Micromechanical Response of Additively Manufactured 316L Stainless Steel Processed by High-Pressure Torsion

Shahir Mohd Yusuf, Ying Chen, Shoufeng Yang, and Nong Gao*

For the first time, the plastic deformation behavior of additively manufactured material processed by severe plastic deformation (SPD) is investigated. Herein, high-pressure torsion (HPT) is applied on 316L stainless steel (316L SS) fabricated by laser powder bed fusion (LPBF) to produce ultrafine-grained (UFG) and nanograned (NG) microstructures. Microscopy analysis reveals a final average grain size of ≈42 nm which is achieved upon torsional strain saturation after ten revolutions. In addition, microhardness mapping indicates significant hardness increase with the increasing number of HPT revolutions and saturates at ≈600 HV after ten revolutions. The evolution in plastic deformation mechanism is assessed by calculating the strain rate sensitivity (SRS), m, and activation volume, V_p^*, based on nanoindentation measurements at both constant and multiple strain rates. The estimated m values remain relatively consistent throughout all processing conditions, suggesting that superior strength can be achieved by HPT processing while maintaining reasonably high plasticity levels. Based on the calculated V_p^* values correlated with microstructure evolution, it is reasonable to infer that the submicron- and nanoscale microstructural features play an important role in plastic deformation before HPT processing, whereas plastic deformation for all HPT processing conditions is significantly influenced by grain boundary (GB)-driven plasticity.

1. Introduction

Numerous studies have reported attractive properties in ultrafine-grained (UFG) and nanograned (NG) metals and alloys compared with their coarse-grained (CG) counterparts, including significantly higher yields and tensile strengths,[1–3] enhanced wear and corrosion resistance,[4–6] and superplasticity at room temperature (RT).[7,8] It is now accepted that UFG and NG materials are defined as those having grain sizes in the range of 0.1–1 μm and <100 nm, respectively. Such remarkable properties are largely attributed to grain refinement and the generation of large amounts of dislocations in these grain scales, highlighting the benefit of fine grain microstructure.[9,10] To date, severe plastic deformation (SPD) processing has emerged as one of the most popular techniques that can produce bulk metals with UFG and NG microstructures, including equal-channel angular pressing (ECAP), high-pressure torsion (HPT), and accumulative roll bonding (ARB). From these techniques, HPT has been shown as the most effective method to produce true NG structures with large amounts of high-angle grain boundaries (GBs) via the application of concurrent compressive force and torsional straining on HPT-processed samples.[11]

In contrast, additive manufacturing (AM) has now been utilized to fabricate metallic components for niche engineering applications, e.g., automotive, biomedical, and aerospace, due to its attractive features of design flexibility, time and cost savings, and minimal material waste.[12] Selective laser melting (SLM) is one of the most widely used AM methods in the laser powder bed fusion (LPBF) category. As its name implies, SLM uses laser as the heat source to selectively consolidate layers of the powder bed into a complete 3D structure according to the initial computer-aided design (CAD) data. To date, a wide range of alloys have been used to fabricate metallic components using SLM, including 316L stainless steel (316L SS), Inconel 718 (IN 718), AlSi10Mg, and Ti6Al4V.[13,14] So far, research on metal AM has focused on optimizing processing parameters to achieve the highest densification levels with minimal porosity or defect contents.[15–28] To minimize

S. Mohd Yusuf, Dr. N. Gao
Materials Research Group
Faculty of Engineering and Physical Sciences
University of Southampton
Southampton SO17 1BJ, UK
E-mail: N.Gao@soton.ac.uk

Dr. Y. Chen
Fujian Provincial Key Laboratory of Functional Materials and Applications
Xiamen University of Technology
Xiamen 361024, P. R. China

Prof. S. Yang
Production Engineering
Machine Design and Automation Section
Department of Mechanical Engineering
Katholieke Universiteit Leuven (KU Leuven)
Leuven 3001, Belgium

© 2020 The Authors. Published by WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.

DOI: 10.1002/adem.202000052
defects and improve properties of AM components, it is normally through post-processing\cite{19,20,21,22} or tailoring the microstructure to obtain the desired mechanical properties for the intended applications\cite{23,24,25,26}.

