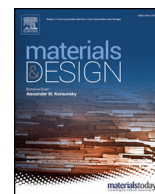




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The role of microstructure on wear mechanisms and anisotropy of additively manufactured 316L stainless steel in dry sliding

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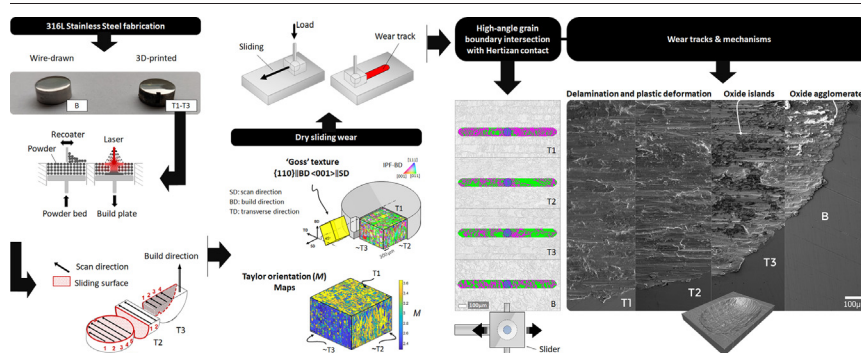
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HIGHLIGHTS

- Wear mechanisms in laser powder-bed-fusion 316L stainless steel (L-PBF 316L SS) can be effectively altered by tailoring the microstructure to control the wear performance.
- Despite L-PBF surface containing a degree of porosity, its wear performance can be the same as another L-PBF surface with no apparent porosity due to dissimilar wear mechanisms.
- Due to the unconventional microstructure of L-PBF 316L SS, it cannot be generalized that wear in L-PBF is 'better' or 'worse' than conventional 316L SS over a wide load spectrum.

GRAPHICAL ABSTRACT



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ABSTRACT

Wear control, which relies on understanding the mechanisms of wear, is crucial in preserving the life of mechanical components and reducing costs. Additive manufacturing (AM) techniques can produce parts with tailored microstructure, however, little has been done to understand how this impacts the mechanisms of wear. Here we study the impact of initial grain arrangement and crystal orientation on the wear mechanisms of austenitic stainless steel (SS) in dry sliding contact. Specifically, the anisotropic sliding wear behavior of as-built, AM-ed 316L SS is compared against annealed, wire-drawn counterparts. We describe, in-detail, how the sliding wear mechanisms of delamination, abrasion, oxidation, and plastic deformation are attributed to the initial surface microstructure under different loading conditions using a number of techniques. This new understanding sheds light on how different AM-induced microstructures affect wear, thereby allowing for better utilization of this technology to develop components with enhanced wear properties.

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1. Introduction

Through decades of research into the friction, lubrication, and wear of bodies in relative motion – the field known as *Tribology* [1], scientists and engineers were able to develop wear-resistant components that enhanced our quality of life across a multitude of

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industries [2–8]. This was achieved thanks to the understanding of the common *mechanisms* by which wear occurs, such as *Adhesion* [9], *Delamination* [10], *Abrasion* [11], and *Corrosion* [12] (Fig. 1 a–d, respectively). The adoption of additive manufacturing (AM) technologies brings new opportunities for manufacturing parts with complex geometries and unconventional microstructure, and thus properties [13]. By altering the process parameters of AM, different microstructure configuration can be induced which results in further variation of those properties. However, understanding the role of microstructure of AM-ed components on the mechanisms of wear remains unexplained. Such understanding is especially important in industries such as automotive, aerospace, and medical implants where both complex part-geometry and superior wear-resistance are desired.

To single out the role of microstructure on wear is challenging, because many contributing variables in the system's design need to be accounted for (load, speed, hardness ratio, contact geometry, temperature, etc.). For instance, in sliding wear of Face-Centered Cubic (FCC)

metals, the evolution of the microstructure (e.g., twinning, lattice rotation, grain refinement) is closely connected with the initial surface and sub-surface microstructure [14]. However, majority of the tribological studies pertaining to AM are concerned with quantifying the wear (and friction) performance of AM-ed alloys in comparison to those traditionally manufactured [15–22]. Hence, many of the aforementioned variables (including microstructure) were changed in order to enhance and quantify the wear performance. And so, a mere review of literature is not sufficient to understand the specific role of microstructure on wear mechanisms of AM-ed components. For this reason, a mechanistic approach accounting for as many variables as possible should help to better understand its role. Such understanding should aid in developing AM-ed alloys with superior wear-resistance without the need for additional strengthening processes.

Here we investigate the role of microstructure on the sliding wear of 316L stainless steel (SS), a workhorse material which comprises of a single-phase austenitic FCC crystal structure. Our specimens were additively manufactured via laser powder-bed-fusion (L-PBF) which uses a

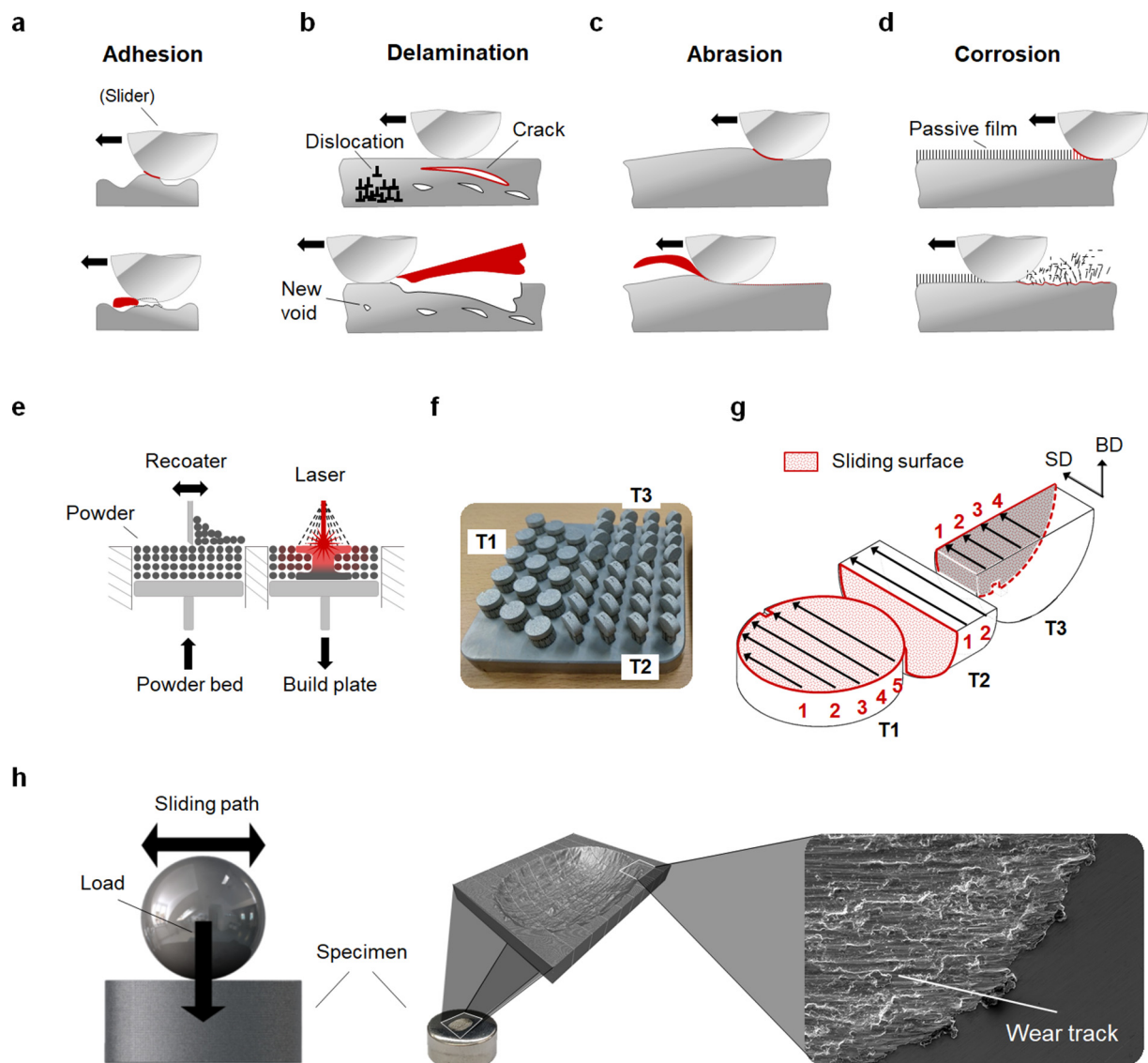


Fig. 1. Common wear mechanisms (a), L-PBF fabrication process (e–g), and tribological set-up (h). a, Adhesive wear where material is transferred from one surface to another. b, Delamination wear where dislocation pile-up creates voids and subsequent cracks that ultimately result in delamination of material as thin sheets. c, Abrasive wear where one surface abrades another's. d, Corrosive wear where sliding breaks the passive film exposing it for corrosive environments. e, The laser powder-bed-fusion (L-PBF) process. f, Actual image of L-PBF specimens, T1, T2, and T3 printed from 316L Stainless Steel (SS). g, The L-PBF specimens' sliding surface planes with respect to the build- and scan-directions (BD and SD respectively). h, The tribometer dry sliding set-up, computed- and SEM visualization of the wear track after the specimen was worn.

high-density laser to melt and fuse metal powder particles layer-by-layer (Fig. 1e,f). The reference coordinates for L-PBF consists of three perpendicular axes, the scan direction (SD), the build direction (BD), and the transverse direction (TD). This process yields grains with columnar microstructure and crystal orientation dependent on the laser's path [23], resulting in anisotropic mechanical behavior. To investigate this behavior, we fabricated three types of L-PBF specimens: T1, T2, and T3, whose sliding surfaces were parallel to a unique reference plane – Namely, T1 ~ SD-TD, T2 ~ SD-BD, and T3 ~ TD-BD (Fig. 1 g). This was done by varying the build orientation for each specimen type while maintaining the same scan strategy. In addition, we used a reference material made from bulk wire-drawn process labelled B. The reference material was heat-treated, while the L-PBF specimens were not (in order to retain the original microstructure generated in AM).

