

Nano-twinning and deformation-induced martensitic transformation in a duplex stainless steel 2205 fabricated by laser powder bed fusion

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ABSTRACT

Duplex stainless steels (DSSs) possess desirable combinations of mechanical properties and excellent corrosion resistance due to their composition and equilibrium microstructure of roughly equivalent fractions of ferrite and austenite. They are used in harsh environments such as marine infrastructures, oil & gas, and paper & pulp industries. Components with complex geometries are often required for these applications. Additive manufacturing (AM) techniques such as laser powder bed fusion (LPBF) can be harnessed to fabricate components with greatest complexity. However, AM fabrication is well-known to promote non-equilibrium microstructures with high dislocation densities and Cr₂N precipitates, resulting in inferior ductility. This is generally regarded as a challenge, however, short heat treatments of such as-built microstructures have been shown to attain refined duplex equilibrium microstructures. Recently, annealed LPBF DSS 2205 has been reported to possess strength higher than wrought counterparts and ductility properties better than the as-built state. However, the microstructural phenomena and deformation mechanisms behind these attractive properties remain poorly understood. Through multi-scale microstructural characterization, we show that the improved strength results not only from the hard ferrite phase, but also fine austenite grain size and nanoscale oxide dispersion strengthening. The enhanced ductility may be attributed to a combination of deformation mechanisms including dislocation slip, stacking fault formation, deformation twinning, and a deformation-induced martensitic transformation. We discuss how the level of microstructural complexity and solid-state phase transformations during LPBF and annealing can unlock multiple strengthening mechanisms during tensile deformation. Such fundamental understanding is crucial for designing AM parts with reproducible and optimised mechanical properties.

1. Introduction

Additive manufacturing (AM) has enabled the rapid production of net-shaped and near net-shaped components with complex geometries. AM involves fabricating parts through a layer-by-layer process informed by a digital computer-aided design (CAD) model, and has therefore unlocked numerous benefits such as design freedom and low waste when compared to conventional subtractive manufacturing routes [1,2]. Among various available metal AM technologies, laser powder bed fusion (LPBF) is one of the most versatile ones and has already found widespread industrial applications due to its capability to process a wide range of alloys [3]. The localized melting and solidification during LPBF and the corresponding cyclic rapid heating and cooling (around 10³ – 10⁸ K/s) [4] mean that each material voxel experiences complex thermal

cycles and steep thermal gradients [1,5]. This promotes the formation of non-equilibrium microstructures with high densities of defects such as dislocations and precipitates in the as-built state [4,6].

Metal AM has received significant attention in recent years with steel AM making up around 1/3 of all publications [2]. Several grades of stainless steels, including austenitic [7–9], duplex [10,11], and martensitic [12] ones have been successfully manufactured by LPBF. The complex effects of non-equilibrium microstructures are exacerbated in those steel grades that undergo subsequent solid-state phase transformations, for example, δ -ferrite to austenite and/or austenite to ferrite/martensite/bainite transformations [10–12]. For instance, 304 L stainless steel processed by LPBF was reported to contain a phase fraction of 3.7% δ -ferrite instead of being fully austenitic due to the fast cooling rate limiting the complete δ -ferrite to austenite transformation

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[7]. Recently, evidence of austenite bypassing was reported in a LPBF 17–4 PH steel, where the as-built microstructure contained more than 95% δ -ferrite [12,13]. Austenite bypassing refers to the phenomenon where δ -ferrite passes through the austenite stability temperature regime to room temperature without a transformation to austenite due to rapid cooling [12]. Similar non-equilibrium microstructures were found in as-LPBF duplex stainless steel (DSS) 2205, where the predominantly δ -ferrite microstructure largely deviated from the desired equilibrium 50/50 austenite-ferrite phase fraction [10,11].

So far, research on AM steels has been largely focused on phase transformation pathways [12,14,15], printing parameter optimization [16–18], and the effects of heat treatments and other post-processing methods [19–21]. Harnessing the metastability and high stored energy in the non-equilibrium microstructures induced by LPBF to unlock desirable mechanical properties remains poorly understood and highly sought-after. It is also understood that LPBF multi-phase steels generally form more refined microstructures via solid-state phase transformations during and/or after printing than their wrought counterparts [10], and this may be harnessed to achieve optimised properties.

Due to their remarkable mechanical (strength, toughness) and corrosion properties achieved via the synergistical combination of the properties of δ -ferrite (referred to as ferrite in the following) and austenite, DSSs are widely employed in harsh environments, including in marine applications, the oil & gas infrastructure, in desalination plants, and in the paper & pulp industries [22]. Apart from their technological importance, DSS can be regarded as a model system [23] for research on δ -ferrite to austenite phase transformations, to potentially control properties via tuning the phase transformation pathways. The predominantly δ -ferritic as-LPBF microstructure contains considerable defects including an abundance of Cr_2N and a high density of dislocations. This leads to degraded ductility, while providing opportunities for nucleation and growth of refined austenite due to the defects acting as nucleation sites for solid-state phase transformations during annealing [10]. This has been shown to result in excellent combinations of strength and toughness, in some cases better than wrought counterparts [10].

Despite the improvement in ductility of LPBF DSSs after annealing being previously reported [10,11], a fundamental understanding of the deformation mechanisms of the duplex microstructure is still lacking when compared to single phase austenitic LPBF stainless steels [7–9]. In particular, the presence of fine austenite grains embedded into a ferrite matrix can potentially activate different types of deformation mechanisms and, hence, in some cases, may enhance plasticity. Austenite was reported to accommodate more plastic deformation compared to ferrite in wrought DSS [24], where additional deformation modes may be unlocked depending on the stacking fault energy (SFE) [25]. Both deformation twinning [26,27] and deformation-induced martensitic transformations [28] have been observed in the austenite phase of wrought DSSs. A combination of these two mechanisms has been reported in a conventionally processed lean DSS [29]. Apart from the SFE, the tendency for deformation-induced martensitic transformations in wrought DSSs may also be sensitive to grain size and the presence of twins [26,30]. Given the distinct austenite morphology obtained through solid-state phase transformations from a highly non-equilibrium LPBF ferritic microstructure to an equilibrium duplex microstructure, it is essential to advance the limited understanding of the microstructural evolution and deformation mechanisms during plastic deformation of fine LPBF DSS microstructures after annealing.

Here, we apply high-resolution electron microscopy techniques to analyse the initial microstructures as well as microstructural evolution during tensile deformation of a LPBF DSS. Correlative scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), conventional transmission electron microscopy (TEM) and scanning TEM (STEM) are used to advance the understanding of the microstructure-property relationships in post-LPBF heat-treated (LPBF+HT) DSS 2205 samples. Our results reveal the underlying fundamental deformation

mechanisms responsible for the excellent combination of mechanical properties in this material, with particular focus on the significantly improved ductility compared to the as-built counterpart.

2. Material and methods

2.1. Materials processing

The DSS 2205 powder used was obtained from Sandvik Osprey Ltd, with a chemical composition of 22.60 Cr, 5.90 Ni, 3.20 Mo, 1.10 Mn, 0.6 Si, 0.18 N, 0.02 P, 0.02 C, 0.01 S (in wt%), and a balance of Fe. The powder particles were of mostly spherical morphology, 97.83% of these particles had particle sizes of 15–53 μm in diameter while 1.37% had a diameter below 15 μm and 0.80% had a diameter more than 53 μm as measured by sieve and laser diffraction analyses. An SLM Solution 125^{HL} machine equipped with a 400 W continuous wavelength fibre laser was used to print the samples under an Ar shielding gas where the O_2 content was less than 200 ppm. The printing parameters were 200 W laser power, 700 mm/s scanning speed, 60 μm hatch spacing, 30 μm layer thickness, and a 67° incremental rotation between subsequent layers. The substrate was a 316 L stainless steel plate pre-heated to 200 °C. Cubic samples with an edge length of 15 mm (Fig. 1(a)) and tensile testing samples with dimensions as shown in Fig. 1(b) (dog-bone shaped, 4 mm wide, 2.20 mm thick, and 18 mm in gauge length) were fabricated using LPBF. The tensile samples were printed horizontally, with the build direction (BD) perpendicular to the future tensile direction. A subsequent heat treatment (HT) was performed on selected samples at 1000 °C for 10 min under Ar atmosphere in a tube furnace, followed by water quenching to achieve a fine equilibrium microstructure [10]. The overall composition of the sample is measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES) and low-element combustion oxidation (LECO) analyses and shown in Table 1. A wrought DSS 2205 with chemical composition of 23.20 Cr, 5.60 Ni, 2.90 Mo, 1.82 Mn, 0.321 Si, 0.034 Co, 0.245 N, 0.153 Cu, 0.013 P, 0.036 C (in wt%), and a balance of Fe was used for mechanical property benchmarking. The wrought samples were fabricated from a commercial DSS 2205 plate hot-rolled with a 20 mm thickness.

