Influence of high-pressure torsion on the microstructure and microhardness of additively manufactured 316L stainless steel

Shahir Mohd Yusufa, Ying Chenb, and Nong Gaoc\*

a Engineering Materials and Structures (eMAST) iKohza, Malaysia-Japan International Institute of Technology (MJIIT), UTM Kuala Lumpur, Jalan Sultan Yahya Petra, 54100 Kuala Lumpur, Malaysia; [shahiryasin@utm.my](mailto:shahiryasin@utm.my) (SMY)

b Fujian Provincial Key Laboratory of Functional Materials and Applications, Xiamen University of Technology, Xiamen, 361024, PR China; [cyj829@163.com](mailto:cyj829@163.com) (YC)

c Materials Research Group, Faculty of Engineering and Physical Sciences, University of Southampton, Southampton SO17 1BJ, UK; [n.gao@soton.ac.uk](mailto:n.gao@soton.ac.uk) (NG)

**\*** Correspondence: N.Gao@soton.ac.uk; Tel.: (+44) 023-8059-3396 (N.G.)

Received: date; Accepted: date; Published: date

**Abstract:** High-pressure torsion (HPT) is known as an effective severe plastic deformation (SPD) technique to produce bulk ultrafine-grained (UFG) metals and alloys by the application of combined compressive force and torsional shear strains on thin disk samples. In this study, the microstructures and microhardness evolution of an additively manufactured (AM) 316L stainless steel (316L SS) processed through 5 HPT revolutions are evaluated at the central region of the disks, where the effective shear strains are relatively low compared to the peripheral regions. Scanning electron microscopy (SEM) analysis shows that the cellular network sub-structures in AM 316L SS are destroyed after 5 HPT revolutions. Transmission electron microscopy (TEM) observations reveal non-equilbrium ultrafine grained (UFG) microstructures with average grain size of ~ 115 nm after 5 revolutions. Furthermore, energy dispersive x-ray spectroscopy (EDX) analysis suggests that spherical Cr-based nano-silicates are also found in the as-received condition, which are retained even after HPT processing. Vickers microhardness (HV) measurements indicate significant increase in average hardness values from ~ 220 HV before HPT processing to ~ 560 HV after 5 revolutions. Quantitative x-ray diffraction (XRD) patterns exhibit a considerable increase in dislocation density from ~ 0.7 x 1013 m-2 to ~ 1.04 x 1015 m-2. The super-high average hardness increment after 5 HPT revolutions is predicted to be attributed to the UFG grain refinement, significant increase in dislocation densities and the presence of the Cr-based nano-silicates, according to the model established based on the linear additive theory.

**Keywords:** high-pressure torsion; laser powder bed fusion; severe plastic deformation; additive manufacturing; microstructure; microhardness

1. Introduction

High-pressure torsion (HPT) is a well-known severe plastic deformation (SPD) technique that imposes extreme torsional strains on thin disk metallic materials that typically result in significant grain refinement down to the ultrafine regime (< 1 µm) with dense dislocation networks and other nano-scale microstructural features (in some materials) [1,2]. However, the radial dependency of the torsional strain often results in heterogenous microstructures and inhomogeneous hardness distribution across the radius of the disk [3]. Nevertheless, compared to other SPD techniques such as equal channel angular pressing (ECAP) and accumulative roll bonding (ARB), HPT is the most efficient approach to produce significant grain refinement with large proportions of high angle grain boundaries (HAGBs) throughout the bulk material [4–6].

In particular, 316L stainless steel (316L SS) is a popular alloy that are widely used in marine, petrochemical, nuclear power, oil and gas, food and beverage, and biomedical industries owing to its excellent corrosion resistance, low neutron radiation absorption rate, and good ductility [7,8]. However, this alloy could not be strengthened by heat treatment procedures due to its very low carbon content, thus the only suitable mean to strengthen 316L SS is by reducing the grain size, i.e. grain refinement route (Hall-Petch mechanism) through cold working. Since there is a limit to grain sizes that can be attained through cold working, thus 316L SS is a suitable alloy to be strengthened by HPT processing. In fact, numerous studies have been conducted on the mechanical (hardness, strength, and ductility) and functional (thermal stability, corrosion, and tribological) properties of wrought and cast 316L SS processed by HPT, demonstrating mostly favorable results after HPT processing [9–19].

On the other hand, HPT processing has also been conducted on additively manufactured 316L SS recently by Mohd Yusuf et al. [20–23]. They have reported significant improvements in hardness, corrosion, and tribological performances after HPT processing compared to the wrought counterpart due to the nano-scale grain refinement (42 nm), nano-scale twins, combined with dense dislocation networks originating from the initial cellular sub-structures and from the HPT-induced torsional straining, and high strain rate sensitivity (SRS). Although it is known that additive manufacturing is more focused on fabricating materials with high design complexity due to the layer-wise build philosophy, another important aspect of this technology is the capability to tailor the microstructures according to the required end applications by adjusting material composition and/or processing parameters. Therefore, AM-fabricated materials, including 316L SS often produce novel microstructures that results in mechanical and functional properties that are on par or even better than the wrought/cast counterparts [8,24–30]. Thus, HPT processing is a viable technique to further enhance the mechanical properties of AM 316L SS [20-23]

Until now, the investigation on microstructures and mechanical properties of HPT-processed wrought/cast/AM 316L SS have always been focused on the analysis at the peripheral regions of the disk (> 2.5 mm away from the centre), at which the HPT-induced torsional strain values are significantly higher than at the central area (0 – 2.5 mm from the centre). However, there has been almost no investigation that has been conducted at the central area where the effective shear strains are very low. Therefore, the main aim of this study is to investigate the microstructural and microhardness evolution of AM 316L SS processed through 5 HPT revolutions at the central area of the disk via various microscopy techniques and Vickers microhardness (HV) measurements, respectively. Subsequently, a model based on the linear additive theory is used to evaluate the individual mechanisms that contribute to the overall hardness after 5 HPT revolutions.