We explored the sliding wear behavior of those specimens using a linear reciprocating tribometer equipped with a ball made of 316L SS. The reason why 316L SS was used as a counter surface, is to reduce interference from differences relating to material composition and hardness (cf. our work with this reference [20]). We generated a profile of wear rate in the normal load range 0.98–4.9 N in a 30-min sliding test (Fig. 1 h). To understand the role of microstructure on the wear mechanisms and anisotropy, we used white-light interferometry (WLI), scanning electron microscopy (SEM), energy-dispersive x-ray (EDS), and electron-backscatter diffraction (EBSD). While this study does not offer a recipe for achieving optimal wear resistance in L-PBF, it provides a thorough investigation into the impact on wear anisotropy pertaining to the initial microstructure of the sliding surface.

2. Methods

2.1. Sample fabrication and preparation

Forty-seven disc specimens (3.5 mm diameter x 10 mm height) were fabricated from gas atomized 316L SS powder (composition in Table 1) using L-PBF 3D printer Concept Mlab cusing (ConceptLaser, Germany) which utilizes a continuous fibre-laser with a beam spot size of ~50 µm. The specimens were printed using laser power of 90 W, scanning speed of 600 mm/s, hatch spacing of 77 µm, and layer thickness of 20 µm, in argon atmosphere. The specimens were printed in a single batch with a unidirectional scanning strategy with no rotation between subsequent layers. The only varying parameter was the build orientation, which was varied at 90° angles to produce the specimens shown in Fig. 1 f. The corresponding L-PBF specimen groups shown in the figure were labelled T1, T2, and T3. In addition, sixteen-disc specimens were fabricated from annealed wire-drawn 316L SS rod (West York Steel, composition in Table 1), hereafter referred to as B specimens. The top flat surface (indicated by a notch) for the specimens was polished to a mirror finish by an external service provider (Watson Metal Polishing, UK) to a surface roughness (R_a) < 800 nm ($\leq N6$ as per ISO 1302:1992 Geometrical Product Specification (GPS)). The surface roughness was measured using WLI (NT9100, VEECO) following a 15-min sonication bath of toluene and subsequent overnight soak in isopropanol bath to remove any polishing suspension or loose particulates.

2.2. EBSD characterization

The SD-TD, SD-BD, TD-BD planes of the L-PBF specimens and specimen B were characterized using EBSD. These then were metallurgically prepared using abrasive paper and polishing cloth to a final step where a 0.04 µm non-crystallizing OPUS colloidal silica was used (MetPrep, UK). The characterization was performed in a Hitachi S-3400 variable pressure SEM equipped with e-Flash¹⁰⁰⁰ EBSD detector (Bruker, USA). Specimens were tilted 70° inside the chamber, with accelerating voltage of 25 keV, an EBSD map size of ~1.5 mm × 1.2 mm, and a step size of 2.0 µm. Subsequently, the microstructural and textural analysis was performed using an open-source MATLAB toolbox MTEX [24]. The segmentation angle chosen for the high-angle grain boundaries (HAGBs) was 15°. Minor processing was employed where grains with less than 10 pixels were removed and grains were smoothed using a Laplacian smoothing with an iteration rate of 2. The inverse-pole figure (IPF) maps were plotted with respect to the build direction (IPF-BD) in all specimens. The grain size was determined based on the *diameter* function in MTEX.

2.3. Taylor factor calculation

Taylor factor was calculated using MTEX's *calcTaylor* function assuming plane strain with symmetrical flow (i.e., $q = 0.5$) with uniaxial loading perpendicular to the sliding surface. Minor processing was employed where grains with less than 10 pixels were removed and grains were smoothed using a Laplacian smoothing with an iteration rate of 2.

2.4. Hertzian contact and HAGB intersections calculations

The radius, a , for the Hertzian contact was estimated based on eq. (1) for elastic contact of a sphere loaded on a flat surface.

$$a = \left(\frac{3}{2} \frac{rN}{E^*} \right)^{1/3} \quad (1)$$

$$\frac{1}{E^*} = \left(\frac{(1-\nu_1)^2}{E_1} + \frac{(1-\nu_2)^2}{E_2} \right) \quad (\text{Where,})$$

r is the radius of the sphere in millimeters, N is the applied normal load in Newtons, E_1 , E_2 are the elastic moduli in Mega-Pascals of the specimen and ball respectively, ν_1 , ν_2 are the poisson ratios of the specimen and ball respectively. r was set to 3 mm, E_1 , E_2 we assumed to be 220,000 MPa for 316L SS, ν_1 , ν_2 were assumed to be 0.27 for 316L SS.

To find the HAGB intersections for the Hertzian contact, we used MTEX's *boundary.intersect* function inside a loop spanning the profile of the Hertzian contact area over an arbitrary distance (stroke length) of 1000 µm.

2.5. Hardness measurement

Following the EBSD characterization, Vickers hardness (HV) measurements were taken using a universal hardness tester ZHU 187.5 (Zwick, Germany). The load force and hold time were 3 kgf and

Table 1

Composition of L-PBF powder and wire-drawn specimens. Reported values are provided by suppliers. The L-PBF powder was gas atomized. The wire-drawn specimen was annealed for 4 h at 1060 °C and water quenched.

	(wt%)												
	Fe	Cr	Ni	Mo	C	Mn	P	S	Si	Cu	N	Co	O
L-PBF	Bal.	17.7	12.6	2.38	0.015	0.72	0.009	0.004	0.60	0.02	0.09	ND	0.03
Wire-drawn	Bal.	17.20	11.05	2.03	0.19	1.60	0.033	0.028	0.58	0.40	0.022	0.15	ND

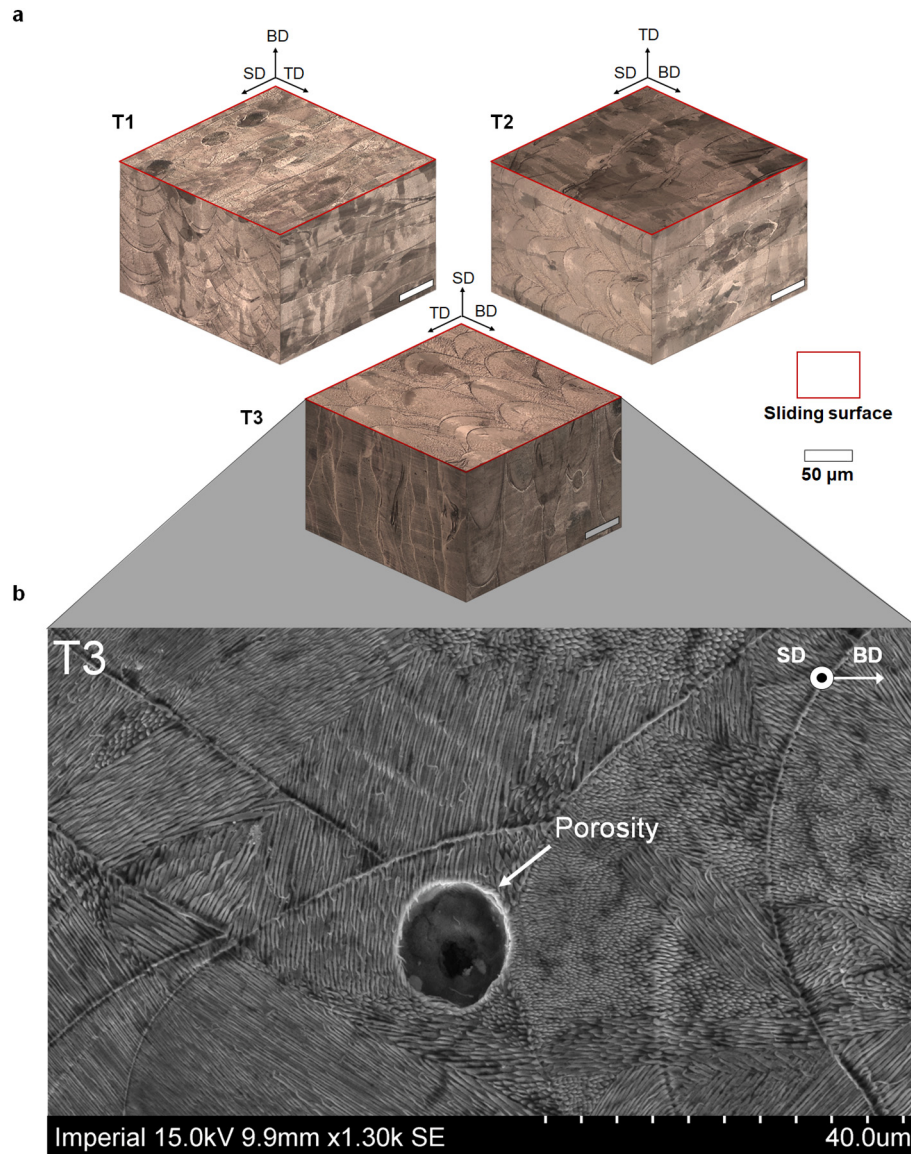


Fig. 2. Etched sections of L-PBF specimens T1, T2 and T3 highlighting the melt pools and sliding surface and cellular domains. **a**, Surfaces etched (5% oxalic acid for 90 s) to reveal the microstructure and melt pools. **b**, Cellular domains aligned to various directions.