Nevertheless, many reports have indicated that AM parts possess improved properties compared with their conventionally manufactured (CM) counterparts, e.g., better yield and tensile strengths, enhanced corrosion and wear performance, and increased fatigue life\cite{27,28,29,30,31,32}. Such improvements are attributed to the unique, fine microstructures and formation of strengthening nanoprecipitates in some cases, all of which are ascribed to the high cooling rates (10^3–10^7 K s^-1) achieved during the SLM process due to short laser–material interaction time\cite{33,34,35}. In addition, SLM-fabricated structures have been shown to attain high dislocation densities (up to \(10^{13} \text{ m}^{-2}\)) compared with their cast or wrought counterparts (\(10^{9}–10^{10} \text{ m}^{-2}\)). This is the result of the unique cellular structure network in the size of 0.2–1 \(\mu\)m for typical AM microstructures, which become additional storage dislocations\cite{36}. However, despite the improvement in mechanical properties, SLM parts still suffer from defects, e.g., porosity, which restricts them to be widely used for various engineering applications. Hot isostatic pressing (HIP) and heat treatment (HT) procedures have been applied as post-processing measures to relieve stress and minimize internal porosity and cracks; however, often they slightly reduce the yield and tensile strengths due to recrystallization that results in grain growth and decreased dislocation densities\cite{37}.

Thus, it is expected that SPD can be significantly reduce porosity and introduce UFG and NG microstructures to further improve the mechanical properties of SLM-fabricated components. In fact, it is only recently that the current authors have investigated the porosity, microhardness, corrosion behavior, and the strengthening mechanisms of SLM-fabricated and then HPT-processed 316L SS through various number of revolutions\cite{38,39}. These results have demonstrated significant porosity reduction from \(0.679\%\) before HPT processing to \(0.058\%\) after only 1/4 HPT revolution (\(91\%\) reduction). True nanometer grain sizes (average: \(42 \text{ nm}\)) were achieved upon strain saturation after ten HPT revolutions due to continuous grain refinement with increasing torsional strains. This resulted in significantly increased microhardness and enhanced corrosion performance of the HPT-processed material. The strengthening was attributed to the extreme grain refinement, generation and multiplication of dislocations, and twin boundaries (twinning), whereas the improved corrosion performance was associated with the homogeneous distribution of the NG microstructure.

To assess the practicality of UFG and NG AM 316L SS for future applications, the micromechanical response, i.e., the mechanism of plastic deformation due to HPT processing, has to be evaluated\cite{40}. 316L SS is of particular interest because it is an important engineering material that has been widely used in a broad range of applications, e.g., oil, nuclear, and biomedical industries, due to its high corrosion and oxidation resistance, and excellent weldability\cite{41,42}. However, so far no attempt was made to monitor the evolution of the plastic deformation mechanism at RT during HPT processing of AM 316L SS. Strain rate sensitivity (SSR), \(m\), and activation volume, \(V_p^*\), are important criteria that could relate to the change or transition of the microstructure from micro- to nanoscale\cite{43,44}. Both parameters can be estimated via nanoindentation, which is a simple testing procedure especially for samples with a small size\cite{45,46} or for examining local properties of bulk materials\cite{46}. Accordingly, the objective of this study is to investigate the micromechanical behavior of SLM-fabricated and then HPT-processed 316L SS at RT via nanoindentation experiments. The results are discussed by correlating the evolution of microstructure, hardness, \(m\), and \(V_p^*\) values.

### 2. Experimental Section

316L SS powder supplied by Concept Laser was used to additively manufacture a cylindrical rod using a Concept Laser M2 SLM machine. The processing parameters and conditions, scan strategy, and build orientation were detailed in the study by Mohd Yusuf et al.\cite{47} The composition of 316L SS powder used in this study as supplied by Concept Laser is shown in Table 1.

Samples for HPT were prepared by slicing thin disks with a thickness of 0.9 mm each from the SLM-fabricated cylindrical rod, which has an initial length of 200 mm and diameter of 9.8 mm, followed by grinding them down to thicknesses of 0.85 ± 0.02 mm using SiC abrasive paper (#800). HPT processing was conducted on the thin disks using a HPT facility with the setup described in the study by Mohd Yusuf et al.\cite{47} at RT under a constant pressure of 6 GPa through 1/4, 1/2, 1, 5, and 10 revolutions. The following equation was used to estimate the equivalent von mises strain, \(\varepsilon_{eq}\), imposed by HPT torsional strain\cite{47}:

\[
\varepsilon_{eq} = \frac{2\pi NR}{h\sqrt{3}}
\]

where \(N\) is the number of revolutions, \(R\) is the distance from disk center, and \(h\) is the initial disk thickness.

For optical microscopy (OM, Olympus BX-51 optical microscope) and scanning electron microscopy (JSM-6500 FE-SEM) observations, the as-received and HPT-processed disks were mechanically ground and polished to a mirror-like finish following normal microscopy sample preparation procedures and then etched using Kalling’s No. 2 reagent to reveal the microstructures. For transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) observations, samples with a diameter of 3 mm were punched out 3 mm from center of the disks following mechanical grinding down to 80 \(\mu\)m. Thin films were then extracted using a dimple grinder (Model 656) and a precision ion polishing system (PIPS, Gatan) before carrying out microstructural observations using a FEI TalosTMF200 TEM/STEM machine.

| Cr  | Ni  | Mo  | Mn  | Si  | C   | P   | S   | Fe        |
|-----|-----|-----|-----|-----|-----|-----|-----|-----------|
| 16.5–18.5 | 10.0–13.0 | 2.0–2.5 | 0–2.0 | 0–1.0 | 0–0.030 | 0–0.045 | 0–0.030 | Bal       |

Table 1. Chemical composition of 316L SS used in this study as supplied by concept laser (wt%).
method described in the study by Thorvaldsen\cite{48} was used to measure the average grain sizes based on >100 grains from 20 micrographs for each processing condition.

Vickers microhardness (HV) measurements were taken throughout the surface of the disks (x-y plane) following a rectilinear grid pattern with indents spaced 0.3 mm between each other using Future Tech FM-300 Vickers hardness tester (100 gf load, 15 s dwell time). The micromechanical response was evaluated at the center and edge (3 mm from center) of the disks using a nanoindentation facility (NanoTest Vantage System, Micromaterials Ltd., UK) with a three-sided pyramidal Berkovich indenter and a centreline-to-face angle of 65.3°.\cite{49} More than 15 indents were made at each location under a constant peak load, \( P_{\text{max}} \) = 50 mN, at various indentation strain rates, \( \dot{\epsilon}_i = h^{-1}(dh/dt) \) of 0.0125, 0.025, 0.05, and 0.1 s\(^{-1}\), corresponding to strain rates, \( \dot{\epsilon} \), of 1.25 \times 10^{-4}, 2.5 \times 10^{-4}, 5.0 \times 10^{-4}, \text{and } 1.0 \times 10^{-3} \text{s}^{-1}, \) estimated using an empirical relationship described in the studies by Choi et al. and Wang et al.\cite{50,51} Thermal drift was maintained below 0.1 nm s\(^{-1}\), and the nanoindentation results were normalized after adjusting the nanoindentation readings with the thermal drift. Throughout the analysis, the as-received disk was denoted as \( N = 0 \) revolution.

3. Results

3.1. Microhardness Distributions after HPT Processing

The HV values for as-received and HPT-processed disks were measured in a rectilinear grid pattern throughout the surface of the disks (x-y plane), and the results are shown as color-coded contour maps in Figure 1, in which the HV values are indicated in the color key to the right of the hardness maps. The as-received disk (Figure 1a) shows a homogeneous microhardness distribution (average: 220 HV) that is higher than conventional wrought or cast 316L SS but this value is typical of SLM-fabricated 316L SS.\cite{40,52,53} The microhardness increases inhomogeneously with increasing HPT revolutions from 1/4 to 1 with noticeably higher values around the peripheral region compared with the central regions of the disks (Figure 1b,c). Hardness saturation throughout the disk surface is obtained after ten revolutions, as shown in Figure 1d (average: 600 HV), similar to previous studies on HPT-processed SLM and CM 316L SS.\cite{37,54} The rapid increase in HV values after 1/4 and 1 revolution is due to the presence of appreciable work hardening at early deformation stages, whereas the low work hardening level at later deformation stages.

![Figure 1. Color-coded Vickers microhardness (HV) maps taken throughout the surface of the disks (x-y plane) for a) as-received, b) N = 1/4, c) N = 1, and d) N = 10 revolutions.](image-url)
contributes to the hardness saturation at maximum values, i.e., after ten revolutions in this study. Nevertheless, this behavior is typical of HPT-processed materials exhibiting strain hardening, as reported in various literatures.\textsuperscript{[55–57]}

3.2. Microstructures before and after HPT Processing

Figure 2 shows the microstructures of the as-received SLM-fabricated 316L SS. The OM image in Figure 2a shows a mix of coarse and fine grains with average 40 ± 30 μm growing through melt pools. Figure 2b shows fine, equiaxed submicron cellular substructure colonies with some nanospherical particles randomly distributed throughout the microstructure. Electron-dispersive X-ray spectroscopy (EDX) analysis indicates that these particles are rich with Cr, Si, and O, which can be considered as Cr-based nanosilicates. This is confirmed by Saedi et al.\textsuperscript{[40]} in their study of SLM-fabricated 316L SS. A bright-field (BF) TEM image in Figure 2c shows a dense dislocation network inside the cells and along the cell boundaries, as marked by the dashed outline.

Figure 3 shows the microstructural evolution after HPT processing. After 1/4 revolution, a dense dislocation network without a clear distinction with the submicron GBs is formed due to generation and multiplication of dislocations as a result of the HPT-imposed torsional strain (dashed oval in Figure 3a). In addition, Figure 3b shows the dark-field (DF) TEM image corresponding to Figure 3a, showing submicron grains (UFG microstructures) that begin to form after 1/4 HPT revolution. The average grain size is measured as 118 ± 25 nm. After one HPT revolution, Figure 3c shows the emergence of true nano-sized grains (NG microstructures) with an average of 68 ± 15 nm containing dislocations within the grain interior (dashed circles) and heterogeneously distributed dislocations (dashed arrows). Upon the saturation stage after ten revolutions, equiaxed nano-sized grains with clear and distinct GBs are achieved with an average of 42 ± 10 nm, as shown in Figure 3d. Dense dislocations (solid arrows) as well as nanotwins (solid circle) can also be observed within the interior of some grains.

XRD analysis results reveal that all HPT-processed disks remain a single face-centered cubic (FCC) γ-austenite, indicating the absence of any phase transformation during HPT processing despite the extreme torsional strain imposed.\textsuperscript{[38]} This suggests that the strengthening of the material after HPT could be attributed to the generation and multiplication of dislocations, twins, and grain refinement, and these microstructural features are shown in Figure 3. Further details on the microstructural evolution and strength of SLM-fabricated 316L SS before and after HPT processing are discussed in the study by Mohd Yusuf et al.\textsuperscript{[38]}

3.3. Micromechanical Properties of HPT-Processed SLM-Fabricated 316L SS

The micromechanical response for as-received and HPT-processed SLM 316L SS was evaluated using the nanoindentation technique. The following sections display results from nanoindentation measurements at a constant strain rate, \(\dot{\varepsilon}\), of \(1.25 \times 10^{-4}\) s\(^{-1}\) and at multiple strain rates, \(\dot{\varepsilon}\), of \(1.25 \times 10^{-4}\), \(2.5 \times 10^{-4}\), \(5.0 \times 10^{-4}\), and \(1.0 \times 10^{-3}\) s\(^{-1}\), respectively.

![Figure 2. a) OM image showing the mix of coarse and fine grains, b) SEM image showing cellular structure network with spherical Cr nanosilicates, and c) BF TEM image showing dislocations inside the cells and along the cell boundaries (dashed outline) in the as-received disk.](image-url)
3.3.1. Nanoindentation Results for a Constant Strain Rate

When the indenter penetrates the surface of the material during nanoindentation testing at a constant indentation rate and at a given peak load, \( P \), force and displacement, \( h \), data are recorded with loading and unloading profiles, yielding \( P-h \) curves that represent the microscale response of the tested material.\(^{[44]}\) Shorter displacements (curve moves to the left) correspond to increased hardness and reduced ductility, whereas less broadening/deviation among a set of \( P-h \) curves indicates a reasonably consistent mechanical response and better plastic stability.\(^{[39,49]}\) Therefore, Figure 4 shows a series of representative indentation curves (load \( P \) vs displacement \( h \)) for as-received and HPT-processed disks both at the center and at the edge of the disks through various revolutions at a constant strain rate \( \dot{\varepsilon} \) of \( 1.25 \times 10^{-1} \), obtained from 15 individual indentations at each location. Two observations could be made based on these curves.

First, it is clear that the displacements of HPT-processed disks become lower with increasing HPT revolutions compared with the as-received disk both at the center and at the edge, suggesting a higher hardness and reduced ductility that are most likely the result of the dense dislocations generated and grain refinement as the result of HPT-imposed torsional strain.\(^{[39]}\) Second, the displacements of the \( P-h \) curves for the HPT-processed disks show less broadening with a higher number of revolutions compared with the as-received disk, in which such effects are more apparent at the edge (Figure 4b) as compared with at the center (Figure 4a) of the disks. This indicates the improved plastic stability of HPT-processed disks, possibly due to increasingly homogeneous microstructural distributions with higher HPT revolutions.\(^{[39,49]}\)

Figure 5 shows the average \( P-h \) curves for as-received and HPT-processed disks at the center and edge locations. The shorter displacements of the \( P-h \) curves for \( N = 1/4, 1/2, \) and 1 revolution at the edge (Figure 5b) in contrast with that at the center of the disks (Figure 5a) indicate rapid increase in hardness at the peripheral area compared with the central region of the HPT-processed disks during the earlier deformation stages. In contrast, the displacements of the \( P-h \) curves become even shorter at later deformation stages (\( N = 5 \) and 10 revolutions) for both the center and edge of the disks. Nevertheless, this suggests that a significant hardness increase and lower ductility are obtained via HPT, regardless of the location of indents.

Subsequently, the nanoindentation hardness values, \( H \), are estimated using the Oliver–Pharr method,\(^{[38]}\) and the results of variation in \( H \) against \( N \) are shown in Figure 6. Figure 6a shows the comparison of the nanohardness at the center and edge of the as-received and HPT-processed disks, whereas Figure 6b,c shows the \( H \) values in comparison with HV values at the center and edge of the disks, respectively. The higher \( H \) values at the edge of the HPT-processed disks compared with that at the center (Figure 6a) are consistent with the HV measurements shown in Figure 1. In addition,
the evolution of $H$ values exhibits a similar trend with that of HV measurements (Figure 6b,c), i.e., remarkable hardness increase due to strong work hardening at the initial HPT stages (1/4–1 revolution) before only increasing marginally upon reaching saturation after ten revolutions due to the absence of significant work hardening. In addition, the consistently higher $H$ values compared with HV (Figure 6b,c) could be attributed to 1) the fact that the area of contact by nanoindentation is measured as projected area, rather than the surface area measured in traditional Vickers microhardness (HV), \[59\] and 2) the indentation size effect (ISE) that manifests as higher $H$ values when the indentation load, $P$, and indentation depth, $h$, are decreased for sharp indentations such as the pyramid indenter used in this study. \[60\]
3.3.2. Nanoindentation Results for Multiple Strain Rates

Nanoindentation measurements at various strain rates, $\dot{\varepsilon}$, of $1.25 \times 10^{-4}$, $2.5 \times 10^{-4}$, $5.0 \times 10^{-4}$, and $1.0 \times 10^{-3} \text{s}^{-1}$, taken both at the center and at the edge of the disks, reveal continuously lower displacement values with increasing $\dot{\varepsilon}$. This trend is shown by the representative $P-h$ curve in Figure 7 for as-received and HPT-processed disks through ten revolutions taken at the edge of the disks. This observation demonstrates a strain rate dependency for the displacement shift at peak load, i.e., shift to the left after HPT, indicating a decrease in ductility and increase in hardness as the strain rate increases.\[8,49,61\]

In contrast, the variations in $H$ values as a function of $N$ for various $\dot{\varepsilon}$ values taken at the centre and edge of the disks are shown in Figure 8, together with HV values as comparison. The results shown in Figure 8 display a similar trend as those observed for nanoindentation at a single strain rate shown in Figure 6. In addition, the estimated $H$ values for as-received and HPT-processed disks are all strain rate sensitive, as indicated by the consistently higher $H$ values at a higher $\dot{\varepsilon}$. However, the rate dependency decreases with increasing HPT revolutions.

Figure 6. a) $H$ versus $N$ at centre and edge, $H$ versus HV at the b) center and c) edge of the as-received and HPT-processed disks. HV values from microhardness measurements are included for comparison.

Figure 7. Representative $P-h$ curve for as-received and HPT-processed disks through ten revolutions obtained at different $\dot{\varepsilon}$ values taken at the edge of the disks.
Nevertheless, the trend of increasing $H$ with $N$ is not the same for all materials but depends on the response of a particular material to HPT straining. For example, ZK60 Mg alloy and the CoCrFeNiMn high entropy alloy (HEA) exhibit strain hardening behavior, thus an increased $H$ with an increasing $N$.\cite{43,62} However, Zn-22\% Al shows a strain softening behavior, thus a decreased $H$ at a higher $N$.\cite{42}

4. Discussion

In this study, the results from both microhardness mapping and nanoindentation testing demonstrate strain hardening characteristics for the HPT-processed disks, which is expected from the extreme grain refinement obtained with increasing HPT revolutions. The strengthening of HPT-processed materials is commonly observed in various studies and is primarily attributed to dislocation hardening and GB strengthening.\cite{9,63,64} as well as twinning and precipitation hardening in some metals.\cite{1,65,66}

In addition, the strain-rate dependency shown in nanoindentation testing can provide some information on the level of plasticity and plastic deformation mechanisms at different strain rates. Therefore, the deformation characteristics for HPT-processed disks at RT can be evaluated by calculating the SRS, $m$, at a given strain, $\epsilon$, and absolute temperature, $T$, by the relation between uniaxial flow stress, $\sigma_f$, and strain rate, $\dot{\epsilon}$, through the following expression:\cite{67,68}

$$m = \frac{\partial \ln \sigma_f}{\partial \ln \dot{\epsilon}} \bigg|_{\epsilon, T} = \frac{\partial \ln(H/C)}{\partial \ln \dot{\epsilon}} \bigg|_{\epsilon, T}$$

(2)

For nanoindentation, the Tabor empirical relationship of $\sigma_f = H/C$ is used to estimate $\sigma_f$, in which $H$ is the nanohardness estimated from the $P-h$ curves based on the Oliver–Pharr method.\cite{58} and the value of $C$ is taken as 3 for fully plastic deformation at a constant strain rate $\dot{\epsilon}$.\cite{67,69} Thus, the values of $m$ are estimated from the gradient of a double logarithmic plot of $H/3$ versus $\dot{\epsilon}$ and shown as inset in Figure 9, whereas the evolution of

Figure 8. $H$ versus $N$ for various $\dot{\epsilon}$ values at the a) center and b) edge of the disks. HV values from microhardness measurements are also included for comparison.

Figure 9. Variation of $m$ with increasing $N$ and flow stress, $H/3$ versus $\dot{\epsilon}$, shown as inset at the a) center and b) edge of the as-received and HPT-processed disks.
values for all processing conditions at the center and edge with increasing N is summarized in the main figure.

The values of m for the as-received disk are estimated as $\approx 0.041$ (center) and $\approx 0.040$ (edge), which are of one order higher than 0.0061 obtained for coarse-grained 316L SS\textsuperscript{[70]} and other typical coarse-grained FCC metals, e.g., $\approx 0.004$ for high-purity Cu\textsuperscript{[71]} $\approx 0.0028$ for pure Ni\textsuperscript{[72]} and $\approx 0.004$ for Al 99.5\textsuperscript{[73]}. However, these values are consistent with the m values for AM 316L SS as explained by Li et al.\textsuperscript{[74]} ranging from 0.02 to 0.03, when measured by tensile tests at different strain rates and strain rate jump tests. In fact, the high values of m obtained for the as-received SLM-fabricated 316L SS in this study are close to or higher than those of CM FCC metals having nanosized grains, e.g., $m = 0.015$–0.034 for pure Ni with grain sizes < 100 nm\textsuperscript{[72,75]}

The values of m only decrease marginally after the early stages of HPT, i.e., 1/4 up to 1 revolution ($\approx 0.033$ and $\approx 0.031$ at the center and edge, respectively) before remaining relatively constant at the later stages of HPT, i.e., 5 and 10 revolutions. Such high m values indicate reasonably high plasticity levels despite the significant hardening achieved through HPT and they are comparable with other FCC metals and alloys with UFG or nanosized grain microstructures.\textsuperscript{[44,62]} In this context, plasticity is referred to as the ability of a solid material to be plastically deformed without fracture, which is dependent on its intrinsic crystal structure and available slip systems, and the microstructural features attained after processing.\textsuperscript{[76]} This is unlike the ductility of materials that is influenced by the strain hardening rate, which is significantly decreased in nanostructured metals because the nanoscale GBs could no longer become effective sites for the accumulation of dislocations beyond a critical value.\textsuperscript{[77]}

It is interesting to see that the mix of fine and coarse grains of as-received SLM 316L SS in this study already possesses m values comparable with that of the HPT-processed counterparts having grain sizes in the UFG and nanoscale regimes. Thus, there could be a change in the plastic deformation mechanism as a result of microstructural transition from the micro- to nanoscale due to HPT torsional straining. Therefore, the strain rate-dependent plastic deformation mechanism can be assessed by estimating the activation volume, $V_p^*$, based on the following relationship\textsuperscript{[50,70]}

$$V_p^* = \sqrt{3}kT\frac{\partial \ln \sigma_f}{\partial \ln f} = \sqrt{3}kT\frac{\partial \ln (0.01\varepsilon_i)}{\partial (H/C)}$$

$$V_p^* = \frac{C\sqrt{3}kT}{mH}$$

(3)

where $k$ is Boltzmann’s constant. The results are shown in Figure 10 for the center and edges of the disk and expressed in terms of the Burgers vector $b$ (taken as 2.5 $\times$ 10$^{-10}$ m for austenitic stainless steels\textsuperscript{[78]}).

Regardless of the location of the disks, a general trend could be observed from Figure 10 that the values of $V_p^*$ for the as-received SLM-fabricated 316L SS are in the region of $\approx 11b^3$, which then decreases to the range $\approx 0$–8$b^3$ with increased number of revolutions. It has been reported that the value of $V_p^*$ depends on the orders of magnitude for different strain rate-limiting processes,\textsuperscript{[79]} e.g., $\approx 100b^3$–1000$b^3$ for dislocation glide in FCC metals\textsuperscript{[80]} $\approx 10b^3$ for grain boundary sliding (GBS)\textsuperscript{[79]} and $\approx b^3$–10$b^3$ for diffusion through the GBs or through the crystalline lattice.\textsuperscript{[80]}

The estimated $V_p^*$ value for the as-received disk in this study is $\approx 11b^3$, which is significantly lower than that observed in conventional coarse-grained FCC materials that are typically deformed via the forest dislocation cutting mechanism ($>100b^3$)\textsuperscript{[80,81]} However, it is closer to the value of 22–28$b^3$ reported by Li et al. for AM 316L SS.\textsuperscript{[74]} This discrepancy can be attributed to the difference in the testing method used to determine m, with tensile and strain rate jump tests used in the study by Li et al.\textsuperscript{[74]} and nanoindentation in this study. Nevertheless, the rather low $V_p^*$ value in AM 316L SS compared with CM coarse-grained 316L SS and other FCC metals suggests the abundance of barriers to dislocation for deformation via plastic flow, including a mix of high-angle and low-angle GBs, pre-existing dislocations, nanoprecipitates, cellular structure networks, numerous fusion boundaries from solidified melt pools, and localized misorientations.\textsuperscript{[75,82]} In fact, the dislocation density in AM 316L SS is already much higher ($>10^{13}$ m$^{-2}$) compared with their cast or wrought counterparts ($10^{10}$–$10^{11}$ m$^{-2}$), in which the walls of the cellular structure network become additional storage for dislocations.\textsuperscript{[16,36,41]}

Therefore, although the value of $V_p^*$ for the as-received disk corresponds to that of GB-driven plasticity, specifically GBS and diffusion through the GBs or the crystalline lattice, the plastic deformation of SLMM-fabricated 316L SS in this study cannot be associated with GB phenomena but rather influenced by numerous submicron and nanoscale microstructural features as mentioned previously.

During the early and the intermediate stages of HPT deformation, e.g., from 1/4 to 1 revolution, the activation volume decreases to $\approx$6–8$b^3$, which remains reasonably consistent toward the later stages (5 and 10 revolutions). Kawasaki et al.\textsuperscript{[44]} explained that although significant hardening is observed at early stages of HPT deformation due to the generation and
multiplication of dislocations, the overall strain hardening rate of HPT-processed metals decreases with increasing hardness and number of revolutions. This suggests that the mechanism for plastic deformation changes as the microstructures transition from micro- to nanoscale upon continuous torsional straining. This is evident in this study as the HV values after 1/4 revolution generally double that of the as-received samples, but only marginally increase after 1 and 10 revolutions, as shown in Figure 1. This is because when the grain sizes continue to decrease to the submicron and then nanoscales, the amount of GBs also increases, and it becomes increasingly difficult for the dislocations to pile up and be stored within the grain interior. At this stage, GBs become the most effective sites in blocking dislocation motions compared with the accumulated dislocations. In the current investigation, TEM observations shown in Figure 3b,c indeed display a significant amount of dislocations and the emergence of submicron grains (UFG microstructures) after 1/4 revolution without a clear distinction between the dislocations and GBs. This implies that the predominant deformation mechanism at the early stages of HPT is related to the combined dislocations due to both the geometrically necessary dislocations (GNDs) and statistically stored dislocations (SSDs) and the UFG microstructures formed as a result of strain gradient during HPT processing. At the intermediate stage, i.e., after one revolution, the microstructures consist of nanosized grains that begin to emerge with apparent boundaries, and the dislocations now appear to be incoherent and no longer continuous, as shown in Figure 3c. This indicates that the GB-related activities, e.g., GBS, or diffusion through GBs or through the crystalline lattice, become the controlling mechanism for plastic deformation at this stage, similar to that observed in nanotwinned nanocrystalline CoCrFeNi alloys studied by Huo et al. After ten revolutions, nanosized grains with clear and distinct GBs dominate the microstructure, with some nanotwins observed within the grain interior at the saturation stage, as shown in Figure 3d. Here, the majority of the grain interior does not contain dislocations, as they are emitted from inside the grains toward the opposing sides of the GBs, but the nanotwins have been found to act as additional GBs that also impede dislocation motions. This suggests that GB-mediated plasticity remains the prevalent deformation mechanism at the HPT saturation stage, either through GB diffusion or crystalline lattice diffusion, similar to the results from other studies on HPT-processed metals and alloys.

Therefore, the as-received SLM-fabricated 316L SS in this study can be considered to possess reasonably high plasticity characteristics, i.e., superior capability to sustain plastic deformation without fracture as indicated by the higher m value (≈0.041) compared with its CM coarse-grain counterparts (e.g., ≈0.0061). Even though the values of m slightly decrease at the early stages of HPT, these values remain consistent with increasing number of revolutions, indicating that a good level of plasticity is maintained along with the significant hardening obtained via HPT processing. Furthermore, based on the evolution of $V_p^*$ values and the microstructural observations in this study, it can be concluded that the predominant deformation mechanism in the as-received AM 316L SS is attributed to the numerous submicron- and nanoscale microstructural features, rather than GB-related activities, although the low value of $V_p^*$ is often related to GB-regulated plasticity. In contrast, the dominant deformation mechanism for all HPT-processed disks is ascribed to GB-mediated plasticity based on the consistently lower values of $V_p^*$ for all HPT processing conditions than the $V_p^*$ of the as-received counterpart, as well as the increasing amount of submicron- and nanoscale GBs with a higher number of HPT revolutions.

5. Conclusions

The microstructure, hardness, and evolution of plastic deformation mechanisms of SLM-fabricated and then HPT-processed 316L SS were studied through OM, SEM, TEM, microhardness mapping, and nanoindentation testing at both constant and various strain rates. The values of SRS, m, and activation volume, $V_p^*$, were calculated based on the parameters obtained from nanoindentation testing. These values were then correlated with the microstructure and microhardness evolution to assess the prevalent mechanisms of plastic deformation for the as-received and HPT-processed samples. The following conclusions can be drawn based on the results of this study: 1) Microhardness mapping demonstrates that the hardness increase did not occur homogeneously throughout the HPT-processed disks up to one revolution, but hardness saturation is eventually achieved after ten revolutions, as indicated by the relatively homogeneous hardness distribution throughout the disk. 2) TEM observations reveal the multiplication of dislocations and the emergence of submicron grains after 1/4 HPT revolution, followed by the emergence of nanosized grains with heterogeneously distributed dislocations after one revolution, and finally achieving nanosized grains with clear and distinct GBs but dislocation free within the majority of the grain interior after ten revolutions (average grain size: 42 ± 10 nm). 3) The estimated m values remain relatively consistent throughout all processing conditions, indicating that a reasonably high level of plasticity is maintained in AM 316L SS compared with the CM counterpart. 4) Based on the estimated $V_p^*$ values and microscopy analysis, the prevalent deformation mechanism changes from plasticity driven by numerous submicron and nanoscale microstructural features for the as-received disk to GB-mediated plasticity after HPT processing.

Acknowledgements

This work was supported by a PhD studentship for S.M.Y. from Faculty of Engineering and Physical Sciences at the University of Southampton, UK. Y.C. acknowledges financial support from the National Natural Science Foundation of Fujian Province, China (grant no. 51601162) and High-Level Talent Funding for Xiamen Oversea Returnee.

Conflict of Interest

The authors declare no conflict of interest.

Keywords

activation volumes, high-pressure torsions, laser powder bed fusion, nanoindentations, strain rate sensitivities
[66] Y. Chen, N. Gao, G. Sha, S. P. Ringer, M. J. Starink, Acta Mater. 2016, 109, 202.
[67] I. C. Choi, Y. J. Kim, Y. M. Wang, U. Ramamurty, J. Il Jang, Acta Mater. 2013, 61, 7313.
[68] M. J. Mayo, W. D. Nix, Acta Metall. 1988, 36, 2183.
[69] S. Shim, J. Il Jang, G. M. Pharr, Acta Mater. 2008, 56, 3824.
[70] F. Khodabakhshi, M. H. Farshidianfar, A. P. Gerlich, M. Nosko, V. Trembošťová, A. Khajepour, Mater. Sci. Eng. A 2019, 756, 545.
[71] R. P. Carreker, W. R. Hibbard, Acta Metall. 1953, 1, 654.
[72] R. Schwaiger, B. Moser, M. Dao, N. Chollacoop, S. Suresh, Acta Mater. 2003, 51, 5159.
[73] J. May, H. W. Höppel, M. Göken, Mater. Sci. Forum 2006, 503–504, 781.
[74] Z. Li, T. Voisin, J. T. McKeown, J. Ye, T. Braun, C. Kamath, W. E. King, Y. M. Wang, Int. J. Plast. 2019, 120, 395.
[75] Y. M. Wang, A. V. Hamza, E. Ma, Acta Mater. 2006, 54, 2715.
[76] Y. T. Zhu, X. L. Wu, Mater. Today Nano. 2018, 2, 15.
[77] Y. T. Zhu, X. Z. Liao, X. L. Wu, Prog. Mater. Sci. 2012, 57, 1.
[78] W. Huo, F. Fang, X. Liu, S. Tan, Z. Xie, J. Jiang, Appl. Phys. Lett. 2019, 114, 101904-1.
[79] H. Conrad, Nanotechnology 2007, 18, 1.
[80] H. Conrad, Mater. Sci. Eng. A 2003, 341, 216.
[81] T. Zhu, J. Li, A. Samanta, H. G. Kim, S. Suresh, Proc. Natl. Acad. Sci. 2007, 104, 3031.
[82] T. Voisin, N. P. Calta, S. A. Khairallah, J. B. Forien, L. Balogh, R. W. Cunningham, A. D. Rollett, Y. M. Wang, Mater. Des. 2018, 158, 113.
[83] V. Yamakov, D. Wolf, S. R. Phillpot, A. K. Mukherjee, H. Gleiter, Nat. Mater. 2002, 1, 45.
[84] H. Van Swygenhoven, Science 2002, 296, 66.
[85] D. Wolf, V. Yamakov, S. R. Phillpot, A. Mukherjee, H. Gleiter, Acta Mater. 2005, 53, 1.
[86] J. Schiøtz, Scr. Mater. 2004, 51, 837.
[87] A. G. Frøseth, P. M. Derlet, H. Van Swygenhoven, Appl. Phys. Lett. 2004, 85, 5863.