2.2. Mechanical testing

Tensile testing was conducted at a constant crosshead speed of 1 mm/minute (strain rate of 0.05/minute) at room temperature, using an Instron 5982 tensile testing machine furnished with an MTS laser extensometer LX500. The tests were repeated using 2–3 samples for each condition. Data reported was collected from as-LPBF, LPBF+HT and wrought samples.

2.3. Metallographic sample preparation

The LPBF and LPBF+HT samples prior to deformation were studied by EBSD on the x-z surfaces in their middle regions along the BD (z-direction). The fractured tensile sample for LPBF+HT was categorised into regions of low strain (~1500 μm from the fracture surface), intermediate strain (~1000 μm), and high strain (~30 μm). Subsequent EBSD mapping was performed on these characteristic regions. Metallographic specimen surfaces were prepared for EBSD by fine polishing using 0.04 μm OPU colloidal silica as the final step. The fracture surface of the LPBF+HT sample was directly observed via SEM. (S)TEM specimens from undeformed samples were prepared using twinjet electropolishing with a Struers Tenupol and a Struers A2 electrolyte at a voltage of 26 V and a temperature of around –20 °C. (S)TEM specimens from the fractured LPBF+HT sample were prepared from EBSD specimens using a standard plasma focussed ion beam (FIB) lift-out technique [31].

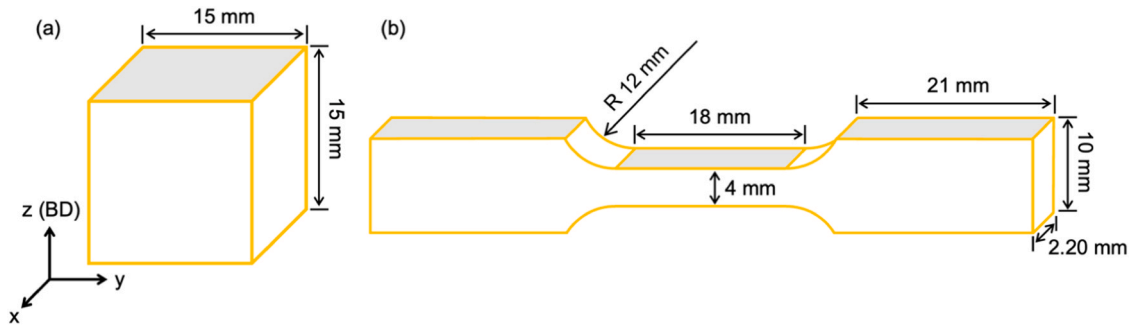


Fig. 1. Schematic illustration of printed samples. (a) Cubic sample and (b) tensile testing sample orientation, geometries, and dimensions. Dimensional accuracy is 0.1 mm.

Table 1

Austenite and ferrite chemical composition (wt%) of LPBF+HT DSS 2205 averaged from atom probe measurements from two different austenite/ferrite volumes. Overall chemical composition (wt%) of LPBF+HT DSS 2205 measured by ICP-AES and LECO.

Element (wt%)	N	Si	Cr	Mn	Fe	Ni	Mo	P
Austenite	0.34	0.88	19.99	1.23	67.28	7.57	2.71	0.003
Ferrite	0.04	1.05	23.72	0.91	66.05	3.95	4.27	0.013
Overall	0.16	0.81	22.40	1.06	66.69	5.63	3.24	0.010

2.4. Electron microscopy

SEM-EBSD characterisation was carried out using a JEOL 7001 F SEM equipped with a Hikari 31 Super EBSD Camera at an accelerating voltage of 20 kV, a probe current of 13 nA, a 6×6 binning, and step sizes ranging from 0.1 to 1 μm . The data were obtained and processed using the TSL-OIM software. It should be noted that a deformation-induced martensitic transformation occurred during tensile testing. To differentiate martensite from ferrite, which both have body-centred cubic crystal structures in this steel, confidence index (CI) and image quality (IQ) are used for thresholding. A similar approach has previously been applied [32], and the method is detailed in [33]. Atom probe tomography (APT) measurements were performed with a CAMECA Local Electrode Atom Probe 4000X Si following a procedure detailed in [10]. FIB lift-out and thinning for (S)TEM specimens was conducted using a Thermo Fisher Helios G4 Xe plasma FIB. Conventional TEM observations were performed in a JEOL 2100 at 200 kV. STEM characterisations were performed in a Titan Themis-Z double-corrected S/TEM equipped with ChemiSTEM (Super-X) EDXS detectors operated at 300 kV. STEM high-angle annular dark-field (HAADF) images were obtained with a collection angle range of 76–200 mrad.

3. Results

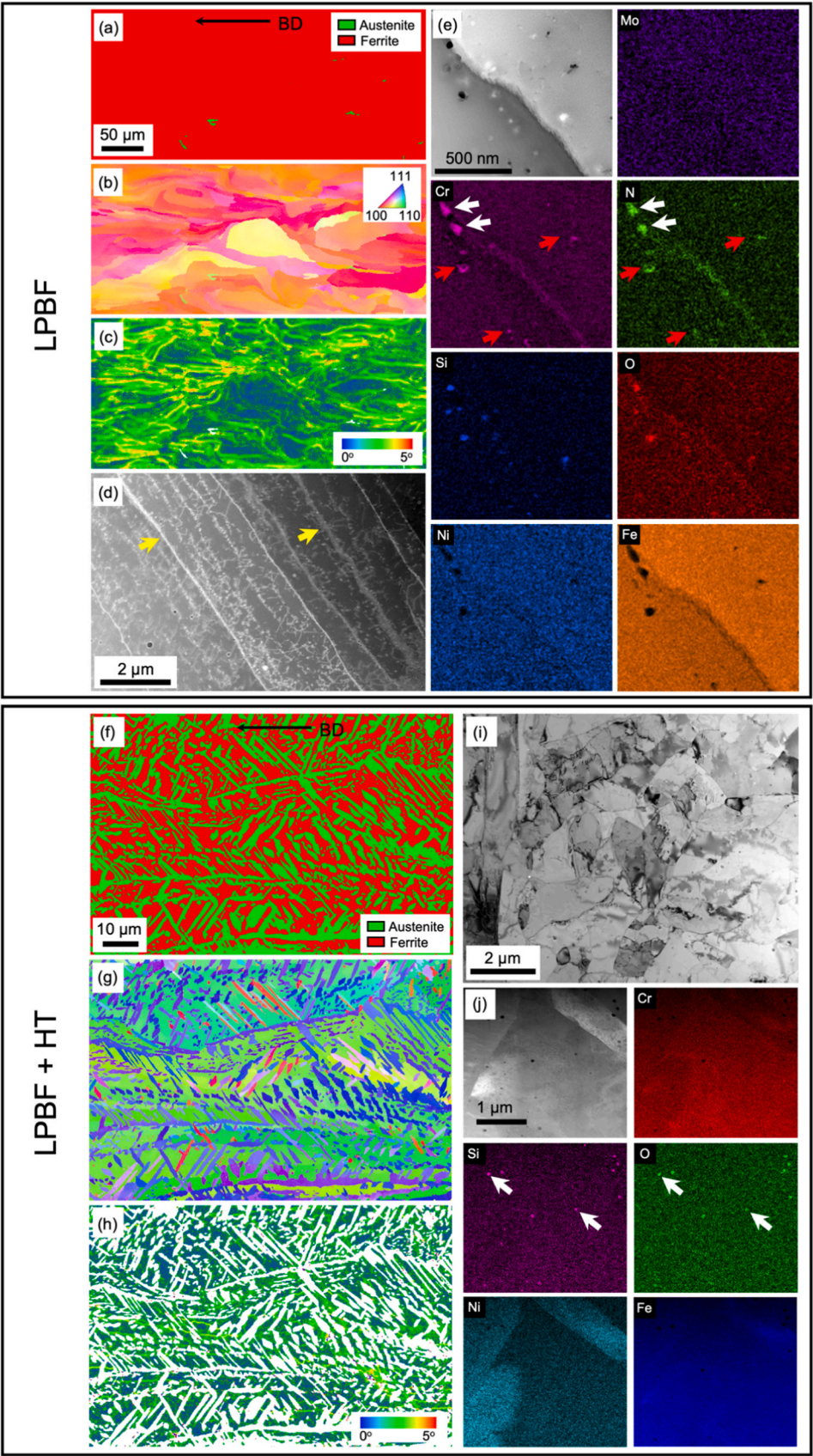
3.1. Microstructures of the LPBF and LPBF+HT DSS 2205 prior to deformation