10 s, respectively (HV3/10). For each specimen, a total of six measurements were performed and averaged. Their standard errors were reported.

2.6. Wear test, measurement, and analysis

To conduct the wear tests, specimens were loaded in a linear reciprocating sliding tribometer HFRR (PCS Instruments, UK). The counter surface used was a 316L SS ball (6 mm diameter, obtained from Bearing Wearhouse, UK). Each test was conducted at ambient temperature in dry conditions with one of the following normal loads applied: 0.98, 1.96, 2.94, 3.92, 4.9 N. In any trial, the ball slid back and forth for 30 min, with a stroke length of 1.5 mm, and a frequency of 50 Hz. For each load, two to three trials were performed from which the standard error bars were calculated and reported. (Note: We also include friction readings, in Supplementary Fig. 1, which may be helpful for other studies though not directly related to the objectives of this work). Each specimen was cleaned in a sonicated isopropanol bath for 15 min to remove any loose debris and contaminants.

We used a motorized WLI, Wyko NT9100 (VEECO, USA) to measure the wear track volume. A full 3D map of the wear track was constructed by stitching multiple smaller 3D maps in different regions encompassing the whole view of the wear track. The 3D map was subsequently post-processed using Vision64 (Bruker, USA) for signal noise removal and realignment of images. The wear volume was calculated based on the total displaced volume from the surface of the specimen (the displaced volume for two selected datasets can be seen in Supplementary Fig. 2). Eq. (2) was used to measure the specific wear rate (in mm^3/Nm) where the total wear volume was divided by the applied normal load and total sliding distance.

$$\text{Wear rate} = \frac{\text{Volume } \text{mm}^3}{\text{Load } N \times \text{Total Distance } m} \quad (2)$$

Specimens of each type (T1, T2, T3, and B) covering the full load spectrum (0.98–4.9 N) were investigated using a combination of SEM and Inca x-act EDS (Oxford Instruments, UK). The accelerating voltage for SEM was varied between 15 and 25 keV depending on the features being investigated. The EDS elemental mapping was performed over a

large rectangular area approximately in the center of the wear track using the same magnification for all inspected specimens such that a comparison would be possible. The EDS accelerating voltage and spot size were 20 keV and 80, respectively.

3. Results and discussion

3.1. Characterization of microstructure

The melt pools of T1, T2 and T3 are shown in Fig. 2a. As they all were fabricated by the same print parameters and scan strategy (only the build orientation was varied), the 3D morphology of grain microstructure in the three conditions should statistically be the same. Thus, we examined the grain morphology and crystallographic texture shown on the three orthogonal planes SD-TD, SD-BD, TD-BD of the same specimen (i.e., T1) using EBSD. We show IPF maps along the BD of the grain orientation in Fig. 3a. The growth of the crystal is governed by the local heat flux [25–28]. For FCC crystals (including the currently studied alloy), $\langle 001 \rangle$ is a preferred growth direction of solidification cells (or dendrites). Due to high speed laser beam, the global heat flux is closely aligned with SD, making one of the $\langle 001 \rangle$ orientations parallel to the SD [29,30]. Although the crystallographic $\langle 001 \rangle$ of cells is preferably aligned with the global heat flux, the cell arrangement is less well aligned to global heat flux due to the local change of heat flux, side-branching of cells, and the high symmetry of FCC [25]. For example, Fig. 2b shows multiple cellular domains aligned to various directions. The cell spacing was found to be about 0.5 μm to 1 μm . Side-branching of crystals

between adjacent deposition tracks broadens grains straddling between pairs of adjacent tracks. Such grains (colored green, Fig. 3a) have the other two $\langle 001 \rangle$ directions aligned about 45° to the BD, in other words, a $\langle 011 \rangle$ direction oriented towards the BD [25], equivalent to a Goss (G) texture. This was also demonstrated using contour pole figures shown in Fig. 4. In contrast to the $\langle 011 \rangle \parallel \text{BD}$ texture, the continuous growth without change cell direction along the centerline of melt pools promotes a set of slender grains with all the $\langle 001 \rangle$ being aligned with BD, SD, and TD (equivalent to a Cube texture component, see Fig. 5). Because side-branching of cells/dendrites is very dominant due to frequent changes in heat flux across the boundary between two melt pools, the Goss-equivalent texture is the strongest component, highlighted in top part of Fig. 3a (specimen T1).

The initial texture plays an important role in the elasto-plastic anisotropy of FCC polycrystalline materials. For common FCC textures (Brass, Copper, Cube, Goss, and S), an initial Goss texture contributes the most to plastic anisotropy, while an initial Cube texture contributes the least [31]. It is known that the stiffest $\langle 111 \rangle$ direction can exhibit up to 2.94 times the Young's modulus of the least stiff $\langle 001 \rangle$ direction in austenitic SS FCC crystals [32]. Thus, altering the microstructure by varying the scan strategy or build orientation [30,33–35] can effectively change magnitude of elasto-plastic anisotropy.

In the bottom part of Fig. 3a we show a schematic diagram of specimen T1 highlighting both its G texture and sliding path from a top-view angle (namely, direction of the applied normal load). It can be seen that the normal load (N) is perpendicular to the SD in the case of T1 and T2. However, the sliding path was parallel to SD in T1, while perpendicular

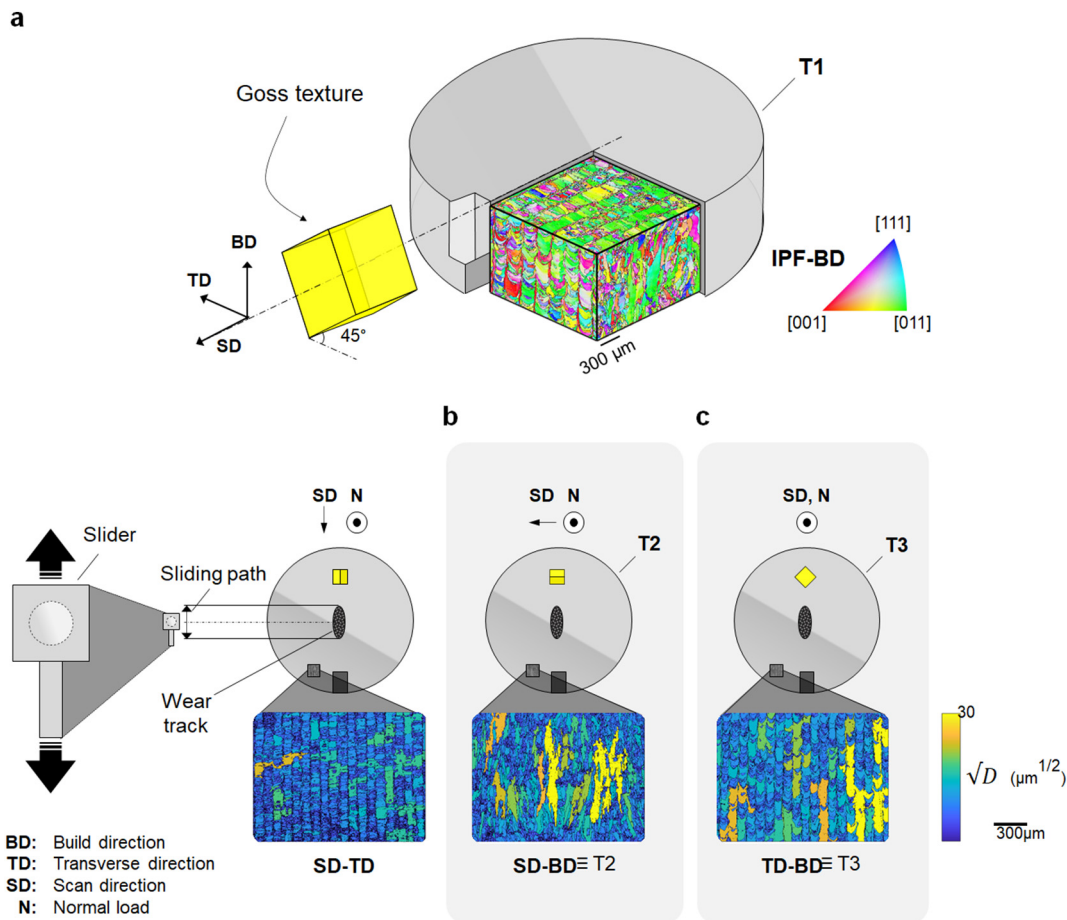


Fig. 3. The components' dominant texture, loading direction, and sliding path. **a.** Inverse-pole figure (IPF) maps in the build direction (BD) taken from three sections of specimen T1. The IPF map of the SD-TD, SD-BD, and TD-BD show a strong Goss (G) texture, namely, $\{110\}\langle 001 \rangle$. Specimen T1 is shown in the bottom highlighting the SD, direction of the normal load (N), and sliding path. **b,c.** Comparing the SD, N, and sliding path of T2 and T3 (respectively) to T1. Below each disc, we present EBSD maps highlighting grain morphology color-mapped by the grain diameter ($\sqrt{D}$).

to SD in T2 (cf. Fig. 3a, Fig. 3b). In T3, on the other hand, N was parallel to SD and both were perpendicular to the sliding path (Fig. 3c). This means that in the case of T1, N is applied to a G texture. In T2, N is applied to a rotated G (RT-G) texture. While in T3, N is applied to a rotated cube (RT-C) texture. A reference condition of the material that was annealed (specimen B) on the other hand, exhibited a mixed texture of $\langle 111 \rangle$ and $\langle 001 \rangle$ parallel to the drawing direction (DD) (Fig. 4), a texture that is typically observed in wire-drawn FCC materials [36]. However, due to the annealing process, its texture was weak when compared to any of the L-PBF specimens as highlighted by the color bars on Fig. 4.