2. Materials and Methods

A cylindrical rod of 316L SS alloy with a length of 200 mm and diameter 10 mm was firstly additively manufactured by laser powder bed fusion (L-PBF) using a Concept Laser M2 LaserCUSING machine (Concept Laser GmbH, Lichtenfels, Germany) with the processing parameters detailed in Ref. [21].

The samples for HPT were prepared by machining the rod to reduce the diameter to 9.8 mm, using wire electrical discharge machining (EDM) to slice the rod into thin disks of ~ 1 mm thick, and further grinding the surface of the disks down to ~ 0.85 mm thick using 800 grits SiC paper. The disks were then subjected to 5 revolutions of HPT processing in a quasi-constrained condition under 6 GPa of pressure and at a speed of 1 rpm.

After HPT-processing, the disks were further prepared for microstructural characterizations via scanning electron microscopy (SEM), electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) observations. For SEM observation, , some disks were ground and polished to mirror-like surface finish, followed by etching in Kalling’s No. 2 reagent based on the procedures explained in Ref. [20]. For EBSD using HKL Nordlys F++ camera and Aztec HKL software (Oxford Instrument, High Wycombe, UK), some disks were electropolished in 80% methanol and 2 perchloric acid at 16 V and 0.5 A for 18 s, and images were exracted within 100 × 100 µm areas and step size of 0.1 µm that covers > 100 grains each. SEM and EBSD images were taken at the central area of the disks, 0 – 2.5 mm from the centre. For TEM using FEI TalosTMF200S machine equipped with energy dispersive x-ray spectroscopy, EDX (FEI, Brno-Cernovice, Czech Republic), some disks were mechanically ground to thickness of 80 µm before punching smaller disks of 3 mm in diameter at the central area of the disks (0 – 2.5 mm from the centre). They are then dimpled using a model 656 dimple grinder and polished into thin foils using a model 695 Gatan PIPS II precision ion polishing system (Gatan Inc., California, USA). The intercept method [43] is used to measure the average grain size from over 300 grains in 20 TEM images.

The phase composition before and after HPT processing was determined via X-ray diffraction (XRD) measurements using Rigaku SmartLab X-ray Diffractometer (Rigaku, Tokyo, Japan) [21]. The resulting XRD peaks and peak broadening data were further analysed to evaluate the microstrain and crystallite size by using the Materials Analysis Using Diffraction (MAUD) software (MAUD, version 1.999, L. Lutterotti, Italy) based on the Rietveld refinement method [34–36]. In addition, Vickers microhardness (HV) measurements were taken throughout the surface of the disk in a rectilinear grid pattern using a Future Tech FM-300 microhardness tester (Future-Tech Corp, Kawasaki, Japan) with a load of 100 gf and a dwell time of 15 s, in which the indents are spaced at 0.3 mm apart from each other.

3. Results and Discussion

3.1. Microstructural analysis

The EBSD grain orientation map of the as-received L-PBF AM-fabricated 316L SS in Figure 1(a) shows overlapping melt pool squares formed due to the ‘island’ scan strategy and the angles of misorientation of the grain growth. About 19% of the grains are considered as high angle grain boundaries (HAGBs) with misorientation angles of > 5%. These HAGBs consist of coarse (~ 40 – 70 µm) and fine grains (~ 10 – 40 µm), together with very fine equiaxed grains (~ 1 – 10 µm) observed at the end/intersection between the melt pools. On the other hand, about 81% of the grains possess misorientation angles < 15% and considered as low angle grain boundaries (LAGBs). They can be observed to be contained within the interior of the HAGBs grains. Such LAGBs in AM-fabricated metals and alloys are ascribed to cellular (equiaxed or columnar) sub-structure networks that are formed due to the high cooling rate of the AM process [37]. An example of cellular sub-structure networks with numerous equiaxed cells (~ 1 µm) is shown by the SEM image in Figure 1(b). Interestingly, Figure 1(b) also reveals spherical nano-sized particles between 20 – 100 nm at the cell boundaries and within the cell interior with an average volume fraction of 0.46 vol.%. EDX point scan analysis on the spherical particles shows an average of 31.78 wt.% Cr, 1.50 wt.% Si, and 66.72 wt.% O. Thus, it can be inferred that these particles could be Cr-based nano-silicates, which is an oxide phase that has also been found in various studies of 316L SS fabricated by L-PBF AM [8,24,25,38,39].

|  |
| --- |
|  |

**Figure 1.** As-received sample: (a) EBSD grain boundary misorientation map and (b) SEM image showing cellular sub-structure network and random dispersions of spherical nano-sized particles.

Figure 2(a) shows the SEM image at the central area of the disk after 5 HPT revolutions. It is clear that the initially coherent cellular sub-structure networks are now destroyed during HPT processing. The Cr-based nano-silicate particles disappear, which implies that they are either annihilated, or possibly displaced during HPT processing. On the other hand, Figure 2(b) and (c), respectively show the bright field (BF) and dark field (DF) TEM images of the HPT-processed disk at a similar location. These TEM images show grain refinement down to the ultrafine regime (< 1 µm) with high dislocation densities and sub-grain boundaries within the grain interior (dashed red circles), which are the typical characteristics of non-equillibrium grain boundaries (GBs) obtained through SPD processing [40–42]. The average grain size is measured as 115 ± 16 nm at the central area of the disk after 5 HPT revolutions, which is in contrast with 48 ± 11 nm when measured at the peripheral disk regions (> 3 mm from its centre) based on the previous research conducted by the current authors [21].

|  |
| --- |
|  |

**Figure 2.** Central area of the disk after 5 HPT revolutions: (a) SEM image showing the annihilation of cellular sub-structure network, (b) BF-TEM image and (c) DF-TEM image revealing non-equilbrium UFG microstructures with dense dislocation densities within the grain interior.

Interestingly, further TEM observations at the central disk area reveal the presence of spherical nano-sized particles after 5 HPT revolutions, with a volume fraction of 0.43 vol.% and average diameter of 46 ± 12 nm (e.g. dashed arrows in Figure 3a), which is suspected to be the Cr-based nano-silicates observed in the as-received disk previously. EDX area scan analysis is conducted on the area shown in Figure 3(a) and the elemental mapping results are shown in Figures 3(b) – (e). The EDX maps clearly indicate dispersions of Cr and O throughout the nano-sized particles, with Si being concentrated at the edges, i.e. on the outer surface of those particles. No Ni is present, while only relatively small dispersions of Fe can be observed on the particles. Hence, it can be inferred that the nano-sized particles are indeed the Cr-based nano-silicates, which are not destroyed but only displaced after experiencing torsional straining through 5 HPT revolutions.

|  |
| --- |
|  |

**Figure 3.** Central area of the disk after 5 HPT revolution: (a) BF-TEM image showing examples of spherical nano-sized particles and (b) – (e) corresponding EDX mapping of (a).

3.2. XRD analysis

Figure 4 shows the XRD spectra at the centre of the disk for the as-received condition and after 5 HPT revolutions. No phase change occurs and the L-PBF AM-fabricated 316L SS retains its single austenitic phase even after HPT processing. However, the presence of microstrains, *ε* and numerous coherently scattered domains (CSD), *Dc* due to the torsional straining cause broadening of the XRD peaks. These quantities are used to calculate the dislocation density, ρ using the following equation [44]:

Eqn. 1

where *b* is the burgers vector (*b* = 0.25 nm for austenitic stainless steels [45]) . The values of *ε* and *Dc* are obtained through the Rietveld refinement method applied in MAUD software using the procedures explained in Refs. [34–36]. After 5 HPT revolutions, the values of *ε* and *Dc* are measured 1.72 ± 0.07 × 10-3 as and 23 ± 2 nm, respectively, while the value of ρ is calculated as 1.04 ± 0.05 × 1015 m-2. This is a significant increase compared to 0.7 x 1013 m-2 for the as-received condition as determined by the current authors in their previous study [21].

|  |
| --- |
|  |

**Figure 4.** XRD spectra of the disk at the as-received condition and after 5 HPT revolutions.

3.2. Microhardness

Figure 5 illustrates the Vickers microhardness (HV) mapping throughout the surface of the disk after 5 HPT revolutions. The hardness distribution ranges from ~ 500 – 600 HV, in which the HV values increase with increasing distances towards the edge of the disk. Hardness saturation, i.e. homogeneous hardness distribution throughout the disk is not achieved at this stage, similar to the results when measuring HV values across the diameter of the disk [21].

|  |
| --- |
|  |

**Figure 5.** Examples of particle circularity for the metal powders used in this study. Representative images of (a) and (b) are taken from 316L SS, while that of (c) – (e) are taken from AlSi10Mg.

4. Discussion

The equivalent von Mises strain, εeq. in HPT-processed materials can be described by the well-known equation [4]:

Eqn. 2

where *r* is the distance from the centre of the disk, *N* is the number of HPT revolutions, and *h* is the thickness of the disk. Therefore, a radial dependency in the torsional strain values imposed throughout the disk during HPT processing is expected, in which the peripheral regions (> 2.5 mm from the centre) experience higher strain hardening levels compared to the central areas (0 – 2.5 mm from the centre). Hence, there will be inhomogeneous distributions of grain sizes, dislocation densities, and microhardness values, particularly at the early stages of HPT processing, e.g. 1/4, 1/2, and 1 HPT revolutions [46–51]. Such inhomogeneity is often attributed to the formation of geometrically necessary dislocations (GNDs) at the central area of the disk due to the presence of strain gradients as a result of the radial torsional shear straining nature of the HPT process [52,53]. However, if the deformation due to HPT-imposed torsional strains exceeds certain von Mises strain values, the strain hardening level will reach a saturation stage (typically after 5 to 10 HPT revolutions), whereby the grain refinement, dislocation density generation and multiplication, and microhardness attained will be fairly homogeneous throughout the disk [3,54–59]. Moreover, there are also reports showing that such homogeneity is not achieved even after 10 HPT revolutions, which is typically attributed to the intrinsic material properties that suppresses further grain refinement or multiplication of dislocation densities, e.g. stacking fault energy (SFE) in stainless steel alloys [9,13,60,61].

In this study, although the εeq. at the centre is theoretically 0 since *r* = 0, the average HV value at the central area of the disk is recorded as ~ 564 HV after HPT revolutions, which is already significantly higher than that of the as-received condition (~ 220 HV). This is because in reality, strain localization takes place due to the low strain rate sensitivity (SRS) coefficient of most HPT-processed materials, thereby some level of torsional strains are still imposed at the central area [62]. Hence, the HPT-imposed torsional straining results in severe plastic deformation that causes significant grain refinement down to the ultrafine regime (< 1 µm) and generation and multiplication of dislocations. Indeed, Figures 2(b) and (c) show exactly the consequences of such deformation; ultrafine grains with average grain size of as 96 ± 24 nm with dense dislocations within the grain interior, i.e. non-equilibrium GBs are attained at the central disk area after 5 HPT revolutions. Furthermore, the analysis of the XRD spectra using MAUD software reveals that the dislocation density of 1.21 ± 0.05 × 1015 m-2 that is significantly higher than 0.7 x 1013 m-2 before HPT processing. This confirms the SRS coefficient theory explained in Ref. [62] that torsional strains are still imposed and dislocations are still generated and multiplied at the central disk area even though the theoretical εeq. value is 0 at *r* = 0. The numerous ultrafine GBs and dense dislocation networks are known to be sites that impede dislocation motions, thereby contributing to the remarkable increase of HV values at the central disk area after 5 HPT revolutions. In addition, the spherical Cr-based nano-silicates have also been found to strengthen L-PBF AM-fabricated 316L SS by Orowan bypassing mechanism [25,39,63,64].

Therefore, a strengthening model based on the linear additive theory is used to evaluate the contribution of each mechanism on the hardness increase at the disk centre after 5 HPT revolutions. The increase in hardness due to dislocations is described by the following equation [65,66]:

Eqn. 3

where *C* is the dimensionless constant that describes the yield strength-hardness relationship, taken as 3 for FCC materials [67,68], *M* is the Taylor orientation factor, taken as 3.05 for FCC materials [69], α1 is an empirical constant with the value 0.3 [70], *G* is the shear modulus of the material (*G* = 77,000 MPa for austenitic SS [69]), and ρTotal is the value of dislocation density, determined as 1.04 ± 0.05 × 1015 m-2 via XRD analysis through Eqn. 2. Thus, the hardness increase due to generation and multiplication of dislocations is calculated as ~ 174 HV. The contribution of HPT-induced grain refinement can be estimated by the Hall-Petch equation [71]:

Eqn. 4

where *K*HP is the material-dependent Hall-Petch constant, taken as 0.3 MPa\*m-1/2 [69], and *d* is the grain size. In this study, the average grain size at the central disk area after 5 HPT revolutions is determined as 115 ± 16 nm through the line intercept method. Therefore, the increase in hardness due to the ultrafine grain size is calculated as ~ 271 ± 8 HV. The contribution of Cr-based nano-silicates through the Orowan mechanism can be described by [64]:

Eqn. 5

where *λ* is the average spacing of the Cr-based nano-silicates, determined as 152 ± 24 nm from TEM observations of 20 TEM images at the central disk area after 5 HPT revolutions. Hence, the presence of the Cr-based nano-silicates provide additional ~ 118 ± 12 HV towards the overall hardness after HPT processing.

The predicted hardness based on the model used in this study is 563 ± 10 HV for the central disk area after 5 HPT revolutions, which is within the error margin and correlates well with the average hardness measured at the central disk area (0 – 2.5 mm from the centre) of 560 HV. Based on the estimated individual hardening mechanisms, grain boundary hardening provides the highest contribution (~ 48%) to the overall hardness at the centre of the disk, followed by dislocation hardening (~ 31%), and Orowan strengthening (~ 21%).

5. Conclusion

In this study, a 316L SS alloy was initially fabricated by L-PBF AM technique, followed by HPT processing through 5 revolutions. The microstructural evolution, phase composition and dislocation density, and hardness at the central disk area (0 – 2.5 mm from the centre) were assessed by microscopy technique (SEM, EBSD, and TEM), XRD analysis, and Vickers microhardness (HV) mapping approaches, respectively. A model based on the linear additive theory was used to predict the contribution of different mechanisms on the overall hardness increase at the central area of the HPT-processed disk. The following conclusions can be drawn based on the results of this study:

1. SEM, EBSD, and EDX analysis reveal that the as-received L-PBF AM-fabricated 316L SS contains unique microstructures comprising of square melt pools with LAGBs and HAGBs, cellular-sub-structure networks, and spherical Cr-based nano-silicates.
2. Microscopy observations show that the cellular sub-structure networks in the as-received disk are destroyed after 5 HPT revolutions, while the Cr-based nano-silicates are not annihilated, just displaced due to the HPT-imposed torsional strain.
3. HPT processing through 5 revolutions successfully produces ultrafine grain sizes with an average of 115 ± 16 nm, accompanied by a significant increase in dislocation density.
4. The model based on linear additive theory estimated that the hardness increase at the central disk area after 5 HPT revolutions are contributed by grain boundary hardening (~ 48%), dislocation hardening (~ 31%) and Orowan strengthening (~ 21%).

**Author Contributions:** SMY contributes primarily on conducting the experiments, data collection, data analysis and writing of the manuscript; YC conducted the TEM experiments and subsequent data analysis; NG is heavily involved in the project conceptualization, administration, and planning, as well as review and editing the manuscript.

**Funding:** SMY thanks the Faculty of Engineering and Physical Sciences, University of Southampton for their financial support. The TEM experiments were carried out by YC with financial aids from the National Science Foundation of Fujian Province, China (No. 51601162) and High-Level Talent Funding for Xiamen Oversea Returnee.

**Conflicts of Interest:** The authors declare no conflict of interest.

References

[1] A.P. Zhilyaev, T.G. Langdon, Using high-pressure torsion for metal processing: Fundamentals and applications, Prog. Mater. Sci. 53 (2008) 893–979. https://doi.org/10.1016/j.pmatsci.2008.03.002.

[2] R.Z. Valiev, A.P. Zhilyaev, T.G. Langdon, Bulk Nanostructured Materials: Fundamentals and Applications, John Wiley & Sons, Inc., Hoboken, 2014.

[3] A.P. Zhilyaev, S. Lee, G. V. Nurislamova, R.Z. Valiev, T.G. Langdon, Microhardness and microstructural evolution in pure nickel during high-pressure torsion, Scr. Mater. 44 (2001) 2753–2758. https://doi.org/10.1016/S1359-6462(01)00955-1.

[4] K. Edalati, Z. Horita, A review on high-pressure torsion (HPT) from 1935 to 1988, Mater. Sci. Eng. A. 652 (2016) 325–352. https://doi.org/10.1016/j.msea.2015.11.074.

[5] J. Wongsa-Ngam, M. Kawasaki, T.G. Langdon, A comparison of microstructures and mechanical properties in a Cu-Zr alloy processed using different SPD techniques, J. Mater. Sci. 48 (2013) 4653–4660. https://doi.org/10.1007/s10853-012-7072-0.

[6] R.Z. Valiev, I. Sabirov, A.P. Zhilyaev, T.G. Langdon, Bulk nanostructured metals for innovative applications, Jom. 64 (2012) 1134–1142. https://doi.org/10.1007/s11837-012-0427-9.

[7] B. Al-Mangour, R. Mongrain, E. Irissou, S. Yue, Improving the strength and corrosion resistance of 316L stainless steel for biomedical applications using cold spray, Surf. Coatings Technol. 216 (2013) 297–307. https://doi.org/10.1016/j.surfcoat.2012.11.061.

[8] Y. Zhong, L. Liu, S. Wikman, D. Cui, Z. Shen, Intragranular cellular segregation network structure strengthening 316L stainless steel prepared by selective laser melting, J. Nucl. Mater. 470 (2016) 170–178. https://doi.org/10.1016/j.jnucmat.2015.12.034.

[9] J. Gubicza, M. El-Tahawy, Y. Huang, H. Choi, H. Choe, J.L. Lábár, T.G. Langdon, Microstructure, phase composition and hardness evolution in 316L stainless steel processed by high-pressure torsion, Mater. Sci. Eng. A. 657 (2016) 215–223. https://doi.org/10.1016/j.msea.2016.01.057.

[10] M. El-Tahawy, Y. Huang, H. Choi, H. Choe, J.L. Lábár, T.G. Langdon, J. Gubicza, High temperature thermal stability of nanocrystalline 316L stainless steel processed by high-pressure torsion, Mater. Sci. Eng. A. 682 (2017) 323–331. https://doi.org/10.1016/j.msea.2016.11.066.

[11] R.K. Gupta, N. Birbilis, The influence of nanocrystalline structure and processing route on corrosion of stainless steel: A review, Corros. Sci. 92 (2015) 1–15. https://doi.org/10.1016/j.corsci.2014.11.041.

[12] M. El-Tahawy, Y. Huang, T. Um, H. Choe, J.L. Lábár, T.G. Langdon, J. Gubicza, Stored energy in ultrafine-grained 316L stainless steel processed by high-pressure torsion, J. Mater. Res. Technol. (2017) 1–9. https://doi.org/10.1016/j.jmrt.2017.05.001.

[13] M. El-Tahawy, J. Gubicza, Y. Huang, H.L. Choi, H.M. Choe, J.L. Lábár, T.G. Langdon, The Influence of Plastic Deformation on Lattice Defect Structure and Mechanical Properties of 316L Austenitic Stainless Steel, Mater. Sci. Forum. 885 (2017) 13–18. https://doi.org/10.4028/www.scientific.net/MSF.885.13.

[14] M. El-tahawy, P. Henrique, R. Pereira, Y. Huang, H. Park, H. Choe, T.G. Langdon, Exceptionally high strength and good ductility in an ultrafine-grained 316L steel processed by severe plastic deformation and subsequent annealing, Mater. Lett. 214 (2018) 240–242. https://doi.org/10.1016/j.matlet.2017.12.040.

[15] E. Hug, R. Prasath Babu, I. Monnet, A. Etienne, F. Moisy, V. Pralong, N. Enikeev, M. Abramova, X. Sauvage, B. Radiguet, Impact of the nanostructuration on the corrosion resistance and hardness of irradiated 316 austenitic stainless steels, Appl. Surf. Sci. 392 (2017) 1026–1035. https://doi.org/10.1016/j.apsusc.2016.09.110.

[16] B. Ravi Kumar, S. Sharma, B. Mahato, Formation of ultrafine grained microstructure in the austenitic stainless steel and its impact on tensile properties, Mater. Sci. Eng. A. 528 (2011) 2209–2216. https://doi.org/10.1016/j.msea.2010.11.034.

[17] S.V. Muley, A.N. Vidvans, G.P. Chaudhari, S. Udainiya, An assessment of ultra fine grained 316L stainless steel for implant applications, Acta Biomater. 30 (2016) 408–419. https://doi.org/10.1016/j.actbio.2015.10.043.

[18] K.D. Ralston, N. Birbilis, Effect of grain size on corrosion, Corrosion. 66 (2010) 1–4. https://doi.org/10.5006/1.3462912.

[19] K.D. Ralston, N. Birbilis, C.H.J. Davies, Revealing the relationship between grain size and corrosion rate of metals, Scr. Mater. 63 (2010) 1201–1204. https://doi.org/10.1016/j.scriptamat.2010.08.035.

[20] S. Mohd Yusuf, M. Nie, Y. Chen, S. Yang, N. Gao, Microstructure and corrosion performance of 316L stainless steel fabricated by Selective Laser Melting and processed through high-pressure torsion, J. Alloys Compd. 763 (2018) 360–375. https://doi.org/10.1016/j.jallcom.2018.05.284.

[21] S. Mohd Yusuf, Y. Chen, S. Yang, N. Gao, Microstructural evolution and strengthening of selective laser melted 316L stainless steel processed by high-pressure torsion, Mater. Charact. 159 (2020) 110012. https://doi.org/10.1016/j.matchar.2019.110012.

[22] S. Mohd Yusuf, Y. Chen, S. Yang, N. Gao, Micromechanical Response of Additively Manufactured 316L Stainless Steel Processed by High-Pressure Torsion, Adv. Eng. Mater. 22 (2020). https://doi.org/10.1002/adem.202000052.

[23] S. Mohd Yusuf, D. Lim, Y. Chen, S. Yang, N. Gao, Tribological behaviour of 316L stainless steel additively manufactured by laser powder bed fusion and processed via high-pressure torsion, J. Mater. Process. Technol. 290 (2021) 116985. https://doi.org/10.1016/j.jmatprotec.2020.116985.

[24] K. Saeidi, X. Gao, Y. Zhong, Z.J. Shen, Hardened austenite steel with columnar sub-grain structure formed by laser melting, Mater. Sci. Eng. A. 625 (2015) 221–229. https://doi.org/10.1016/j.msea.2014.12.018.

[25] W.M. Tucho, V.H. Lysne, H. Austbø, A. Sjolyst-Kverneland, V. Hansen, Investigation of effects of process parameters on microstructure and hardness of SLM manufactured SS316L, J. Alloys Compd. 740 (2018) 910–925. https://doi.org/10.1016/j.jallcom.2018.01.098.

[26] M.S. Pham, B. Dovgyy, P.A. Hooper, Twinning induced plasticity in austenitic stainless steel 316L made by additive manufacturing, Mater. Sci. Eng. A. 704 (2017) 102–111. https://doi.org/10.1016/j.msea.2017.07.082.

[27] A. Hemmasian Ettefagh, S. Guo, Electrochemical behavior of AISI316L stainless steel parts produced by laser-based powder bed fusion process and the effect of post annealing process, Addit. Manuf. 22 (2018) 153–156. https://doi.org/10.1016/j.addma.2018.05.014.

[28] K. Geenen, A. Röttger, W. Theisen, Corrosion behavior of 316L austenitic steel processed by selective laser melting, hot-isostatic pressing, and casting, Mater. Corros. 9999 (2017) 1–12. https://doi.org/10.1002/maco.201609210.

[29] H. Li, M. Ramezani, M. Li, C. Ma, J. Wang, Tribological performance of selective laser melted 316L stainless steel, Tribiology Int. 128 (2018) 121–129. https://doi.org/10.1016/j.triboint.2018.07.021.

[30] H. Li, M. Ramezani, M. Li, C. Ma, J. Wang, Effect of process parameters on tribological performance of 316L stainless steel parts fabricated by selective laser melting, Manuf. Lett. 16 (2018) 36–39. https://doi.org/10.1016/j.mfglet.2018.04.003.

[31] L. Cordova, M. Campos, T. Tinga, Revealing the Effects of Powder Reuse for Selective Laser Melting by Powder Characterization, Jom. 71 (2019) 1062–1072. https://doi.org/10.1007/s11837-018-3305-2.

[32] J.A. Slotwinski, E.J. Garboczi, K.M. Hebenstreit, Porosity Measurements and Analysis for Metal Additive Manufacturing Process Control, J. Res. Natl. Inst. Stand. Technol. 119 (2014) 494. https://doi.org/10.6028/jres.119.019.

[33] O.A. Quintana, J. Alvarez, R. Mcmillan, W. Tong, C. Tomonto, Effects of Reusing Ti-6Al-4V Powder in a Selective Laser Melting Additive System Operated in an Industrial Setting, Jom. 70 (2018) 1863–1869. https://doi.org/10.1007/s11837-018-3011-0.

[34] L. Lutterotti, S. Gialanella, X-Ray Diffraction Characterization of Heavily Deformed Metallic Specimens, Acta Mater. 46 (1998) 101–110.

[35] L.B. Mccusker, R.B. Von Dreele, D.E. Cox, D. Louër, P. Scardi, Rietveld refinement guidelines, J. Appl. Crystallogr. 32 (1999) 36–50. https://doi.org/10.1107/S0021889898009856.

[36] R.A. Young, D.B. Wiles, Profile Shape Functions in Rietveld Refinements, J. Appl. Crystallogr. 15 (1982) 430–438.

[37] W.M. Tucho, P. Cuvillier, A. Sjolyst-Kverneland, V. Hansen, Microstructure and hardness studies of Inconel 718 manufactured by selective laser melting before and after solution heat treatment, Mater. Sci. Eng. A. 689 (2017) 220–232. https://doi.org/10.1016/j.msea.2017.02.062.

[38] K. Saeidi, X. Gao, F. Lofaj, L. Kvetková, Z.J. Shen, Transformation of austenite to duplex austenite-ferrite assembly in annealed stainless steel 316L consolidated by laser melting, J. Alloys Compd. 633 (2015) 463–469. https://doi.org/10.1016/j.jallcom.2015.01.249.

[39] N. Chen, G. Ma, W. Zhu, A. Godfrey, Z. Shen, G. Wu, X. Huang, Enhancement of an additive-manufactured austenitic stainless steel by post-manufacture heat-treatment, Mater. Sci. Eng. A. 759 (2019) 65–69. https://doi.org/10.1016/j.msea.2019.04.111.

[40] X. Sauvage, N. Enikeev, R. Valiev, Y. Nasedkina, M. Murashkin, Atomic-scale analysis of the segregation and precipitation mechanisms in a severely deformed Al-Mg alloy, Acta Mater. 72 (2014) 125–136. https://doi.org/10.1016/j.actamat.2014.03.033.

[41] X. Sauvage, G. Wilde, S. V. Divinski, Z. Horita, R.Z. Valiev, Grain boundaries in ultrafine grained materials processed by severe plastic deformation and related phenomena, Mater. Sci. Eng. A. 540 (2012) 1–12. https://doi.org/10.1016/j.msea.2012.01.080.

[42] X. Sauvage, A. Ganeev, Y. Ivanisenko, N. Enikeev, M. Murashkin, R. Valiev, Grain boundary segregation in UFG alloys processed by severe plastic deformation, Adv. Eng. Mater. 14 (2012) 968–974. https://doi.org/10.1002/adem.201200060.

[43] A. Thorvaldsen, The intercept method—1. Evaluation of grain shape, Acta Mater. 45 (1997) 587–594. https://doi.org/10.1016/S1359-6454(96)00197-8.

[44] G.K. Williamson, R.E. Smallman, III. Dislocation densities in some annealed and cold-worked metals from measurements on the X-ray Debye-Scherrer spectrum, Philos. Mag. 1 (1956) 34–46. https://doi.org/10.1080/14786435608238074.

[45] M. Tikhonova, N. Enikeev, R.Z. Valiev, A. Belyakov, R. Kaibyshev, Submicrocrystalline austenitic stainless steel processed by cold or warm high pressure torsion, Mater. Sci. Forum. 838–839 (2016) 398–403. https://doi.org/10.4028/www.scientific.net/MSF.838-839.398.

[46] Y.Z. Tian, Z.F. Zhang, T.G. Langdon, Achieving homogeneity in a two-phase Cu-Ag composite during high-pressure torsion, J. Mater. Sci. 48 (2013) 4606–4612. https://doi.org/10.1007/s10853-012-7105-8.

[47] C. Xu, Z. Horita, T.G. Langdon, The evolution of homogeneity in an aluminum alloy processed using high-pressure torsion, Acta Mater. 56 (2008) 5168–5176. https://doi.org/10.1016/j.actamat.2008.06.036.

[48] Y. Song, W. Wang, D. Gao, E.Y. Yoon, D.J. Lee, C.S. Lee, H.S. Kim, Hardness and microstructure of interstitial free steels in the early stage of high-pressure torsion, J. Mater. Sci. 48 (2013) 4698–4704. https://doi.org/10.1007/s10853-012-7031-9.

[49] S. Descartes, C. Desrayaud, E.F. Rauch, Inhomogeneous microstructural evolution of pure iron during high-pressure torsion, Mater. Sci. Eng. A. 528 (2011) 3666–3675. https://doi.org/10.1016/j.msea.2011.01.029.

[50] C. Xu, Z. Horita, T.G. Langdon, The evolution of homogeneity in processing by high-pressure torsion, Acta Mater. 55 (2007) 203–212. https://doi.org/10.1016/j.actamat.2006.07.029.

[51] M. Kawasaki, H.J. Lee, B. Ahn, A.P. Zhilyaev, T.G. Langdon, Evolution of hardness in ultrafine-grained metals processed by high-pressure torsion, J. Mater. Res. Technol. 3 (2014) 311–318. https://doi.org/10.1016/j.jmrt.2014.06.002.

[52] Y. Estrin, A. Vinogradov, Extreme grain refinement by severe plastic deformation: A wealth of challenging science, Acta Mater. 61 (2013) 782–817. https://doi.org/10.1016/j.actamat.2012.10.038.

[53] Y. Chen, Y. Tang, H. Zhang, N. Hu, N. Gao, M.J. Starink, Microstructures and hardness prediction of an ultrafine-grained al-2024 alloy, Metals (Basel). 9 (2019) 1–10. https://doi.org/10.3390/met9111182.

[54] A. Loucif, R.B. Figueiredo, T. Baudin, F. Brisset, T.G. Langdon, Microstructural evolution in an Al-6061 alloy processed by high-pressure torsion, Mater. Sci. Eng. A. 527 (2010) 4864–4869. https://doi.org/10.1016/j.msea.2010.04.027.

[55] A. Loucif, R.B. Figueiredo, T. Baudin, F. Brisset, R. Chemam, T.G. Langdon, Ultrafine grains and the Hall-Petch relationship in an Al-Mg-Si alloy processed by high-pressure torsion, Mater. Sci. Eng. A. 532 (2012) 139–145. https://doi.org/10.1016/j.msea.2011.10.074.

[56] A.P. Zhilyaev, G. V. Nurislamova, B.K. Kim, M.D. Baró, J.A. Szpunar, T.G. Langdon, Experimental parameters influencing grain refinement and microstructural evolution during high-pressure torsion, Acta Mater. 51 (2003) 753–765. https://doi.org/10.1016/S1359-6454(02)00466-4.

[57] R.B. Figueiredo, M. Kawasaki, T.G. Langdon, An evaluation of homogeneity and heterogeneity in metals processed by high-pressure torsion, Acta Phys. Pol. A. 122 (2012) 425–429. https://doi.org/10.12693/APhysPolA.122.425.

[58] J. Wongsa-Ngam, M. Kawasaki, T.G. Langdon, The development of hardness homogeneity in a Cu-Zr alloy processed by equal-channel angular pressing, Mater. Sci. Eng. A. 556 (2012) 526–532. https://doi.org/10.1016/j.msea.2012.07.022.

[59] M. Kawasaki, R.B. Figueiredo, T.G. Langdon, An investigation of hardness homogeneity throughout disks processed by high-pressure torsion, Acta Mater. 59 (2011) 308–316. https://doi.org/10.1016/j.actamat.2010.09.034.

[60] S. Scheriau, Z. Zhang, S. Kleber, R. Pippan, Deformation mechanisms of a modified 316L austenitic steel subjected to high pressure torsion, Mater. Sci. Eng. A. 528 (2011) 2776–2786. https://doi.org/10.1016/j.msea.2010.12.023.

[61] Y. Mine, Z. Horita, Y. Murakami, Effect of hydrogen on martensite formation in austenitic stainless steels in high-pressure torsion, Acta Mater. 57 (2009) 2993–3002. https://doi.org/10.1016/j.actamat.2009.03.006.

[62] R. Kulagin, Y. Beygelzimer, Y. Ivanisenko, A. Mazilkin, H. Hahn, Modelling of High Pressure Torsion using FEM, Procedia Eng. 207 (2017) 1445–1450. https://doi.org/10.1016/j.proeng.2017.10.911.

[63] A. Hadadzadeh, C. Baxter, B.S. Amirkhiz, M. Mohammadi, Strengthening mechanisms in direct metal laser sintered AlSi10Mg: Comparison between virgin and recycled powders, Addit. Manuf. 23 (2018) 108–120. https://doi.org/10.1016/j.addma.2018.07.014.

[64] B. Chen, S.K. Moon, X. Yao, G. Bi, J. Shen, J. Umeda, K. Kondoh, Strength and strain hardening of a selective laser melted AlSi10Mg alloy, Scr. Mater. 141 (2017) 45–49. https://doi.org/10.1016/j.scriptamat.2017.07.025.

[65] M.J. Starink, X.G. Qiao, J. Zhang, N. Gao, Predicting grain refinement by cold severe plastic deformation in alloys using volume averaged dislocation generation, Acta Mater. 57 (2009) 5796–5811. https://doi.org/10.1016/j.actamat.2009.08.006.

[66] M.J. Starink, X. Cheng, S. Yang, Hardening of pure metals by high-pressure torsion: A physically based model employing volume-averaged defect evolutions, Acta Mater. 61 (2013) 183–192. https://doi.org/10.1016/j.actamat.2012.09.048.

[67] J. li Ning, E. Courtois-Manara, L. Kurmanaeva, A. V. Ganeev, R.Z. Valiev, C. Kübel, Y. Ivanisenko, Tensile properties and work hardening behaviors of ultrafine grained carbon steel and pure iron processed by warm high pressure torsion, Mater. Sci. Eng. A. 581 (2013) 8–15. https://doi.org/10.1016/j.msea.2013.05.008.

[68] M.A. Meyers, A. Mishra, D.J. Benson, Mechanical properties of nanocrystalline materials, Prog. Mater. Sci. 51 (2006) 427–556. https://doi.org/10.1016/j.pmatsci.2005.08.003.

[69] M.V. Karavaeva, M.M. Abramova, N.A. Enikeev, G.I. Raab, R.Z. Valiev, Superior strength of austenitic steel produced by combined processing, including equal-channel angular pressing and rolling, Metals (Basel). 6 (2016). https://doi.org/10.3390/met6120310.

[70] J.G. Kim, N.A. Enikeev, J.B. Seol, M.M. Abramova, M. V. Karavaeva, R.Z. Valiev, C.G. Park, H.S. Kim, Superior Strength and Multiple Strengthening Mechanisms in Nanocrystalline TWIP Steel, Sci. Rep. 8 (2018) 1–10. https://doi.org/10.1038/s41598-018-29632-y.

[71] N. Hansen, Hall-petch relation and boundary strengthening, Scr. Mater. 51 (2004) 801–806. https://doi.org/10.1016/j.scriptamat.2004.06.002.

|  |  |
| --- | --- |
|  | © 2019 by the authors. Submitted for possible open access publication under the terms and conditions of the Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/). |