Owing to the steep thermal gradient and rapid cooling rate during LPBF [1,4,5], the LPBF DSS 2205 exhibits a mostly ferritic microstructure with < 5% austenite as shown in the EBSD phase map in Fig. 2(a). A [100] crystallographic texture is observed along the BD, and columnar grains develop along this direction, implying epitaxial growth, as seen in the EBSD inverse pole figure (IPF) map in Fig. 2(b). An EBSD Kernel average misorientation (KAM) distribution map collected at a step size of 1 μm is displayed in Fig. 2(c), with a considerable area fraction exhibiting KAM values > 2.5°, which is the result of intrinsic deformation due to the steep thermal cycles and fast cooling rates experienced during LPBF [1,5]. The dark-field TEM micrograph in Fig. 2(d) reveals a high dislocation density in the ferrite grains which is attributed to the rapid thermal gyrations during LPBF [1,5], where the formation of dislocation substructures can also be observed as pointed out by yellow

arrows. STEM-HAADF and EDXS along a ferrite-ferrite grain boundary in Fig. 2(e) indicate the formation of intergranular and intragranular Cr_2N precipitates as well as Si- and O-rich inclusions.

The LPBF+HT DSS 2205 retrieves more balanced phase fractions as shown in EBSD phase map in Fig. 2(f), with a ~45% phase fraction of austenite according to EBSD. The fine austenite grain size is around 1–2 μm , with intergranular and intragranular austenite present as illustrated in the EBSD IPF map in Fig. 2(g). While ferrite presents a [110] crystallographic texture along the tensile direction, no apparent texture is observed in the austenite along the tensile direction as shown in Fig. 2(g). The EBSD KAM distribution map collected at a step size of 0.1 μm for ferrite in Fig. 2(h) shows that most ferrite has KAM values < 2.5°, implying that intrinsic lattice distortion due to thermal cycling has been largely recovered during the heat treatment. Fig. 2(i) shows the typical TEM microstructure of the LPBF+HT DSS 2205 where the dislocation density has significantly decreased compared to the LPBF condition in Fig. 2(d). There is no indication of the formation of twins or stacking faults (SFs) in the undeformed microstructure. STEM-HAADF and EDXS presented in Fig. 2(j) indicate that Cr_2N precipitates have dissolved while nano-sized Si- and O-rich inclusions remain present in both the austenite and ferrite phases as distinguished via chemical segregation, with Ni-enriched areas being austenite and Cr-enriched areas being ferrite. The formation of Si-O nanoprecipitates is either from the laser melting or from powder feedstock given the high affinity of Si for O_2 [10]. The rapid cooling rates typical to LPBF have been reported to generate O-rich inclusions in both single-phase and duplex stainless steels [16,34,35]. The spherical instead of faceted morphology of these Si-rich oxides indicates that they are of an amorphous nature [16,35].

Table 1 displays the overall, austenite, and ferrite chemical composition in the LPBF+HT DSS 2205. In total, two measurements were taken at different austenite/ferrite volumes, and the average for each element including N, Si, Cr, Mn, Fe, Ni, P, and Mo is tabulated. It should be noted that the APT measured austenite and ferrite compositions are very similar to those measured by SEM- Energy-dispersive X-ray spectroscopy and averaged from 19 different regions of austenite and ferrite respectively, except that APT may give a more reliable measurement on N content. It is shown that Ni tends to partition towards austenite with a measured composition of 7.570 wt%, significantly higher than the nominal composition of 5 wt%. On the other hand, Cr tends to segregate away from the austenite, with a measured composition of 19.990 wt%, lower than the nominal 22 wt%. This is consistent with Ni and Cr being



(caption on next page)

Fig. 2. Typical microstructures of the (a) – (e) LPBF and (f) – (j) LPBF+HT DSS 2205 samples. (a) EBSD phase map showing mostly ferrite, arrow indicating the BD. (b) EBSD IPF map (along BD) representing columnar grains and a [100]//BD texture. The same colour key applies to all IPF maps throughout this manuscript. (c) Corresponding KAM map collected at a step size of 1 μm . (d) TEM micrograph displaying the high dislocation density in the ferrite. Arrows point to examples of dislocation sub-structures. (e) STEM-HAADF image and corresponding elemental maps showing local enrichments in Cr and N, and Si and O. (f) EBSD phase map showing balanced fraction of austenite and ferrite after the HT. (g) EBSD IPF map along tensile direction (perpendicular to BD), representing ferrite and austenite morphology. (h) KAM map of ferrite collected at a step size of 0.1 μm . (i) TEM micrograph displaying typical microstructures of ferrite and austenite. (j) STEM-HAADF image and corresponding elemental maps showing local enrichment in Si and O, and chemical segregation of Cr and Ni towards ferrite and austenite, respectively.

austenite and ferrite stabilizing elements, respectively.

3.2. Mechanical properties and fractography of LPBF+HT DSS 2205

The engineering stress–strain curves of the LPBF, LPBF+HT, and wrought DSS 2205 are shown in Fig. 3(a) for comparison. Relevant data for mechanical properties are summarized in Table 2. This DSS exhibits a high yield strength of ~ 710 MPa and ~ 625 MPa in its LPBF and LPBF+HT states, respectively, both of which are better than that of the wrought counterpart with a yield strength of ~ 510 MPa. While the

Table 2

Average values of mechanical properties of LPBF+HT, LPBF, and wrought DSS 2205.

	LPBF+HT	LPBF	Wrought
Yield strength (MPa)	625 $\pm$ 9	710 $\pm$ 8	510 $\pm$ 5
Ultimate tensile strength (MPa)	790 $\pm$ 5	850 $\pm$ 4	700 $\pm$ 4
Uniform elongation (%)	18 $\pm$ 1	11 $\pm$ 0.5	24 $\pm$ 0.5