The grain size is known to strongly affect the yield strength (σ_y) of the material according to the Hall-Petch relationship [37,38]. Accounting for the dislocation density and crystal orientation, σ_y can be rewritten as eq. (3) [39,40].

$$\sigma_y = \sigma_0 + M\alpha\mu b\sqrt{\rho} + \frac{k_y}{\sqrt{D}} \quad (3)$$

Where, σ_0 is the frictional stress, ρ is the dislocation density, M is the Taylor factor depending on the relationship between the loading direction with respect to the crystallographic orientations, α is the constant accounting the interactions of dislocations in different slip systems, μ is the shear modulus, b is the Burgers' vector magnitude, k_y is the Hall-Petch coefficient, and D is the average grain diameter. Because all specimens used in this study are of the nominally same material, it is reasonable to assume σ_0 and k_y are the same for all the annealed and L-PBF specimens. Thus, a material with larger grain size generally tends to have lower yield strength (hence the hardness) and vice-versa. The quantification of grain

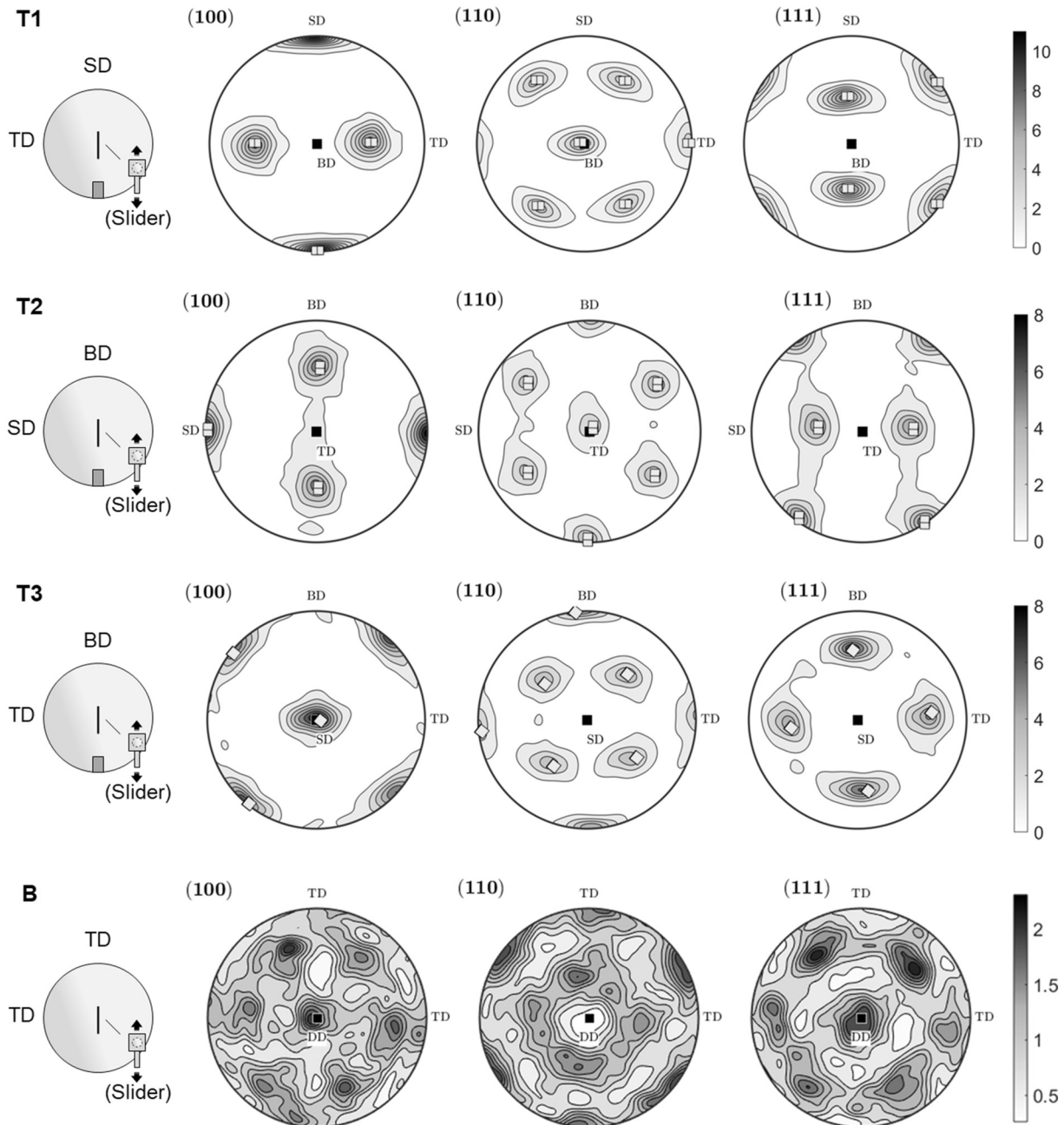


Fig. 4. Pole-figure diagrams showing the dominant Goss texture in L-PBF specimens T1-T3 (denoted by cube shape) and the texture of specimen B. It is clear that the texture of specimen B is much weaker and more homogenous than L-PBF specimens due to the annealing process.

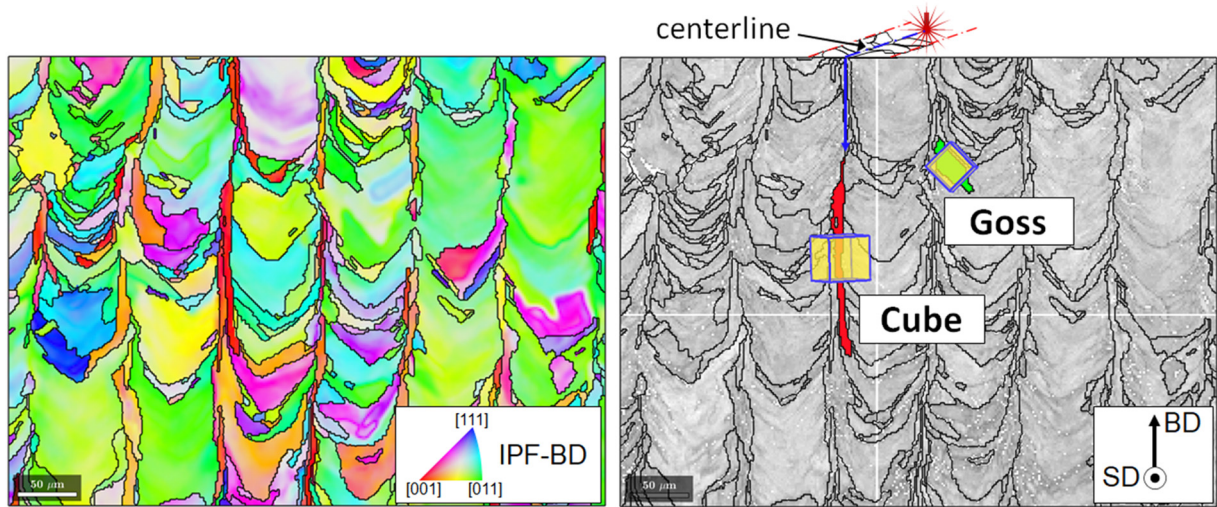


Fig. 5. Inverse-pole figure (IPF) in the build direction (BD) highlighting the slender $\langle 001 \rangle$ components. The $\langle 001 \rangle$ component parallel to the BD results in a weak cubic texture and is located along the centerline of the melt pool.

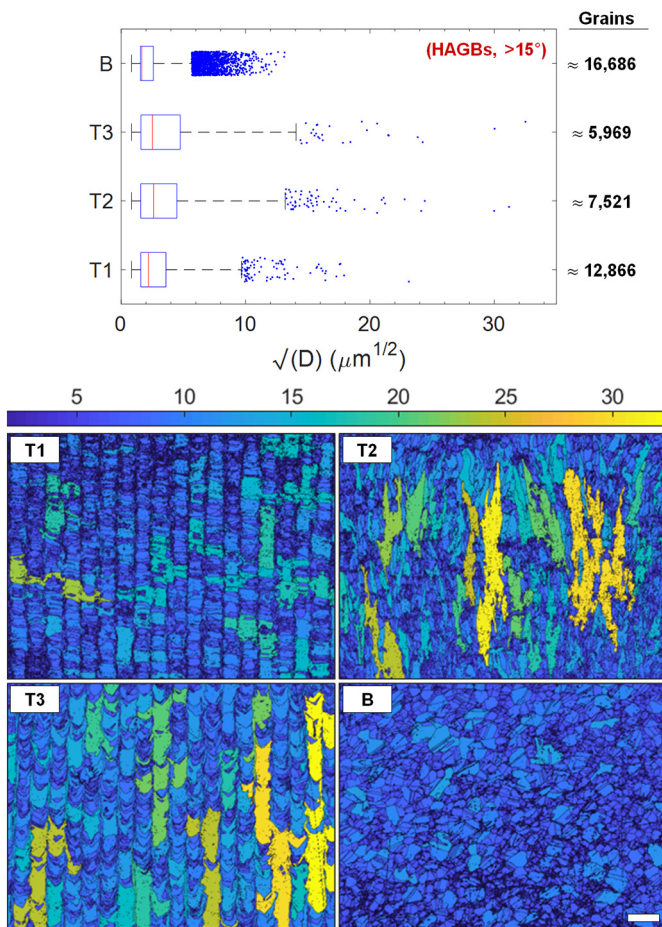


Fig. 6. Comparison of microstructure using box-plots of high-angle grain boundaries (HAGBs, $>15^\circ$) of all specimen types. Box-plots depicting the variability of L-PBF average $\sqrt{D}$ compared with the wire-drawn specimen. The number of grains is also shown on the right-hand side, where B has the highest number of grain counts per unit area, followed by T1 (SD-TD), T2 (SD-BD), then T3 (TD-BD). In the bottom we highlight a visual comparison between the microstructures color-mapped using $\sqrt{D}$ (scale bar = $150 \mu\text{m}$) (EBSD area for each specimen = 1.8 mm^2).

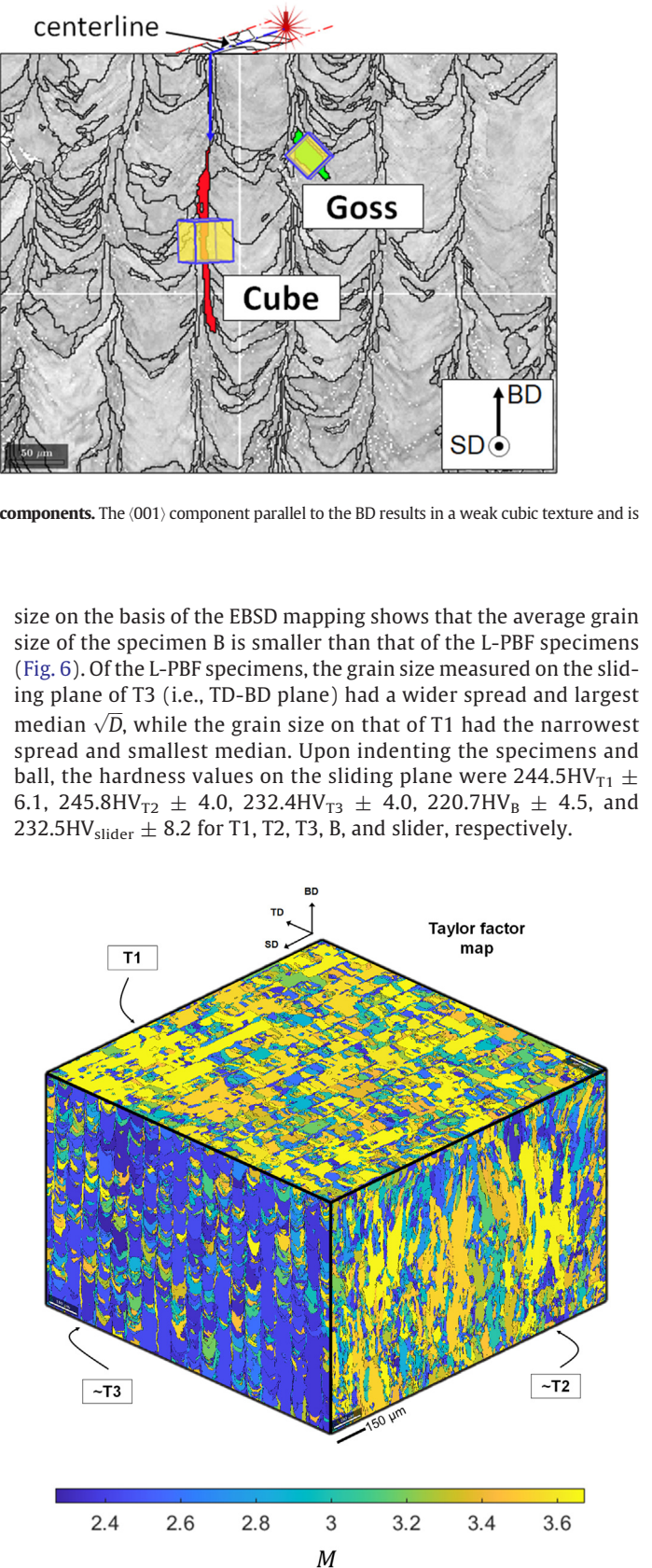


Fig. 7. The computed Taylor factor (M) for each L-PBF orthogonal plane. Upon loading perpendicular to the any of the planes shown, it can be seen that T3 (i.e., TD-BD plane) has the lowest M values. (EBSD area for each specimen = 1.8 mm^2).

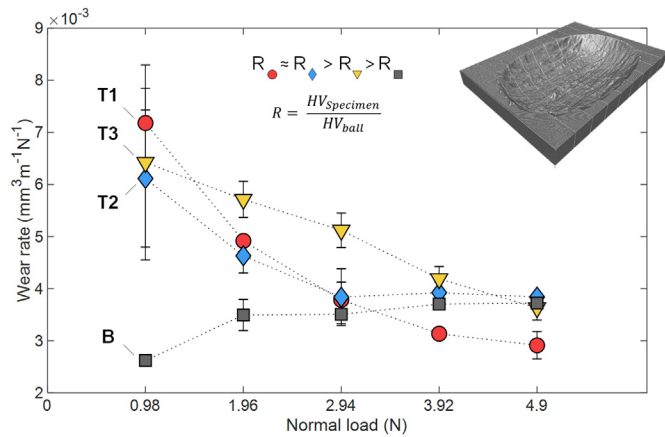


Fig. 8. Wear rates under different loading conditions, corresponding relative hardness ratios of specimen-to-ball (R). The plot shows that wear rate decreases with increased load for the L-PBF specimens (T1–T3) due to work hardening, unlike the wire-drawn one (B). The measured specimen-to-ball hardness ratios (R) were: $R_{T1} \approx R_{T2} \approx 1.05 > R_{T3} \approx 0.99 > R_B \approx 0.94$. Error bars correspond to standard error.

Because of the large grain dimension and low Taylor factor on the TD-BD section, namely, T3's surface (Fig. 7), indentation on this section should give lowest hardness compared to indentation on the other two sections. This partly explains why the hardness of T3 is significantly lower than those of T1 and T2. The reason why T3's hardness was greater than the hardness of B, despite B's smaller grains, is because high cooling rates and thermal cycles in L-PBF generated fine cellular structures which contain high densities of dislocations at cell boundaries [13], making T3 harder than B.

From a tribological perspective, hardness measurements of specimen and slider can be used as a rough indicator of the wear resistance [41] as experimental evidence showed a critical hardness ratio ($R = HV_{specimen} / HV_{slider}$) that results in severe wear in the range 0.8–1.0; though it is believed that the actual critical value is closer to 0.8 [42]. The corresponding hardness ratios were: $R_{T1} \approx R_{T2} \approx 1.05 > R_{T3} \approx 0.99 > R_B \approx 0.94$.

3.2. Wear performance

The wear rates were determined using WLI based on the total displaced volume (Fig. 8a). The wear rate of the annealed condition (specimen B) was rather stable with normal load increments (except in the first load). On the other hand, the wear rate of the L-PBF specimens decreased as the normal load increased. Combined SEM-EDS investigation of the wear tracks revealed that wear mechanisms varied for certain specimen types and also when different loads were used. Fig. 9 shows a side-by-side comparison of the wear tracks at the end of the 30-min sliding wear run under a load of 4.9 N. The figure reveals that the wear mechanisms were indeed different. For instance, agglomeration of oxides is seen on specimen B, while oxide islands – a situation where oxidation occurs more uniformly [43] – were observed in T3 (compare B and T3 in Fig. 9). On the other hand, T1 and T2 were dominated by plastic deformation and delamination mechanisms (a detailed look on the wear track is available in Supplementary Vid.1).

3.3. Porosity-induced fracture

Porosity is a common issue in additively manufactured components (Fig. 2b) which is not only known for compromising the mechanical integrity of the components, but also for accelerating the wear rates [15,44,45]. Unlike the other specimens, fracture chips as large as 200 μm were observed on T3's wear tracks (Fig. 10a). These fractures were typically observed irrespective of the applied normal load magnitudes. We ascribe the presence of such large fractures to the pre-existing porosity, in particular lack of fusion (LoF) pores beneath T3's sliding surface (note: those LoF pores also visible on some T3 specimens outside the wear track, see Fig. 10b). When the ball slid over these porosities, stress concentrations induced by sharp corners of pores propagated quickly which led to the formation of such large fracture chips.

It is known that scan strategy can alter the heat transfer, which affects the melting and solidification of the alloy. The density and distribution of porosity are affected by the implemented build orientation and scan strategy [46]. The build orientation of T3 made the sliding surface (viz., a TD-BD plane) parallel to the plane consisting of the end points

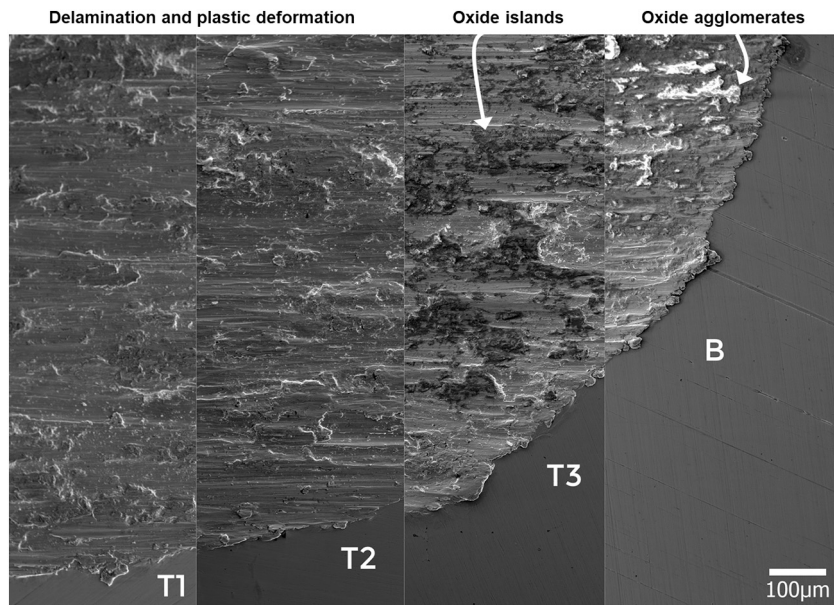


Fig. 9. Wear track comparison between all specimens using scanning electron microscopy (SEM). T1 and T2 show wear by delamination and plastic deformation. On the other hand, T3 shows traces of oxide islands (a thin layer of oxides). Finally, specimen B shows traces of agglomerated oxides (hard oxides that cause surface abrasion). The micrographs shown correspond to an applied normal load of 4.9 N. Detailed look into the wear track of each individual specimen can be seen on Supplementary Vid.1.

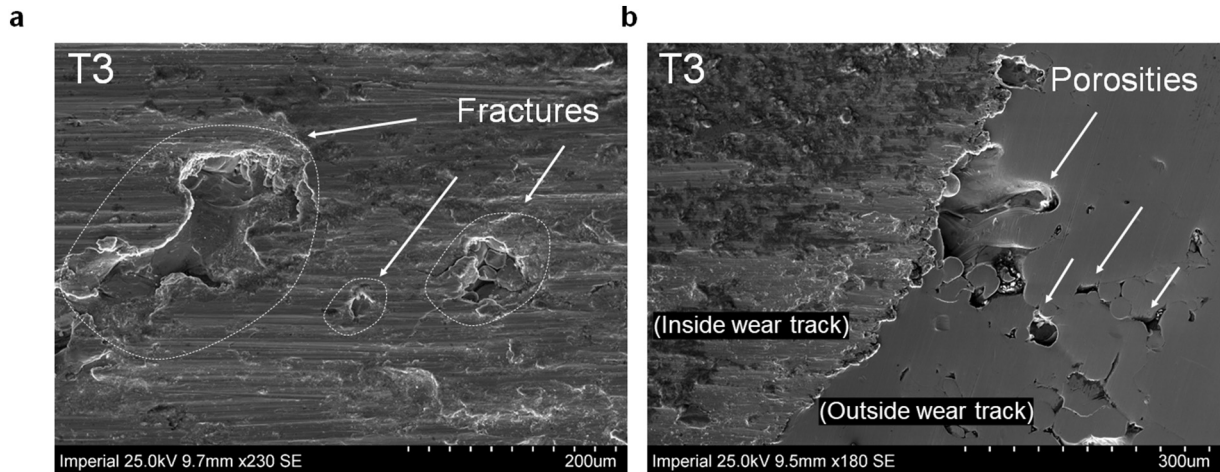


Fig. 10. Lack of fusion (LoF) pores assisting subsequent fractures during sliding. **a**, Fractures caused by presence of LoF pores inside the wear track. **b**, Evidence of LoF pores on unworn surface. Both micrographs were taken of specimen T3 under a normal load of 2.94 N.

of laser path (Fig. 11). Sudden changes in laser energy density occur at the end of laser path (i.e., a drop in energy density due to the switch off of laser in this study), causing more porosity on the beginning and end of scan lines [47,48]. In other words, more pores were located beneath the sliding surface in T3 when compared with the other L-PBF specimens.

The subsequent fractures seen in Fig. 10b may have contributed to the wear rate behavior of T3. While we observed a decline in wear rate with increased load for all three L-PBF specimens (Fig. 8a), T3 showed the slowest rate of decline (in terms of trend) amongst them. Thus, we suggest that the accelerated wear caused by the fracture chips contributed to the observed trend in the wear rate of T3. Note however, that at the greatest load of 4.9 N, T3 and T2 showed the same wear rate, hinting at an isotropic behavior. This was interesting as T3's surface had higher porosity content compared to T2. It may be that the oxide islands in T3 helped alleviate the wear rate as that is a commonly observed phenomenon. Interestingly, in contrast to T2 and T3, T1's wear rate for the same load was lower than both T2 and T3.

Since wear is affected by many parameters (load, speed, sliding distance, material, geometry, temperature, etc.), certain wear mechanisms can emerge/disappear when one or more of these parameters is changed. Most importantly, the total wear is the collective result of all individual wear mechanisms present. Thus, although porosity does impose a negative impact on the wear rate [44], the other wear mechanisms associated with the underlying microstructure significantly contribute to the observed wear rates.

3.4. Abrasive-oxidative wear

Thanks to the coherent passive film due to the high content of Chromium, 316L SS offers excellent corrosion resistance. If this film is depleted or scraped off by abrasion, the metal's exposed surface can oxidize by reacting with atmospheric air. One instance of abrasion can result when asperities of the ball shear off asperities of the surface underneath it. Typically, that happens when the two materials' asperities interlock during the sliding motion. Though, this depends on the hardness ratio of the two materials (R) and on the applied normal load. With that in mind, it is useful to map out areas where oxygen is present as an indicator of abrasive-oxidative wear.

All specimens showed evidence abrasive-oxidative wear under SEM-EDS investigation, however, the magnitudes varied from specimen-to-specimen and from load-to-load, as seen in Fig. 12. Despite being fabricated from the same material, the oxidation behavior is different for each type of specimen. The sub-grain boundaries and

dislocation density can stimulate the generation of the coherent passive film [49,50] which reduces the susceptibility for oxidative wear. To explain, the boundaries of the cellular structure, in the sub-grain region of as-built L-PBF 316L SS, are typically enriched with Chromium (Cr) and Molybdenum (Mo). These densely present micro-galvanic couples (nobler than Fe) serve as nucleation sites for accelerated growth of the passive protective film in those enriched regions in the sub-grain. This reason was attributed by Kong. et al. [51] for the low passive current density in as-built L-PBF 316L SS when compared with either annealed L-PBF 316L or wrought 316L SS. While statistically T1, T2, and T3 should be quite similar, the tribological surface upon which the normal load is applied is not (in terms of density of surface dislocations, density of cellular structures, and Taylor factor). Furthermore, the presence of porosity in L-PBF specimen has been correlated with an increase in the susceptibility to corrosive attacks [52]. This may very well have contributed to the gradual increase in oxidative wear that was observed in T3, due to the dense presence of porosities. In the case of specimen B, the thickness of its protective film should be lower than the (as-built) L-PBF specimens due to the annealing process [49], which makes the material susceptible to corrosive attacks. We thus expect dissimilar oxidative wear behavior amongst the specimens we studied.

In general, the oxygen content in the wear track of B was the highest of all and it increased with increased load. Conspicuously, with

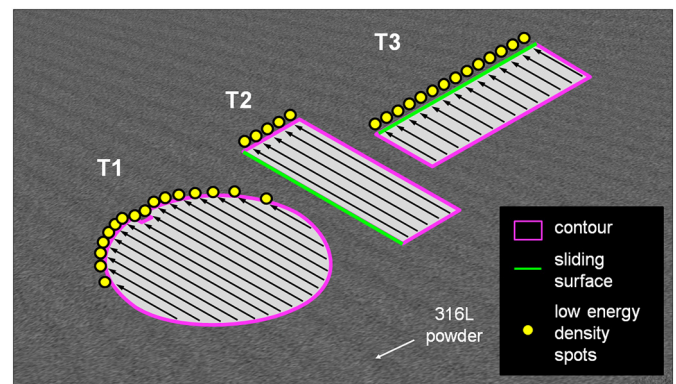


Fig. 11. The laser scan strategy of the L-PBF specimens with respect to the sliding surfaces. An arbitrary single layer of the L-PBF build is shown to highlight the contour scan in addition to the sliding surface. In the case of T3, the end and beginning of the laser scans are perpendicular to the sliding surface (i.e., where sudden energy density drop occurs resulting in higher porosity content). Note that the sliding surface of T1 (first from left) is only visible once the final layer has been deposited.

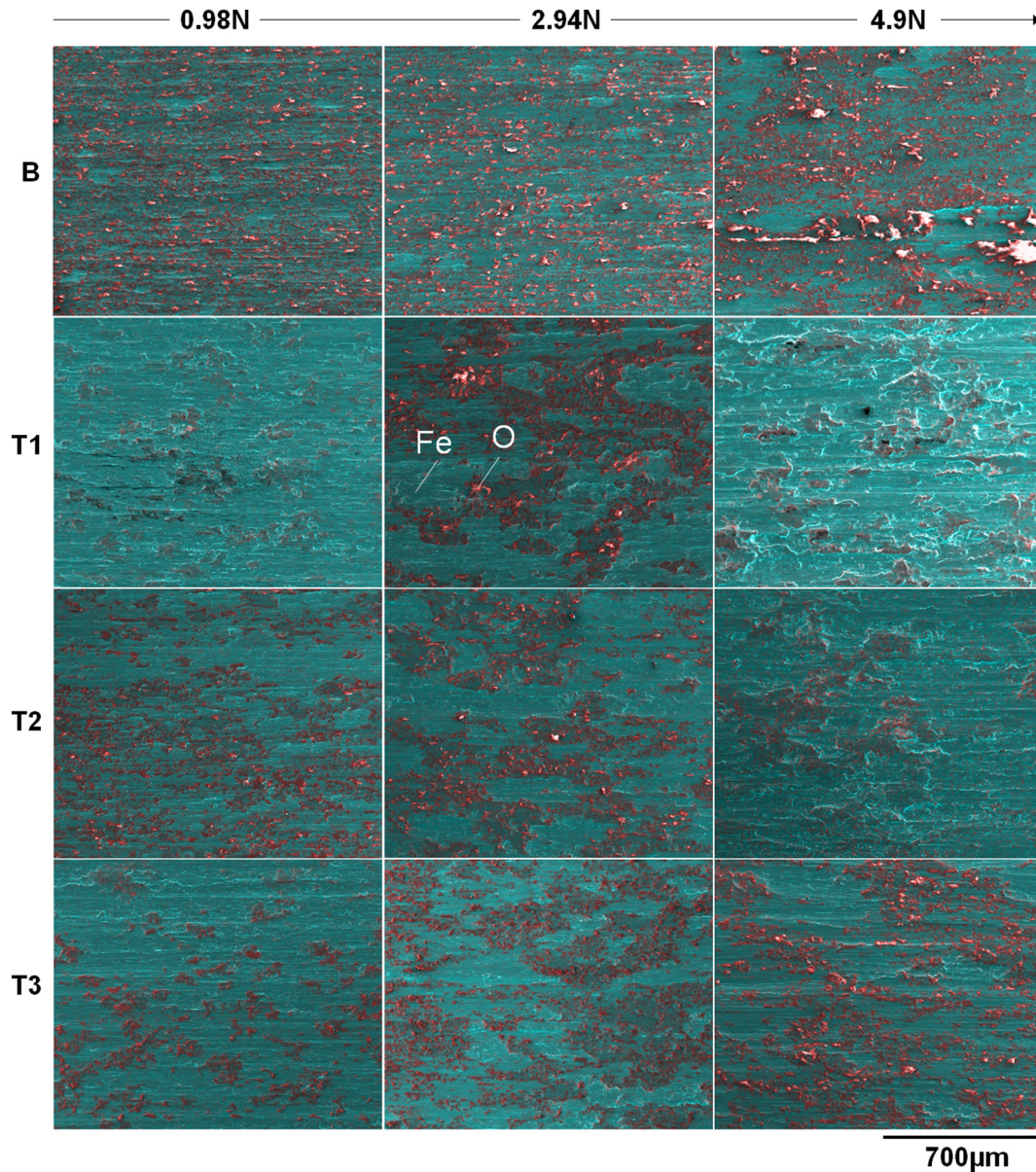


Fig. 12. Energy dispersive x-ray (EDS) maps of iron and oxygen content in wear track of specimens for different normal loads. Specimen B shows the highest impact by oxidative wear, which seemed to worsen with increased load. Followed by specimen T3. Specimens T1 and T2 showed an increase in oxidative with increased normal load, but the oxygen content diminished under 4.9 N. The RGB color map was based on Red: oxygen, Green: iron, Blue: iron. The map was taken in the center of the wear track for all specimens.

increased load these oxides coalesced into agglomerates which became larger in the case of specimen B (Fig. 12). We verified that these (bright) agglomerates were indeed oxides (Fig. 13). As displayed in the figure, these agglomerates were sometimes found inside deep confined grooves, likely indicating that they were hard enough to abrade the specimen's surface creating such grooves. In other instances, agglomerates were formed but not inside grooves. Contrarily, oxides in T3 were scattered across the surface and only started to agglomerate towards the maximum load of 4.9 N. T1 and T2 showed an interesting behavior where the oxides present were highest in the middle normal load (2.94 N) with little agglomerates and diminished substantially at the maximum load. Though in the set of specimens that we selected for analysis via EDS, we were able to observe a trend in which B suffered the most oxidation followed by T3. While it is expected that B would be most susceptible to oxidative wear, (as shown by Kong et al. [51]), this may also be linked to the hardness ratio; as R_B was the smallest followed by R_{T3} (i.e., of all specimens, B possesses the least abrasion

resistance which promotes the even more film removal and subsequent corrosion by oxidation). Similarly, T3's low R may have contributed to its consistent oxidation trend for different loads when compared with T1 and T2. However, we note that even in the same specimen, the level of oxidation may vary from the bottom layers of the specimen towards the top surface due to oxygen content in the build-up process. Even for the L-PBF specimens, the oxidation wear may be different based on the density of sub-grain cellular structures and dislocation density at the sliding surface. In addition, oxidation may vary significantly from location to location, and also due to uncontrollable changes in ambient temperature and/or humidity, all of which must be accommodated for in a controlled experiment.

3.5. Delamination wear and plastic anisotropy

The observed plastic deformation and delamination wear mechanism in T1 and T2 is best explained using the delamination wear theory

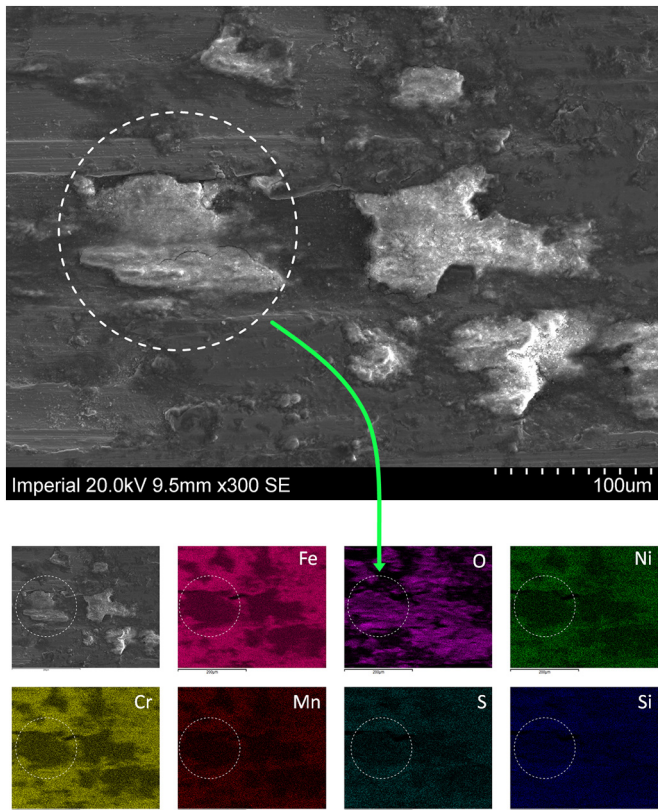


Fig. 13. EDS map confirming the bright bodies consisted of high-oxygen content. These oxide agglomerates were hard enough to further abrade the surface underneath it creating deep grooves. Micrograph corresponds to wear track of specimen B subject to a normal load of 4.9 N.

[10] (note that the delamination does not refer to the delamination between deposited layers in AM specimens). Unlike the widely accepted adhesive wear theory [9], the delamination theory is based on the mechanisms of ductile fracture due to plastic deformation in sub-surface regions during sliding. To understand the plasticity in sub-surface regions, we need to estimate the evolution of yield strength that can be described as a function of microstructure (such as crystallographic orientation, dislocations and grain size) according to the Taylor forest hardening Eq. [53] and Hall-Petch relationship, namely eq. (3). Apart from the variations in M discussed previously, it is also likely that ρ would be different between T1, T2, and T3 due to the different build directions; The laser path, cross section of a build and the height of the build affect the thermal gradient cooling rate and cell orientation/arrangement, hence the dislocation density. It should be noted that because cell boundaries consist of dense dislocations, the cell arrangement contribution to the wear behavior is accounted for via the dislocation density distribution in eq. (3). In addition, wear behavior is an average over a volume (e.g., more than 2000 μm in width, 1000 μm in depth and 3000 μm in length for the 4.9 N load) much larger than cell spacing (0.5 μm to 1 μm). Considering that solidification cells were not well arranged (Fig. 2b), the contribution of cell arrangement to the difference in wear behavior between T1, T2 and T3 must be much smaller than that of the crystallographic orientation and grain boundaries. Nevertheless, the contribution of cell arrangement is increasingly significant with reducing the wear load as the wear volume decreases. This explains why the arrangement of solidification cells increasingly affects the wear behavior with reducing the wear load as recently reported by Yang et al. [20].

It would also be useful to visualize the Hertzian contact area as the ball comes into contact with the sliding surface at the initial sliding stroke because dislocations tend to pile up at the HAGBs during sliding and loading. We show the Hertzian contact area (radius $\sim 45.29 \mu\text{m}$) for an applied normal load of 4.9 N over a sliding distance of 1000 μm in Fig. 14. It can be seen that the number of HAGB intersections in T1 is significantly greater than any other specimen. As such, it is expected to harden at a faster rate than T2 and T3 (due to significant pile-up of dislocations at the HAGBs during sliding). While that is true however, as dislocations pile-up at a finite distance away from the sliding surface, this eventually leads to the formation of voids that combine to form a crack parallel to the sliding surface [10,54]. Once this crack reaches a critical length, material will then ‘delaminate’ in the form of thin-sheets [10] (i.e., wear debris). Despite that, the specific role of HAGBs in crack formation is a complex problem and beyond the scope of this project. On the other hand, the number of HAGB intersections for the same Hertzian contact area is much less in T2, and therefore, its work hardening rate occurs at a slower rate than T1. For the same reason, T3’s work hardening rate should be slower than T2.

The representative SEM micrograph of T1 in Fig. 14 shows material removal in the form of small chips (circled craters with sharp angles). In T2, voids and cracks formation occur on a much smaller scale, possibly due to the pile-up of dislocation at the low angle grain boundaries (LAGBs), which are also known for piling-up dislocations [55]. Despite specimen B also having a large number of HAGB intersections for the same Hertzian contact, the representative SEM micrograph shows that abrasive-oxidative wear is prominent. This is because, initial density of dislocations in specimen B is lowest as it underwent an annealing process. Therefore, this material condition is least susceptible to cracking due to dislocation pile up, explaining its constant wear rate despite the loading increments. On the other hand, the initial dislocation density in L-PBF 316L is significantly higher than that in annealed condition [56].

The wear debris is either ejected from the sliding contact or mechanically mixed forming what is known as a *tribo-layer* [57]. This layer can subsequently increase or decrease the wear rate, depending on its hardness which sometimes can be greater than that of its parent material [5]. In this work, the negative impact the tribo-layer had was observed when these oxides agglomerated and created deeper grooves (as seen in Fig. 12) which only took place when abrasive-oxidative wear was dominant. However, in other cases such as T1 and T2, it seems that the tribo-layer was soft enough that it plastically deformed into finer debris. It is unclear whether or not this may have contributed to the wear rate, however, it did not seem to exacerbate it.

4. Conclusion

In this study, we focused on the effect of microstructure (grain morphology and crystal texture) on the wear mechanisms in dry sliding of as-built additively manufactured 316L stainless steel compared against specimens made from annealed wire-drawing process. The sliding surface of the additively manufactured specimens, T1, T2, and T3 were parallel to the SD-TD, SD-BD, and TD-BD planes, respectively. The wear rate was calculated in the range of 0.98–4.9 N. The results can be summarized as follows:

- As the load increased from 0.98 N to 4.9 N, the as-built additively manufactured specimens exhibited a declining trend in wear rate due to the accelerated work-hardening effect, while the trend in the annealed wire-drawn specimen was mostly stable.
- The large 2D-grains and low Taylor factors (characterized by loading upon a Cube texture) on T3’s sliding surface contributed to the abrasive-oxidative wear seen when compared with T1 and T2. Furthermore, we attributed fractures that were larger than 200 μm , to the lack-of-fusion porosities. These porosity-induced fractures were

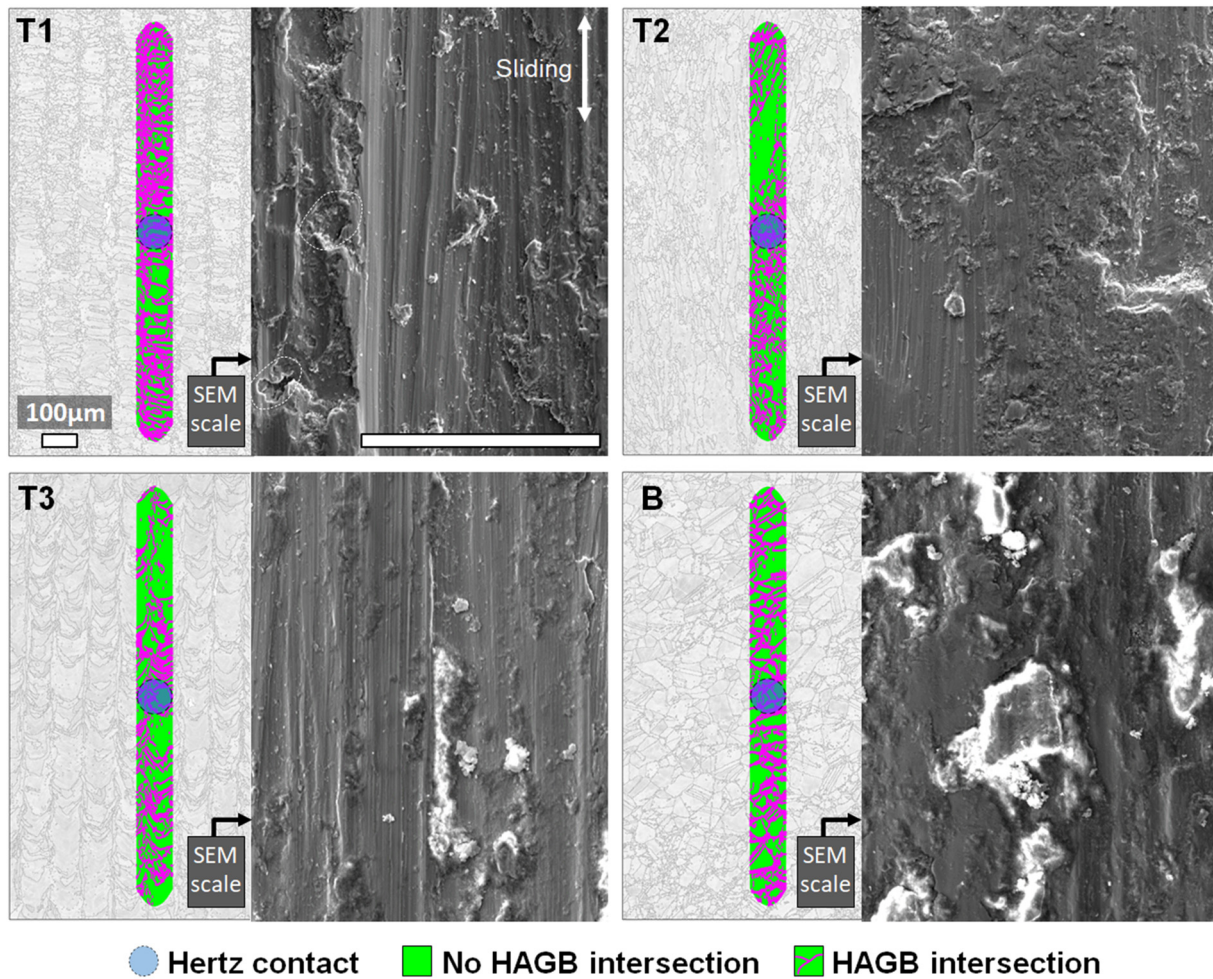


Fig. 14. The density of high-angle grain boundary (HAGB) intersections with the Hertzian contact during sliding. T1 showed the greatest density of HAGB intersections with the Hertzian contact during sliding, followed by B. The difference in HAGB intersections between T2 and T3 is not easily distinguishable. The micrograph of T1 shows evidence of delaminated grains as a result of cracks shown on the figure. In T2, delamination occurs after severe plastic deformation where cracks are much smaller than in T1. Both T3 and B show evidence of oxidation, however, the oxidation is more severe in specimen B. The Hertzian contact has a radius of 45.29 μm and correspond to an applied normal load of 4.9 N. An arbitrary sliding distance of 1000 μm was chosen such that it can fit within the margins of the EBSD map.

specific to T3 and were ascribed to the significant drop in laser energy density. However, despite the presence of porosities on T3's sliding surface, its wear rate at 4.9 N was surprisingly equal to the T2 which did not show any apparent porosities. This means that porosity alone is not the final determiner of the wear rate in AM-ed components.

- The annealed wire-drawn specimens exhibited the lowest hardness (as expected). However, their wear mechanisms were mostly characterized by abrasive-oxidative wear with large oxide agglomerates that were frequently observed inside deep grooves. The susceptibility to oxidative wear in these specimens was attributed to the annealing process.
- The greatest hardness was equally found in T1 and T2 sliding surfaces, both of which exhibited high Taylor factors (characterized by loading on Goss and rotated-Goss textures, respectively). As the load increased to 4.9 N, the wear track in both types of specimens was characterized by delamination and plastic deformation.
- Despite the equal hardness of T1 and T2, the number of high-angle grain boundary intersections with the Hertzian contact in T1 was significantly higher than that of T2 (or any other specimen). This was correlated with T1's accelerated work-hardening rate as the load

increased, resulting in a better wear performance at the highest load of 4.9 N compared to all other specimens.

5. