinning cuts to be made approximately parallel to the surface, with a $\pm 2^\circ$ over/under-tilt to allow for the mill taper (largely due to beam shape and redeposition [29]) to ensure the thinned section was uniform thickness throughout the depth. An initial thinning step was made to align the lamella with the specimen mount and to reduce the thickness to approximately $40\text{ }\mu\text{m}$ (Figure 1c). The $350\text{ }\mu\text{m}$ wide lamella was milled at $1.3\text{ }\mu\text{A}$ to a depth of approximately $500\text{ }\mu\text{m}$ using a dynamic scan in all directions. As this high current milling resulted in deep, pronounced curtaining approximately $2\text{ }\mu\text{m}$ in width and $1\text{ }\mu\text{m}$ deep, a rocking mill was employed at a lower beam current to polish the faces. Platinum was also deposited on the leading edge in order to protect from further curtaining during the subsequent thinning stage (Figure 1d). The protective Pt layer was deposited using the ion beam at 8 kV and a current of $0.13\text{ }\mu\text{A}$ ($\sim 170\text{ pA}/\mu\text{m}$), until the layer reached a thickness of $\sim 5\text{ }\mu\text{m}$.

The rocking mill, whereby the angle of the milled face is alternated by $\pm 2^\circ$ relative to the incident beam throughout the milling process so that each stage of the mill reduces the curtains from the previous step (explained in detail in section 2.3 of [23]) is carried out in an automated procedure using FEI Auto Slice and View 4 (ASV4) software package. In order for the software to track the region, fiducial markers were milled on the top edge of the lamella with a relatively low beam current of 15 nA to ensure sharp edges, and thus good contrast for automated tracking by the software. This rocking mill was performed across a $300\text{ }\mu\text{m}$ wide region on each face at 30 kV and a current of $0.18\text{ }\mu\text{A}$ to produce a final lamella of $\sim 30\text{ }\mu\text{m}$ thickness (Figure 1e). Each step in the rocking mill took approximately 7 minutes to complete after which the mill line was advanced 150 nm . Following completion of each line, a secondary electron (SE) image was acquired to monitor progress before the stage automatically proceeded to the next mill

angle. Images were acquired at 8 kV, with a dwell time of 3 μ s taking approximately 10 seconds to collect each frame. As it is required for the mechanical testing that the thinned region be entirely uniform, an additional issue is presented by the microscope stage only possessing a single tilt axis. With the tilt axis utilised for the 2° automated rocking mill, overtilt to take account of mill taper is not possible simultaneously. In an effort to mitigate the tapering inherent to FIB sample manufacture, using a top-down approach [30] (i.e. the sample will be wider at the bottom compared to the top of the specimen), a bespoke pre-tilted specimen stub was prepared to provide 2° overtilt. In order to investigate the effect of this taper, two specimen batches were prepared in total, one prepared using the pre-tilt holder, and the other mounted vertically with no overtilt.

Milling of the thinned lamella to define the shape of the test specimens required remounting the lamella back to the 45° pre-tilted holder to orientate the ion beam normal to the sample surface. The width of the lamella was sufficient to arrange 2 parallel samples (Figure 1f). Each cut was milled in series at 0.18 μ A, with the gauge sections milled last to avoid redeposition of sputtered material back onto the gauge length. The cuts were made with a width of 5 μ m and a z depth of 300 μ m. The excessive milling depth was found to produce a square edge and the best possible surface finish. This mill was performed with no overtilt resulting in a trapezoidal, but constant, cross section. Rectangular holes, to allow for loading pin insertion, were milled into the tab at 0.18 μ A. Specimens were prepared to maintain a minimum of a 5:1 width to length ratio, outlined in ASTM E8/E8M [28] for testing of bulk materials which does not reference a minimum gauge section dimension provided the criteria for ratios are met.

The entire PFIB preparation process takes approximately 2 work days to complete when preparing the 2 samples as shown but can be further optimised using thinner initial foils and

improved alignment on the specimen mount. The automated routines do not require an operator to be present, which accounts for approximately 50% of the preparation time.

Orientation mapping

Orientation mapping was performed using a FEI Helios FEG-SEM-PFIB equipped with an Oxford Instruments Nordlys Max² EBSD detector operating at 20 kV, with a current of 22 nA and a 0.1 μm step size. Kikuchi patterns, taken at 8x8 binning, were indexed using AZtecHKL software at ~ 50 Hz. Separate maps were taken over the entire gauge section for each tensile sample post milling. Maps were processed using HKL Channel 5, with grain boundaries defined by a misorientation $> 10^\circ$.

Micromechanical testing

In-situ mechanical tests of the PFIB milled samples were performed using a Microtesting Solutions (Hilliard, OH, USA) μ -Test Rig (MTR-3), mounted in a Zeiss Sigma FEG-SEM. A schematic of the piezo-actuated MTR is illustrated in Figure 2. In order to load the sample, a voltage of 150 V is placed across the piezo actuator that is connected to the loading pin, extending it ~ 100 μm . The sample is then manipulated via the piezo x-y-z positioning stage so that the loading pin sits within the rectangular holes in the specimen tab (as described in ref. [16]). During testing, decreasing the applied voltage causes the piezo actuator to contact and load the specimen via the loading pin. The quasi-static tests were performed using voltage control, which would correspond to displacement control in a conventional mechanical test. For these experiments, the displacement rate was ~ 100 nm s^{-1} with hold points at 200 nm displacement increments. Secondary electron images were taken at each hold point at 8 kV and ~ 0.5 nA. Scans took approximately 4 seconds to complete, with a dwell time of 50 ns and line averaging x 4 to increase signal/noise ratio from the rapid acquisition. Images were taken at

hold points throughout the test in order to record displacement by tracking two Pt fiducial markers which were deposited using the PFIB (Figure 3). Macroscopic strain was calculated from the fiducial displacement by the MTR-3 control software written in Labview. The applied load was recorded by the test rig using an internal load cell with a total capacity of 453 g.

Full-field plastic strain mapping

The correlation of a series of deformed images to an unstrained reference image allows local displacement to form an image of spatially resolved strain intensity. This can be applied to the raw surface, provided there is sufficient contrast [31]. However, to achieve a high spatial resolution a specimen surface is typically decorated with a dense array of markers. Pattern size and density ultimately dictates spatial resolution and to achieve full field coverage a variety of techniques have been employed to achieve resolutions capable of measuring sub grain strains [32–35]. As these techniques would be difficult to apply to small scale specimens in this study a sufficiently fine surface pattern is achieved using electron beam deposited platinum nano-dots [36]. A bitmap image of a desirable pattern was created for the deposition software to follow. It was found in order to obtain a pattern at a sufficient resolution it was necessary to deposit at a high magnification resulting in a field width that was significantly less than the area to be covered. Therefore, it was required to move the sample repeatedly during deposition in order to cover the entire gauge length in nano-dots. It was found that a distinct pattern with a mean particle diameter of 150 nm could be deposited over an area of 15 μm by 18 μm in just under 10 minutes using a focused electron beam of 11 nA at 20 kV with a 1 μs dwell time. Therefore, in order to cover the gauge face of 30 μm by 250 μm an array of 2 x 13 individual depositions was required (Figure 4). While it is possible to apply a repeating pattern manually, the process is time consuming and sensitive to errors so automation is desirable. An automated routine was prepared using FEI iFast, a visual scripting software package that can control many

aspects of the dual beam. The only inputs for the iFast routine are the desired field width for each pattern to be deposited over, and the number of rows and columns for the pattern to be repeated over. The routine automatically inserts the GIS (Gas Injection System) needle, fine focuses the image, sets the correct deposition conditions, proceeds with deposition, moves the stage, and repeats. The beam is rastered as successive single line scans rather than a serpentine scan to maintain distinct features. In the event that there is drift or charging, the single line scan will simply distort the image which would not be a problem in case of providing markers, whereas, a degradation in particle distinction would have a deleterious effect on the strain calculation. Following completion of each tile, the stage is moved to the next position and the process is repeated. Patterning was applied to a specimen prepared using the method that employed the pre-tilted specimen holder. Local strain was calculated using the commercially available LaVision's DaVis 8.4 digital image correlation software package.

X-ray computed tomography

X-ray computed tomography (X-ray CT) was performed on specimens prepared by PFIB, with and without overtilt, to assess the degree of gauge section taper. This was carried out using a Zeiss Versa XRM-520. An accelerating voltage of 100 kV was used with a current of 91 μA , equating to 9 W of power. 1601 projections of circular scanning were taken of each specimen, with a 40 s exposure, using a low energy filter at the source (LE3). The source and detector were positioned 14.04 mm and 37.02 mm from the specimen respectively. Images were collected with an optical magnification of 20 x with 2 x camera binning; the resulting voxel size was 0.3693 μm . Images were processed and reconstructed using Zeiss Scout and Scan Reconstructor software. Analysis and visualisation was performed using FEI Avizo 9.2 software.

Finite element analysis

Finite element analysis (FEA) was performed to differentiate the influence of geometry from scale and microstructure. This was performed using Dassault Systemes SIMULIA Abaqus/CAE 6.14-2, with 2-D plane-strain models constructed using the software package. Synopsis Scan IP software package was used to reconstruct and apply an adaptive tetragonal-mesh to the tomography results. Figure 5 illustrates the meshing of each example, a) plane strain and b) based on X-ray CT data. Plane strain models comprised of $1.5\text{ }\mu\text{m}$ three node linear elements (CPS3) and $1.6\text{ }\mu\text{m}$ - $1.8\text{ }\mu\text{m}$ four node tetrahedral elements (C3D4) for the tomography-based model. The plane strain test contained three boundary conditions in order to best represent the experimental conditions: a static boundary at the right-hand y-surface and a displacement ($25\text{ }\mu\text{m}$ lateral) boundary condition at the left-hand x-plane. A symmetric boundary was applied to the lower y-plane (Figure 6a). In the tomography-based model, a static boundary applied to all element nodes at the base, with movement in all linear and rotation axes disallowed, to simulate the mount. Loading was applied to the same region as in the experiment and was achieved by a displacement boundary condition at the surface elements making up the loop interior (Figure 6b), constrained in all but the displacement direction. Since the scale of the specimens is assumed to be representative of bulk behaviour, bulk material properties were obtained experimentally according to ASTM E8/E8M [28] and the full flow curve applied as separate elastic and plastic parameters.

Results & Discussion

Microstructure and sample geometry

An example of the final geometry for specimens prepared with and without overtilt, as measured by X-ray CT, is displayed in Figure 7, and SE micrographs of each batch are shown in Figure 8. The mean gauge section measurements for the sample with no overtilt were: 132

μm (L); 17.5 μm (W); 40 μm (T). The gauge section thickness was observed to deviate by $\pm 8\%$ from the mean value across its length, corresponding to a taper angle of 1.6° . Furthermore, the taper was not a constant gradient, visible by the mean value (0% deviation) being slightly off centre towards the top of the tab in Figure 7. The discontinuity is also visible in the electron micrographs (Figure 8a), where the pronounced curtains are a result of the high current used during the initial thinning step, which could not be removed during the lower current automated rocking mill process. Both of the samples prepared using a 2° overtilt had mean gauge dimensions of: 151 μm (L); 19 μm (W); 32 μm (T). The overtilt of 2° resulted in a reverse taper of -0.44° , relating to $\pm 2\%$ deviation in thickness from the mean. The mean cross section is situated at the centre of the gauge section indicating a constant taper and an adequate line current density in the rocking mill step. In both batches prepared with an without overtilt, all taper was due to top down milling so the only gradient along the gauge length was through thickness. No tapering was present along the width in either preparation method, other than the aforementioned trapezium, with no variation along the gauge length in this axis. From the microstructure maps acquired using EBSD in Figure 9, it is estimated that these samples have approximately 8 - 10 prior austenite grains (PAGs) within their gauge length. The average lath size was $\sim 9\ \mu\text{m}$ in length, and $3\ \mu\text{m}$ in diameter. Despite the slight variation in gauge dimensions between the two sample sets, the sampled volume remains approximately equal, and is calculated to be ~ 1000 laths in both cases. Measurement of the width of both sides of the sample was performed using Image-J [37] and used to calculate the cross sectional area assuming an ideal trapezoid, however blunt edges and bowing were not accounted for.

Mechanical testing

Stress strain curves for the overtilted and non-overtilted specimens are displayed in Figure 10. For comparison, a flow curve from a standard cylindrical specimen of the same material with

a 7 mm diameter which was carried out according to ASTM E8/E8M, is also included in this figure. Young's modulus for the measured samples during the primary loading stage was far higher than would be expected: 254 ± 3 GPa compared to 189 ± 2 GPa for the bulk. This is thought to be due to a slight specimen misalignment leading to a degree of compliance. A more accurate modulus was obtained during an unload cycle in one of the tests, which gave a more consistent measure of 178 ± 3 GPa. Proof stresses (0.2 % yield stress) were 651 MPa and 641 MPa for the overtilted and non-overtilted samples respectively and were in good agreement with the bulk value of 646 ± 9 MPa. The yield transition was dampened and gradual in the sample without overtilt, which is evident in the proportional limit of 555 MPa as opposed to 598 MPa for the sample with overtilt and 610 MPa for bulk.

The strain hardening parameter were calculated by taking the log-log gradient of the true stress-true strain curve for the region between proof and peak stress, which provides the strain exponent of the Hollomon equation (n) [38]. In the bulk material, this parameter was calculated as 0.122, whereas the values for the PFIB prepared samples were significantly lower. While the proof stress region was well represented, the largest variation from the bulk properties was in the plastic regime, with a strain hardening exponent of 0.094 and 0.083 for overtilted and non-overtilted respectively. Peak stress and strain to failure are also reduced, albeit to a lesser extent. There is a possibility that this behaviour is due to gauge section aspect ratio (T/L) rather than scale or taper. Previous work by Zhao et.al found that the post necking strain to failure can be extended by increasing the ratio of gauge section thickness relative to its length [39,40].

An anomalous flow curve was generated during testing of one the overtilted samples. The behaviour displays similar characteristics to the overtilted specimen shown in Figure 10, but

with a sudden load drop at 4.4% strain. This is shown in Figure 11 (a), a comparison of the orientation map and the micrographs taken during the test (Figure 11 (b) and (c)) indicates that this behaviour is due to highly localised strain at a PAG boundary, which is highlighted on the figure. Analysis of the micrographs found that the onset of the strain localisation began manifesting at 1.76 % strain. Unsurprisingly, as this test exhibited a slightly diminished plastic response due to the sharp loss in strain hardening at the peak stress, a slightly lower peak stress of 728 MPa was measured than in any of the other tests, however, a similar strain hardening parameter was observed to the other overtilted specimen of 0.092. Localised shearing of this type was not observed in any of the other tensile tests despite all containing a similar number of PAG boundaries. One possibility is that this may have been a unique situation where the PAG boundary may have extended through the full gauge thickness and was the only one in the tested samples that did so. However, the finite element modelling of tetragonal meshed X-ray CT samples, displayed in Figure 13, indicate that the PAG boundary is situated in a location where the strain is expected to be highest due to geometry. It is therefore also possible that the other samples do contain PAG boundaries that facilitate this mode of localisation but are situated outside of this high strain region. The localisation is arrested at the neighbouring boundary with subsequent deformation occurring in the same manner as the other tests. Arresting of strain localisation implies that the neighbouring PAG boundary does not span the entire gauge thickness.

Full-field plastic strain mapping

Deformation mapping in conjunction with orientation mapping provides a level of insight with regards to intergranular strains that cannot be achieved with standard electron microscopy. Due to the short working distance generally required for high resolution strain mapping in an SEM, the majority of high resolution digital image correlation (HRDIC) is performed ex situ [32,41–

43], and therefore with load removed during imaging and only a small proportion of the specimen imaged. In the current work, correlated images are collected under static load, therefore, include elastic as well as plastic strain. Furthermore, the reduced specimen scale allows for data to be collected over the entire gauge length.

Full-field strain maps were calculated at every ~ 200 nm displacement of the loading pin for the patterned, overtilted sample. Figure 12a displays the progression of strain in the region experiencing significant localisation, with deformation mapped to the undeformed coordinates. In this region a packet of laths is clearly the origin of the localisation, which occurs at the onset of plastic deformation. As the test progresses, the strain in the initial region saturates as the region hardens and imposes a plastic strain on the surrounding grain. The surrounding regions saturate and plastic deformation extends to the surrounding regions, with strain appearing to radiate out as plumes from the initial point of localisation at the conclusion of the test. The accumulated maximum shear strain, at 3.9% applied strain, is displayed in Figure 12b. Although the resolution is limited when compared to some other HRDIC work, due to the larger scale decoration, it is sufficient to reveal deformation within individual grains. Clearly evident in the maximum shear map is a highly localised straining in a single location that is orientated at 45° to the loading axis. Also apparent is localised deformation at prior austenite grain boundaries at the opposite end of the gauge length. Some artefacts are present in the maximum shear map consisting of vertical and horizontal lines that are alternately above and below the average background. These artefacts are clearly related to the construction of the underlying bitmap used as a template for each tile, which itself comprised of 4 repeating tiles. A noise is generated in the overlapping regions of the bitmap occurring at twice the frequency of each deposited tile. The orientation map of this sample is given in Figure 12c with the region of highly localised shearing marked. Shear is found to be manifested in a packet of laths

orientated perpendicular in the $\{250\}$ and $\{05\bar{2}\}$ with respect to the loading direction, visible as red and blue in marked region of the Euler map (Figure 12a & c). This orientation is shared with some nearby laths which show no apparent strain localisation. Therefore, it is thought that the preferential deformation of this group of laths is encouraged by the geometry of the laths, rather than to Schmid factor alone. The cluster of laths in the centre of the shear banding has the longest dimension orientated at 45° to the loading axis.

Finite element analysis

Figure 13 a and b are profiles of strain in ϵ_{xx} (parallel to loading axis) recorded by image correlation of the undecorated surface imaged in the SEM (Figure 3) and FEA simulations applied to X-ray CT measurements. Both profiles were taken at 8 % total elongation and were normalised against their maximum value to allow direct comparison of distribution between specimens. Correlation of raw surface features leads to lower spatial resolution ($\sim 5 \times 5 \mu\text{m}^2$), and significant noise in local strain measurements when compared to the Pt decorated surface. Despite the noise, the measurement agrees with the normalised maximum principal strain measurements of the model. Both the samples were found to undergo localised deformation in the regions predicted using FEA. A broader distribution of plastic strain is visible in the 2° overtilt sample, whereas, it is noticeably more localised in the sample with no overtilt. The strain profiles for the overtilted sample displays a broad distribution of strain gradually decaying from the neck region over the majority of the gauge section. Conversely, the specimen prepared with no overtilt displays a sharp peak in the region of the neck with steep decay.

To assess the effect of tapering geometry alone, a plane strain model was constructed with the only varying parameter being the taper. Figure 14 shows the effect of various tapers angles (in degrees) on stress-strain behaviour. Stress-strain data was extracted as the average of in-plane

stress and strain for all elements in the model up to 10 % strain. It was found that increased taper suppresses the plastic response of specimens and dampening of the yield transition, showing good agreement with the experimental observations in Figures 9 and 12. This is also in agreement with observations made by Singh et al [44].

Figure 15 shows a comparison of the effect of taper angle on (a) the maximum stress, and (b) the strain hardening exponent for both the FEA and experimental results. The dashed line represents the bulk response at 10 % applied strain. The FEA responses are given for both the X-ray CT 3D and 2D models in Figure 5. FEA data plotted as a function of taper converges with the bulk properties in a linear fashion. The results from the FEA modelled from the X-ray CT geometry agree well with both the 2D plane strain FEA and the experimental results. A similar trend is observed with both FEA models with good agreement to bulk properties at a taper angle of 0° , and both the maximum stress and the strain hardening exponent dropping away in a linear fashion as the taper is increased. Also plotted on Figure 15 are the experimental measurements of the non-tilted and overtilted samples taken from the stress strain curves in Figure 10. The maximum stress recorded experimentally is in good agreement with the FEA results, however, the experimentally recorded strain hardening exponent is not so accurately represented by the models. The difference in strain hardening behaviour is thought to be due to the effect of relative grain size, which is not taken into account in the models. An effect of specimen size on strain hardening in polycrystalline samples is well known, and is thought to arise from the ratio of grains sitting within the volume to that of grains intersecting the free surface [14]. Fewer constrained grains at a free surface accumulate dislocations at a lower rate during deformation, due to an easier path for annihilation, than those that are constrained in the bulk [45,46]. In addition, the build-up of back stresses is limited in those grains and therefore they do not inhibit the operation of dislocation sources to the same extent as a fully constrained

grain. A recent study was performed by Kumar et.al into the effect of specimen geometry on bulk samples including the specimen thickness to grain diameter ratio (t/d) for various steels [47]. It was demonstrated that the loss of strain hardening due to t/d was in the order of 0.03 for the most similar alloy (CrMoV) to that studied here. In the present study, the measured loss of geometry-calibrated strain hardening ($n^{(FEA)}-n^{(exp)}$) in this study was ~ 0.02 . Work by Keller et.al [49–51] showed that flow stress, hardening, internal back stresses and dislocation structures is modified in specimens containing less than 4 grains through the smallest dimension. Initial flow stress (yielding) was not greatly reduced in the present study, but the negative effect of hardening is consistent with their observations. Although there are a large number of tempered martensite laths within the gauge length, which were originally thought to provide sufficient boundaries within the material to exhibit bulk behaviour, they clearly do not satisfy this requirement. This effect is most likely due to the common orientation of the tempered martensite laths within the prior austenite grains. It is understood that coherent boundaries meeting the criteria for slip transmission provide less effective barriers to dislocation motion [48]. This would further reduce the magnitude of back stresses and have a diminishing effect on strain hardening. Therefore, the effect of t/d due to a loss of constraint coupled with common orientation within PAGs is considered to be the likely contributor to the degradation of strain hardening behaviour observed here.

Conclusions

A method for preparation of micro-scale specimens for mechanical testing by Xe^+ Plasma FIB has been developed and tested. The main conclusions are as follows:

- The use of a Xe^+ plasma FIB has allowed for the manufacture of samples at a scale approaching the smallest representative volume in a reasonable timescale of ~ 2 days for a sample with a lath size of about $5\text{ }\mu\text{m}$.
- A methodology has been developed that produces samples with a relatively parallel gauge length and a deformation-free, polished surface that can be used for subsequent EBSD analysis and also allows the application of patterns for HRDIC.
- By comparison with FEA modelling, it has been demonstrated that the observed yield dampening and reduction of peak stress are a result of a gauge length taper rather than scale.
- Currently it is not possible to fabricate specimens with no gauge section taper using pre-tilted holders. This is due to the length of the specimens and the lack of fine control provided by a pre tilted holder. A double tilt axis or gimballed microscope stage would allow for greater flexibility and control of the overtilt during specimen fabrication. Eliminating specimen taper will allow for truly uniaxial tests and increase the reliability of results.
- The resulting mesoscale specimens exhibit bulk behaviour with the only scaling effect being intrinsic and microstructural, which is understood to be related to the t/d ratio, this could be overcome by grain size reduction or increasing gauge volume.

Acknowledgements

The authors would like to thank the EPSRC for funding through EP/J021172/1. The authors would like to acknowledge the assistance provided by the Manchester X-ray Imaging Facility, which was funded in part by the EPSRC (grants EP/F007906/1, EP/F001452/1 and

EP/I02249X/1). E. Bousser would like to acknowledge the support of the Natural Sciences and Engineering Research Council of Canada through the Postdoctoral Fellowship program.

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Figures

Figure 1. Schematic of the specimen preparation route, (a) mechanically thinned 3 mm diameter, 60-100 μm thick disk; (b) Mechanically ground into a half moon attached to brass mounting fixture; (c) High current (1.3 μA) PFIB mill to remove cold worked layer; (d) Fiducial markers placed for automated positional correction; (e) 2 $^\circ$ automated rocking mill of both sides at 0.18 μA to a curtain free 300 x 500 x 30 μm lamella; (f) Final milling of T-bone specimen geometry.

Figure 2. Schematic of piezo actuated μ -Test Rig used for in-situ testing within SEM

Figure 3. Secondary electron image used for strain measurement. Displacement is recorded by imaging at each hold point as the test progresses and images are later processed using digital image correlation to calculate strain.

Figure 4: Electron beam deposited platinum decorated specimen, comprising of 26 repeating tiles with a mean speckle diameter of 150 nm.

Figure 5: Examples of meshes used for FEA analysis using, a) 2D plane strain & b) mesh modelled from X-ray CT reconstructions.

Figure 6. Schematic of boundary conditions for each of the models simulated using finite element analysis

Figure 7. X-ray CT reconstructed models of samples prepared using (a) no overtilt and (b) 2° overtilt, coloured according to the deviation from the mean thickness in Z. Blue arrow indicates region where specimen cross section is equal to mean cross section in both specimens.

Figure 8: Secondary electron images of sample batches prepared (a) without overtilt and (b) with overtilt.

Figure 9: Orientation maps recorded using EBSD, displayed in Euler colours, showing typical microstructures in tensile samples, both orientation maps are taken from the overtilted batch.

Figure 10. a) Flow curves of PFIB samples prepared with and without over tilt, bulk standard curve is overlaid; b) Yielding behaviour of the same curves shown in (a), preparing with overtilt is observed to mitigate the dampening of the yield point.

Figure 11. a) Flow curve of specimen containing PAG running through the gauge section thickness; b) secondary electron image illustrating point of strain localisation prior to failure; c) Location of PAG boundary highlighted on EBSD orientation map. *Euler colourbar is displayed in Figure 9.*

Figure 12: a) Progression of maximum shear strain throughout tensile test in the region of localisation. b) Full-field strain maps correlated using 12x12 binning windows with 25 % overlap, displayed in terms of maximum shear strain (%). The corresponding orientation map displayed in Euler colours (c) illustrates the majority of strain is localised to a single lath. *Euler colourbar is displayed in Figure 9.*

Figure 13. Normalised strain profiles at 8% global strain (ϵ_{xx}) of DIC and FEA applied to meshed X-ray CT models for specimens prepared with no overtill (a) and with overtill (b)

Figure 14. 2D FEA of tensile stress-strain curves for samples with varying taper angles.

Figure 15. (a) Peak stress and (b) Strain hardening exponent measured in the modelled 2D plane strain, modelled tomography and experimental data.