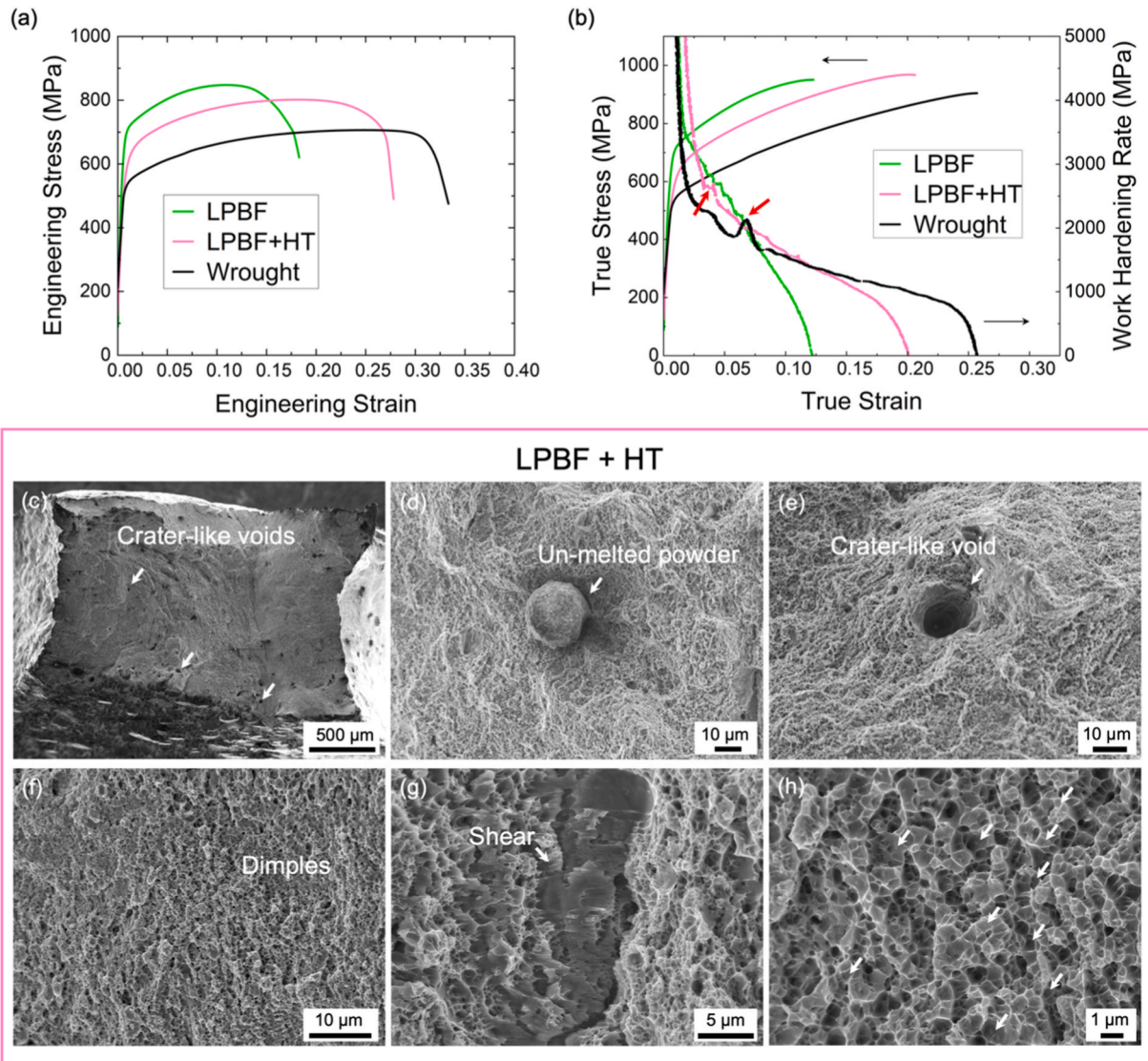


Fig. 3. Mechanical properties and fracture surfaces of DSS 2205. (a) Engineering stress–strain curve for LPBF, LPBF+HT, and wrought conditions. (b) True stress–strain and work-hardening curves for LPBF, LPBF+HT, and wrought conditions. Small black arrows point to vertical axis for true stress on the left and work-hardening on the right. Red arrows indicate the transition point of work-hardening for LPBF+HT and wrought conditions. (c) – (h) Fracture surfaces for the LPBF+HT condition at different scales showing crater-like voids, un-melted powder, typical ductile dimples, shear regions, and nano-scale inclusions (pointed out by white arrows).

uniform tensile ductility ($\sim 11\%$) of the LPBF sample is adversely impacted by its imbalanced phase fraction of predominantly ferrite and high densities of Cr_2N and dislocations, the LPBF+HT sample presents an improved uniform ductility of $\sim 18\%$, more comparable although still lower than its wrought counterpart ($\sim 24\%$). The LPBF+HT sample exhibits an excellent ultimate tensile strength at ~ 790 MPa, relatively lower than its LPBF counterpart (~ 850 MPa) but significantly greater than the wrought sample (~ 700 MPa). Fig. 3(b) presents the work-hardening rate and true stress versus true strain curves for the LPBF, LPBF+HT, and wrought samples. The work-hardening of the LPBF+HT sample is sustained for longer than the LPBF sample and higher compared to the wrought sample until a true strain of $\sim 12.5\%$ is reached. The LPBF+HT DSS 2205 even reaches higher true stress and true strain compared to its as-built counterpart. Therefore, the current LPBF+HT DSS 2205 possesses notably enhanced yield strength and ultimate tensile strength compared to its wrought counterpart, while achieving similar or even better fracture ductility when compared to previously fabricated LPBF+HT samples [11,21]. According to the Considère criterion where the onset of necking can be specified by the intersection between the true stress-strain curve and the work-hardening curve [36], the onset of necking of the LPBF+HT DSS2205 sample occurs at a later stage compared to both wrought and LPBF counterparts. This means that the ability to delay plastic instability of the LPBF+HT DSS 2205 is better than both counterparts. While no distinct stages can be identified from the true stress-strain curves, a small step indicating slip transition can be seen on the work-hardening curve of the LPBF+HT sample, immediately after the onset of necking. Similarly, a more apparent step can be observed in the curve of the wrought counterpart, occurring at a much later stage compared to that of the LPBF+HT sample. This could be caused by the occurrence of interphase slip transition [37]. However, there is no obvious sign of similar event for the LPBF sample, implying no definite stage of dislocation transmission. The fractographies shown in Fig. 3(c) – (h) capture a mix of ductile fracture morphologies with typical fine dimples alongside shear regions, occasional brittle fracture appearance, and crater-like voids with diameters of tens of microns. Nanoscale inclusions are also observed inside these ductile dimples as pointed by arrows in Fig. 3(h). During LPBF, low density weak regions are reported surrounding melt pools due to un-melted or partially un-melted powder particles getting stuck between adjacent melt pools due to insufficient heat input [38].

3.3. Microstructural evolution during deformation of LPBF+HT samples

To reveal the microstructural evolution and deformation mechanisms that led to the outstanding strength–ductility combination and work hardening capability of the LPBF+HT samples, characterisations using EBSD and TEM were performed at various distances to the fracture surface (low, intermediate, and high strain regions, see experimental

section). From the geometrically necessary dislocation (GND) density distributions collected at a step size of $0.1\text{ }\mu\text{m}$ in Fig. 4, both austenite and ferrite exhibit a higher density of GNDs with higher strain. At lower strain levels, a lower density of GNDs and a narrower distribution profile is presented. It is noticed that the GND value of ferrite is slightly higher than that of austenite when undeformed. However, in the low strain condition, the GND value of austenite starts to exceed that of ferrite, where austenite is observed to experience increasingly more severe plastic deformation compared to ferrite as deformation progresses from low to high strain. Ferrite is therefore considered to be the harder phase compared to austenite, and more strain is concentrated in austenite as it better accommodates plastic flow [7,26].

Fig. 5 shows the deformed microstructures at low and intermediate strain levels. In the low strain region, grain orientations are shown in the EBSD IPF map along the tensile direction (Fig. 5(a)) with only very slight phase transformation according to the phase map in Fig. 5(b), where the α' martensite coloured blue accounts for 1.21% area fraction of original austenite. Meanwhile, deformation twinning starts to occur as can be observed from the image quality (IQ) map in Fig. 5(c). It should be noted that the morphology of deformation twins is distinct from that of annealing twins under EBSD observation, where annealing twins are characterized by large parallel straight facets that reduce interfacial energy while deformation twins tend to be lenticular with sharp edges that minimize long range elastic strains [39,40]. Therefore, the nano-twins observed in this study match the morphology of deformation twins by being lenticular with sharp edges and curved facets. Enlarged views of deformation nano twins are illustrated in Fig. 5(e1) – (e4) where they can be observed as they are starting to form. The KAM map for ferrite in Fig. 5(d) indicates that the degree of deformation in ferrite has increased compared to the initial LPBF+HT condition. The IPF map in Fig. 5(f) shows grain orientations along the tensile direction at an intermediate strain level. Still, very small amount of phase transformation is observed in the phase map in Fig. 5(g), where the α' martensite coloured blue accounts for 2.10% area fraction of original austenite. Deformation twinning in austenite and deformation in ferrite continue to develop at intermediate strain, as evidenced by the presence of additional twin boundaries in the austenite (Fig. 5(h)) and higher KAM values in the ferrite (Fig. 5(i)). Enlarged views of deformation nano twins are shown in Fig. 5(j1) – (j4) which are higher in number and size when compared to those at low strain (Fig. 5(e)).

Fig. 6 shows the deformed microstructure in a high strain region. At high strain levels, the grains are more distorted due to severe deformation as shown in the IPF map (Fig. 6(a)) and corresponding phase map (Fig. 6(b)). However, fewer deformation twins are noticeable in the austenite at this strain level as shown in Fig. 6(c), despite more severe deformation especially in the ferrite when compared to previous stages (Fig. 5) as indicated by the KAM map in Fig. 6(d). This may be attributed to the deformation-induced martensitic transformation using the intersection between twin boundaries and dislocations as nucleation sites,

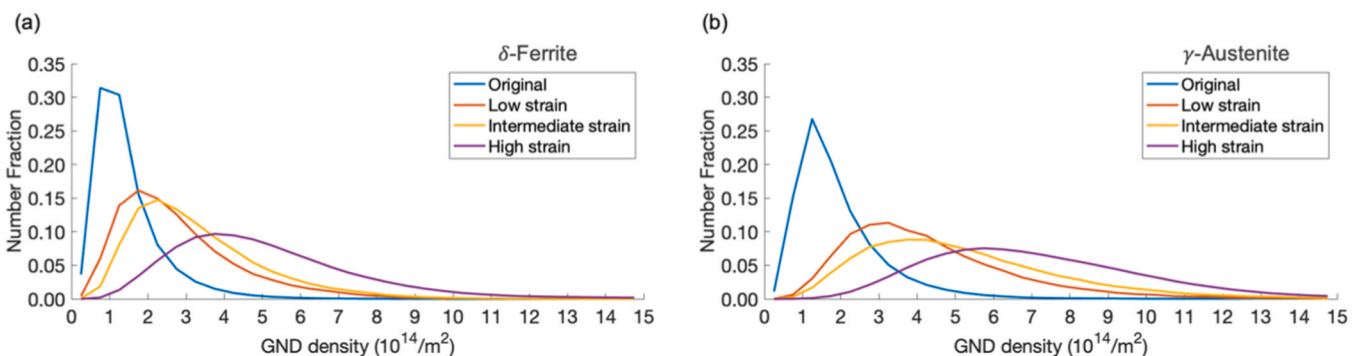


Fig. 4. GND density distributions of (a) ferrite and (b) austenite at various deformation stages including original (undeformed), low strain, intermediate strain, and high strain regions. EBSD data was collected at a step size of $0.1\text{ }\mu\text{m}$.

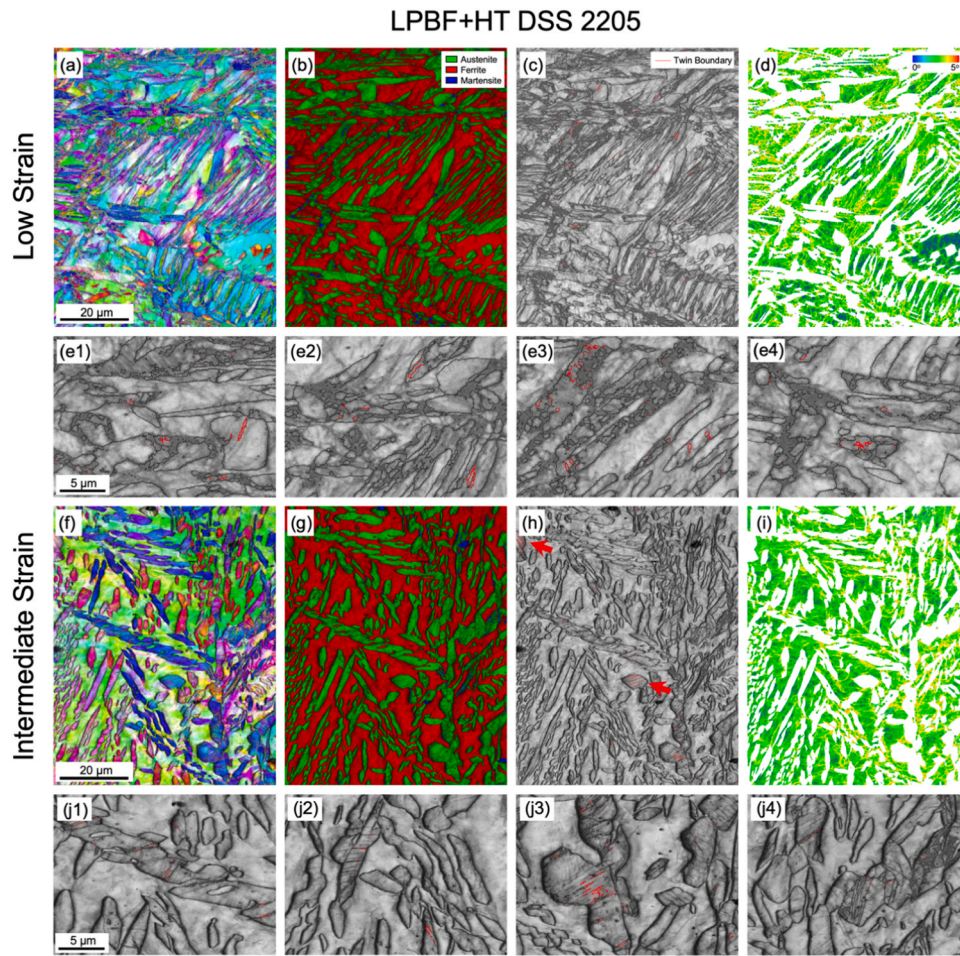


Fig. 5. Microstructures of LPBF+HT DSS 2205 at (a) – (e) low and (f) – (j) intermediate strain levels. (a,f) EBSD IPF map overlapped with IQ map. (b,g) EBSD phase map overlapped with IQ. (c) EBSD IQ map with mechanical twin boundaries shown as red lines. (d,i) KAM map of ferrite collected at a step size of 0.1 μm . (e1) – (e4) Enlarged areas from (c) with mechanical twins. (h) IQ map where twin boundaries are shown as red lines, red arrows point to austenite grains with frequent formation of deformation twins. (j1) – (j4) Enlarged areas from (h) with mechanical twins.

consuming the twinned microstructures, and leading to the disappearance of twin boundaries [41]. The blue areas in the phase map shown in Fig. 6(b) are the α' martensite, where a large area of martensitic transformation products is pointed to by an arrow. α' martensite accounts for 10.92% area fraction of original austenite at high strain level here. Enlarged views of areas with combinations of deformation nano twins and martensitic transformation are presented in Fig. 6(e) – (f), where the martensitic transformation tends to occur directly next to deformation twins and consequently consume some of them by using their intersection with dislocations as nucleation sites [28,42,43]. Arrows in Fig. 6(f1) point to large areas of martensitic transformation. As can be seen in Fig. 5 and Fig. 6, while the mechanical behaviour of ferrite during tensile deformation is governed by dislocation slip, the deformation mechanisms in austenite are more complex. Dislocation slip, deformation twinning, and deformation-induced martensitic transformation occurred in sequence, enabling multiple pathways to accommodate plastic deformation to achieve desirable mechanical properties. It should be noted that EBSD may not provide all details regarding deformation twinning and martensitic transformation due to limited spatial resolution and the fine austenite grain size here. However, it does enable consistent comparisons between different deformation stages by mapping relatively large areas (Fig. 5 and Fig. 6) and supplying statistical information such as GND distributions (Fig. 4). It is to be noted that some of the EBSD datapoints presented in Fig. 5 and Fig. 6 may represent noise, as data cleaning and filtering done were limited so that the deformed martensite containing lattice distortion and high dislocation

density can be preserved in the EBSD maps. However, IPF maps are shown for grains with $CI > 0.3$ to provide better orientation information. A more in-depth microstructural investigation is therefore performed focusing on the austenite and martensite in the following.

Detailed observations of deformation nanotwins are displayed in Fig. 7. Fig. 7(a) is the result of stitching multiple conventional TEM images to enable a more complete observation of a selected typical austenite grain with nanotwins displayed in Fig. 7(b). It shows a high density of deformation twins of different widths at the nanoscale spanning across nearly the entire austenite grain. This emphasizes that the extent and density of the deformation nanotwins could not be fully resolved by EBSD, with significantly less twin boundaries being labelled in Fig. 7(b) compared to the TEM observation in Fig. 7(a). Fig. 7(c) shows an enlarged view from within the blue rectangle in Fig. 7(a), where secondary nanotwins form and cause distortion of the primary nanotwins, which provides extra work-hardening capability via introducing lattice strain and barriers for dislocation slip [7,44]. Fig. 7(d) and inset display STEM-HAADF images and the corresponding fast Fourier transformation (FFT) showing SFs and deformation twins with thicknesses of a few nanometers or less. These observations provide insights into the deformation twinning spanning from around 30 nm down to <1 nm.

Apart from the abundant presence of deformation nanotwins, a high density of SFs is also observed as shown in Fig. 8. A conventional TEM micrograph in Fig. 8(a) displays SFs in two $\{111\}$ plane groups together with their interactions. A more detailed observation is provided in Fig. 8

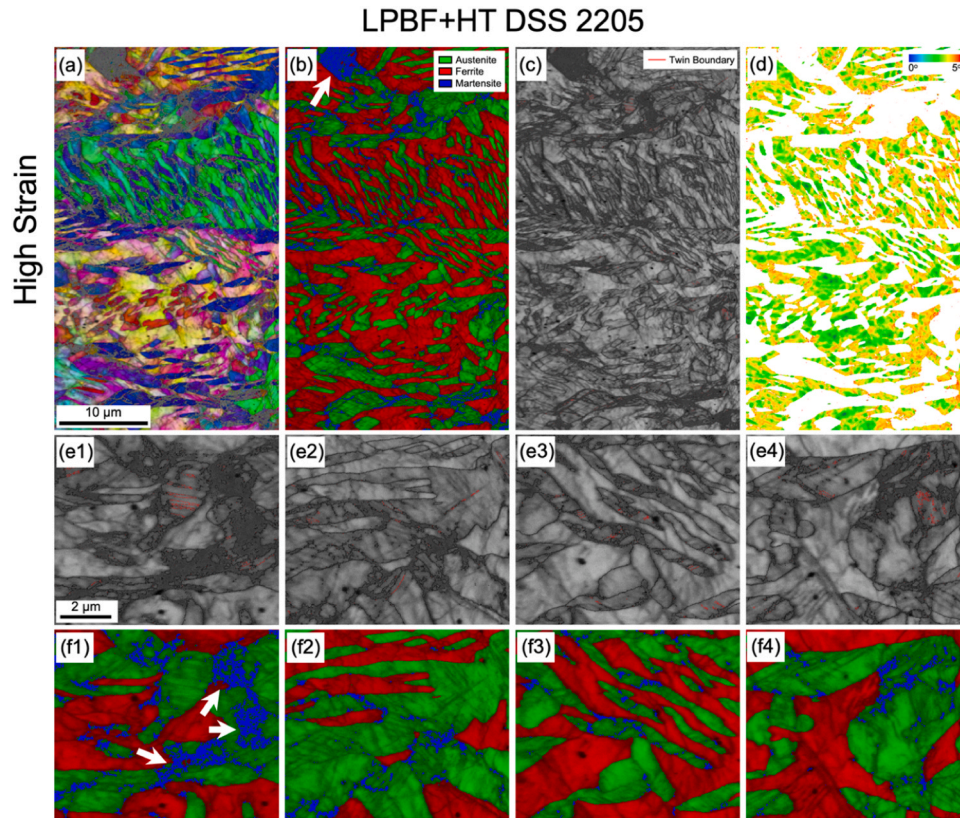


Fig. 6. Microstructures of LPBF+HT DSS 2205 at high strain level. (a) EBSD IPF map overlapped with IQ. (b) EBSD phase map overlapped with IQ, white arrow points to large areas of martensitic transformation products. (c) IQ map, twin boundaries are shown as red lines. (d) KAM map of ferrite collected at a step size of 0.1 μm . (e1) – (e4) Enlarged areas from (c) with mechanical twins. (f1) – (f4) Enlarged areas from (b) with deformation-induced martensitic transformation, white arrows point to large areas of martensitic transformation that also corresponds to darker IQ regions in (e1) – (e4).

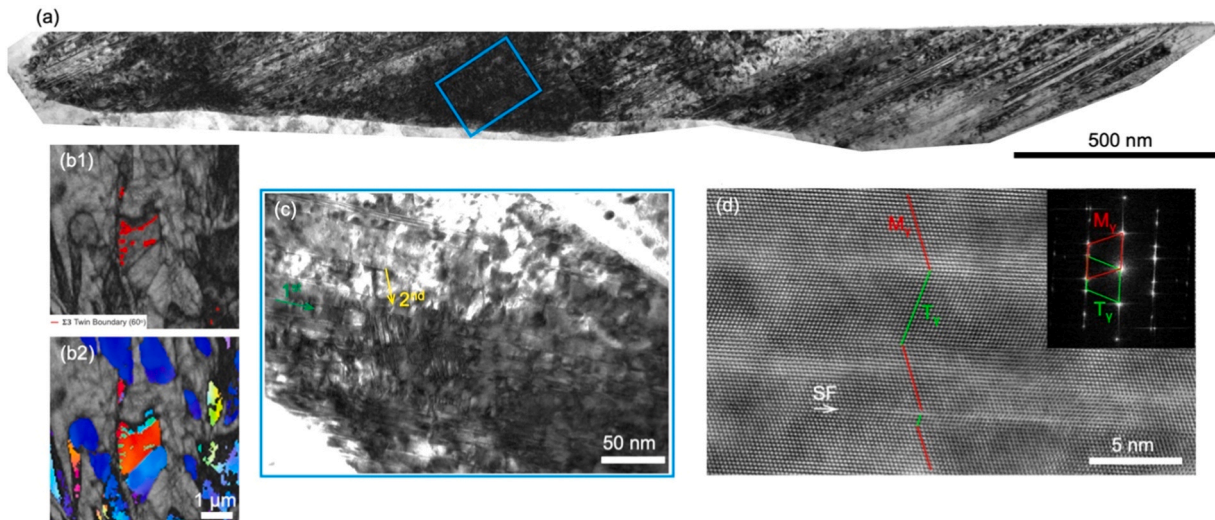


Fig. 7. Deformation-induced hierarchical nanotwins with concurrent stacking faults in the fractured LPBF+HT sample. (a) High density of deformation nanotwins in a typical austenite grain. (b1) Region including the same austenite grain as in (a), displayed as EBSD IQ map with twin boundaries marked by red lines. (b2) Region including the same austenite grain as in (a), displayed as IPF map for austenite and IQ map for ferrite. (c) Zoomed-in area from the blue rectangle in (a) showing primary and secondary deformation. (d) STEM-HAADF image and corresponding FFT showing SFs and nanotwins.

(b), where the spacings between adjacent SFs range from around 4 nm to < 1 nm along with SFs from another $\{111\}$ plane group. In addition to the dense SFs, the STEM-HAADF micrograph in Fig. 8(c) reveals the formation of sub-grain boundaries in an austenite grain, intersecting with multiple SFs. The inset indicates a low-angle misorientation

between the two austenite sub-grains. The formation of such a high density of SFs is due to plastic deformation during tensile testing. These features contribute to the improvement in mechanical properties via introducing more barriers for dislocation slip [7,44].

Fig. 9 demonstrates that the deformation-induced martensitic

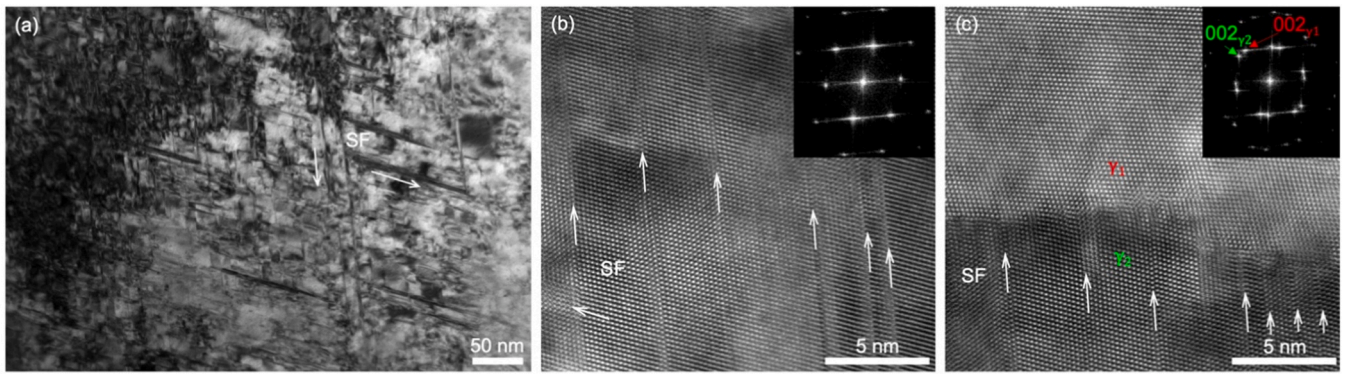


Fig. 8. Stacking faults at the nanoscale and their interactions in the fractured LPBF+HT sample. (a) Conventional TEM image showing the high density of SFs in two $\{111\}$ plane groups. (b) STEM-HAADF image showing SFs at the nanoscale, inset shows corresponding FFT. (c) STEM-HAADF image showing the formation of a sub-grain boundary within an austenite grain alongside with SFs emissions from the sub-grain boundary, inset demonstrates 2 sets of austenite FFTs with a slight misorientation in between, indicating the formation of 2 sub-grains.

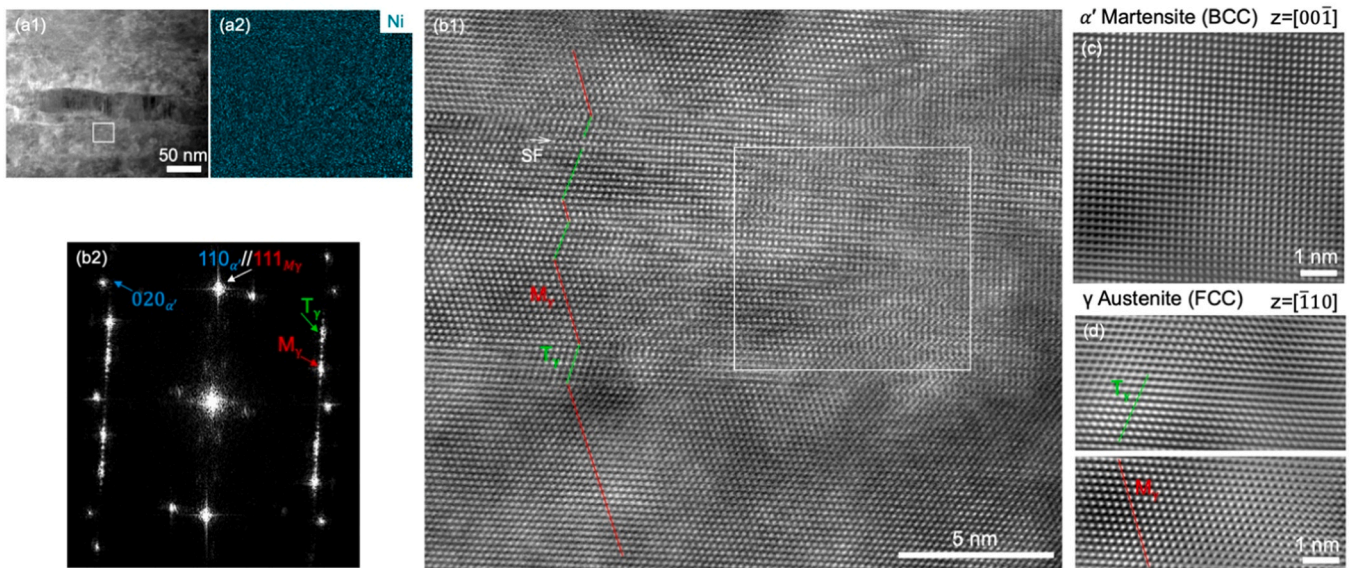


Fig. 9. Deformation-induced martensitic transformation on austenite twins and stacking faults. (a1) STEM-HAADF map showing a region within an austenite grain. (a2) EDXS Ni map from the same region as in (a1) showing no Ni segregation, implying that no diffusional phase transformation has occurred within this austenite region. (b1) STEM-HAADF image of the area within the white rectangle in (a1) showing the α' martensitic transformation on SFs and deformation nanotwins. (b2) The corresponding FFT of (b1) showing the coexistence of the austenite matrix, austenite twins, and α' martensite, pointed out by red, green, and blue colours, respectively. (c) Filtered inverted FFT image showing α' martensite from the white rectangular region in (b1) only. (d) Filtered inverted FFT image showing the austenite matrix and twinning from the white rectangular region in (b1) only.

transformation tends to occur adjacent to or at austenite nanotwins and SFs, where twin boundaries and SFs serve as nucleation sites and/or embryos for martensite [28,42,43] to facilitate a martensitic transformation in such small austenite grains, which would otherwise be inhibited as reported previously [26]. Fig. 9(a1) features the HAADF image of an area containing austenite and martensite, with the elemental map for Ni shown in Fig. 9(a2). Compared to the Ni partitioning between ferrite and austenite shown previously in Fig. 2(j), there is no indication of Ni segregation here, implying that no diffusional phase transformation has occurred within this region. This means this phase is not ferrite but that the observed phase within this region is indeed α' martensite. Fig. 9(b1) is a STEM-HAADF micrograph showing the enlarged view of the area framed by the white square in Fig. 9(a1). Austenite nanotwins at the sub-nano scale and SFs are observed in the left part of the micrograph in Fig. 9(b1), while a mix of austenite deformation twins and deformation-induced martensite are present in the right hand side of the micrograph. The FFT pattern in Fig. 9(b2) verifies the above observation by demonstrating the spots for the

austenite matrix, austenite twins, and α' martensite, pointed out by red, green, and blue colours, respectively. Viewing along the $[\bar{1}10]_{\gamma} // [00\bar{1}]_{\alpha'}$ directions, this FFT pattern also shows the orientation relationship between austenite and martensite: $(111)_{\gamma} // (110)_{\alpha'}$, same as reported in [45].

Using the FFT spots belonging to the α' martensite in Fig. 9(b2), a filtered STEM-HAADF image for the α' martensite only along the zone axis of $[001]$ is shown in Fig. 9(c). Similarly, using the FFT spots corresponding to both the matrix and twins in austenite in Fig. 9(b2), a filtered STEM-HAADF image for the austenite matrix only and twinning along the $[110]$ zone axis is shown in Fig. 9(d). This demonstrates that no ε -martensite has formed during the deformation-induced martensitic transformation process since the α' martensite is observed directly adjacent to the austenite twinning without other intermediate phases present [41].

4. Discussion

We have shown in Fig. 3 that the post-LPBF heat-treated (LPBF+HT) DSS 2205 displays an excellent combination of strength, ductility, and work-hardening rate. This material also exhibits increased yield strength and ultimate tensile strength compared to its wrought counterpart and achieves a significant improvement in ductility when compared to its as-built state. Such strengthening capability is the result of the combined effects of the fine austenite grain size of $\sim 1 - 2 \mu\text{m}$, precipitation strengthening from oxide dispersions (note that Cr_2N precipitates are mostly dissolved after heat treatment hence they do not play a role here), the abundant stacking faults, the high density of hierarchical deformation twinning, and the deformation-induced martensitic transformation. In the following, we will discuss the underlying mechanisms for each of those microstructural characteristics in more detail.

4.1. Grain refinement in the post-LPBF heat treated DSS 2205

The layer-by-layer manufacturing and the subsequent rapid solidification during LPBF result in steep thermal gradients [1,4,5], prompting high residual stresses [46,47] and plastic deformation [48,49] and, thus, a high dislocation density in the as-built ferrite matrix as evidenced in Fig. 2(d). These dislocations can form dislocation sub-structures with morphologies similar to sub-boundaries as pointed out in Fig. 2(d). The formation of dislocation sub-structures due to AM thermal cycles has been extensively reported for face-centred cubic (FCC) microstructures [7,9,38,50]. The fast cooling associated with LPBF also results in the formation of numerous Cr_2N particles (Fig. 2(e)) due to the supersaturation of N in δ -ferrite [51]. These dislocation sub-structures and precipitates provide nucleation sites for fine austenite grains upon annealing [23,52]. Dislocation sub-structures result in high free energy, which increases the diffusion rate of elements, contributing to the nucleation and growth of austenite. Cr_2N particles facilitate austenite formation via the classical minimisation of the interfacial energy [10], where austenite tends to nucleate on existing interfaces and obey the low-energy interfaces between $(111)_\gamma$ and $(0001)_{\text{Cr}_2\text{N}}$ according to a previous report [53]. Post-mortem observation of austenite nucleation on nitrides is difficult since the trapped nitrides inside the austenite grains would have been dissolved promptly [10,54]. During annealing, austenite grains only grow to a certain extent without substantial further coarsening, since the soft impingement effects imposed by the diffusion field overlapping among adjacent grains would slow down the overall phase transformation kinetics [55].

Such a level of grain refinement is barely achievable in conventional processing of DSSs due to the difficulties in introducing sufficiently high dislocation densities to form dislocation sub-structures in the ferrite matrix before austenite starts to nucleate. Attempts to achieve this via rapid quenching from the liquid regime followed by annealing treatment may introduce secondary austenite that is detrimental to the corrosion properties due to its different chemical composition [56]. It should be noted that the current LPBF+HT DSS 2205 has better corrosion properties compared to its as-built counterpart and satisfies the criteria for in-service applications [57]. Besides, if high densities of dislocations are introduced into DSSs during conventional hot forming, dynamic recovery will lead to dislocation annihilation [58,59]. Alternatively, if dislocations are introduced via cold working, deformation accumulation in the less ductile ferrite at lower temperature can result in cracking [22]. These challenges have been overcome via application of the LPBF + HT route. The significant refinement in grain size made available by LPBF is instrumental in improving the strength of the current DSS 2205 as opposed to its conventional counterpart.

4.2. Mechanical behaviour of the post-LPBF heat treated DSS 2205

Both yield strength ($\sim 625 \text{ MPa}$) and ultimate tensile strength ($\sim 790 \text{ MPa}$) of the LPBF+HT DSS 2205 are substantially higher than

those manufactured by LPBF before (yield strengths of ~ 600 [11], ~ 610 [11], and $\sim 465 \text{ MPa}$ [21], and ultimate tensile strengths of ~ 720 [11], ~ 770 [11], and $\sim 622 \text{ MPa}$ [21]). The fracture ductility of the LPBF+HT DSS 2205 ($\sim 27.5\%$) is similar or even better than DSS subjected to a heat treatment after LPBF in previous studies ($\sim 28\%$ [11] and $\sim 21.3\%$ [21]). While ferrite acts as a harder phase that mainly deforms via dislocation slip [7,26], the ability of austenite to activate multiple deformation mechanisms, including stacking fault emission and deformation twinning, is essential to achieve desirable mechanical properties. Furthermore, the higher population of coherent austenite–ferrite interfaces after the heat treatment in LPBF DSS 2205 as reported in [10] also benefits slip transition at interphase boundaries, improving ductility. This is underpinned by the presence of a slip transition point on the work-hardening curve (Fig. 3(b)) of the LPBF+HT DSS 2205 that takes place earlier than in the wrought counterpart, as opposed to the absence of such a transition point in the LPBF sample.

As can be seen in Fig. 5(e), during the initial deformation stages, dislocation slip dominates the deformation mode while deformation twinning starts to occur as evidenced by the low twin density. As strain increases, a higher density of SFs and nano scale deformation twins =start to appear (Fig. 7 and Fig. 8). The strong interactions between SFs help to sustain the plastic flow and achieve higher strength. Extended dislocations with wide SFs usually lead to planar slip as it is difficult for them to cross-slip, leading to high dislocation densities on individual planes and strengthening [60]. On the other hand, deformation twin boundaries support work-hardening by acting as strong barriers for dislocations and dislocation accumulation sites. At the later stages, the α' martensitic transformation also occurs (Fig. 6 and Fig. 9). Therefore, the deformation sequence in austenite is initially via full and/or partial dislocation slip, followed by deformation-induced nano-twinning and a martensitic transformation. This means that the (true) stress needed for slip is lower.

Although being previously reported for other metallic materials at cryogenic temperature [62], the co-existence of deformation twinning and the α' martensitic transformation in fine austenite grains of LPBF DSS 2205 is novel. While a martensitic transformation was previously observed in conventionally processed DSS 2205 with a much larger grain size of $\sim 50 \mu\text{m}$ [28], the formation of α' martensite was so far believed to be inhibited in DSS 2205. However, we demonstrate that this is possible with a fine grain size of $\sim 500 \text{ nm}$ and deformation twinning, and attributed this to the increased stability of refined austenite [26,63]. Such increased austenite stability can be associated with the difficulties in simultaneously achieving several martensite variants within one austenite grain due to limited room, which is detrimental for strain energy minimization via the martensitic transformation, making it energetically unfavourable [64].

In the current study, such unique and complex deformation behaviour can also be related to the SFE of the austenite. The SFE of austenite in a commercial DSS 2205 sheet is reported to be around 10 mJm^{-2} with bulk Mn content of 1.72 wt% and austenite N content of 0.22 wt% [65]. Changes in compositions of individual phases will affect their SFE, where the reduction of Mn [25] and/or addition of N [27] are reported to significantly increase the SFE of austenite. With a lower Mn and higher austenite N content (Table 1) present in the austenite of the current LPBF+HT DSS 2205, the SFE of its austenite is expected to be much higher than 10 mJm^{-2} . A model that correlates the SFE of austenitic steels and their average electron vacancy number (NLE) suggests that the SFE of austenite in the current DSS 2205 must be between $18 - 22 \text{ mJm}^{-2}$ with a calculated NLE of around 3 using the austenite composition from Table 1 and Eq. 1 (compositions in wt%) [65]:

$$\langle \text{NLE} \rangle = [0.61\text{Ni} + 4.66(\text{Cr} + \text{Mo}) + 2.66\text{Fe} + 3.66\text{Mn} + 6.66\text{Si}] / 100 \quad (1)$$

This resides near the upper limit for a martensitic transformation and the lower limit for deformation twinning, where twinning is found to occur at SFE of roughly $12 - 35 \text{ mJm}^{-2}$ and martensite transformed from

austenite is favoured at SFEs $<18 \text{ mJm}^{-2}$ [61,66]. With such SFE, it is possible that most dislocations structures in the austenite appear as extended dislocations, i.e., each full dislocation breaks up into two partial dislocations connected by a SF, enhancing the strengthening effect.

For a given SFE, a smaller grain size usually suppresses the nucleation of twins and favours dislocation slip in FCC alloys when the grain size is above 100 nm [67–69]. However, a high density of deformation twins is observed here, and this is beneficial for strengthening. The secondary deformation nanotwins could have formed via defective twin boundaries involving inherent kinks on a set of primary twin boundaries, similar to what has been reported for a 304 stainless steel [70]. It should be noted that, in conventional manufacturing routes, such high density of deformation nano-twinning is usually only achievable via complex processing involving severe plastic deformation such as high pressure torsion and surface mechanical attrition treatments [71], which is more onerous and less technologically attractive compared to a short heat treatment after LPBF presented here. Grain refinement is also reported to increase the austenite stability [63] and, thus, inhibit martensitic transformations [26]. However, the deformation-induced martensitic transformation observed here could be related to the presence of abundant deformation twins, which enables twin boundary intersections with dislocations, serving as nucleation sites for martensite. Alternatively, the martensitic transformation could have happened via dislocation and/or SFs multiplications. This is opposed to nucleation from intersections of shear bands of ϵ -martensite in coarse-grained materials [72,73]. Similar observations have been reported in metastable austenitic stainless steels [41]. The formation of ϵ -martensite is associated with plastic accommodation and dislocation debris in the neighbouring austenite [74], while internal grain boundaries are rarely present within austenite phase in the current LPBF+HT DSS 2205, limiting the number of austenite-austenite boundaries and hence ϵ -martensite nucleation sites. The increase in length of grain boundaries due to finer grain size and the formation of sub-grain boundaries also provides extra nucleation sites for martensite, as they are believed to be one of the most favourable nucleation sites for martensitic transformations [41,75].

While the non-equilibrium microstructures including un-balanced phase fractions and defects have previously been regarded as a major challenge in metal AM of DSS, we believe that their defects such as dislocation sub-structures and precipitates can be harnessed to serve as nucleation sites for solid-state phase transformation during subsequent thermal treatments. The austenite phase that develops possesses a finer grain size beneficial for strengthening, and introduces deformation nano-twinning during deformation, instrumental for sustained plastic flow. This unlocks unprecedented mechanical properties that are unavailable through traditional manufacturing routes.

5. Conclusions

In the present work, tensile testing and correlative electron microscopy using SEM, EBSD and advanced TEM have provided new fundamental understanding of the attractive microstructure-property relationships in DSS 2205 processed by LPBF. The complex deformation mechanisms in ferrite and austenite that facilitate the excellent strength-ductility combination in a post-LPBF heat-treated (LPBF+HT) DSS 2205 are revealed and discussed in detail. This knowledge explains the significant ductility improvement of LPBF DSS 2205 after short heat treatments, and the balanced property profile compared to the as-LPBF condition and wrought counterparts. The main conclusions are drawn below:

- (1) The LPBF+HT DSS 2205 exhibits an excellent strength ($\sim 625 \text{ MPa}$ yield strength and $\sim 790 \text{ MPa}$ tensile strength) and ductility ($\sim 18\%$ uniform ductility and $\sim 27.5\%$ fracture ductility) combination, with increased yield strength and ultimate tensile

strength compared to its wrought counterpart, while reaching similar fracture ductility when compared to DSS 2205 previously fabricated by AM.

- (2) The microstructural defects resulting from the non-equilibrium nature of the LPBF processing and subsequent solid-state phase transformations achieve significant austenite grain refinement. The stacking fault energy of the austenite obtained plays an important role in achieving the optimal combination of mechanical properties in the LPBF+HT DSS 2205.
- (3) The better strength is due to contributions from the hard ferrite phase, fine austenite grain size, deformation twins in the austenite, and nanoscale oxide dispersions, while the outstanding ductility is the result of the dissolution of Cr_2N and a complex combination of multiple deformation mechanisms to accommodate plastic flow and provide sustained work hardening.
- (4) The deformation-induced martensitic transformation of austenite in the LPBF+HT DSS 2205 occurs alongside deformation twinning but without the presence of ϵ -martensite, while the dense network of mechanical twins and stacking faults exhibits a hierarchical nature of deformation, with length scales spanning from micron- to nano-dimensions.

We discuss how the level of microstructural complexity and solid-state phase transformation resulted from LPBF and heat treatment can be harnessed to unlock multiple strengthening routes during deformation. This fundamental understanding is crucial for guiding alloy design of AM parts with reproducible and optimised combinations of mechanical properties, for applications in the marine infrastructure, oil & gas, and paper & pulp industries.

CRedit authorship contribution statement

H. Wang: Data curation, Formal analysis, Writing – review & editing. **X.Z. Liao:** Conceptualization, Funding acquisition, Writing – review & editing. **S.P. Ringer:** Conceptualization, Funding acquisition, Writing – review & editing. **N. Haghdadi:** Conceptualization, Funding acquisition, Methodology, Supervision, Writing – review & editing. **S. Primig:** Conceptualization, Funding acquisition, Methodology, Supervision, Writing – review & editing. **X.Y. He:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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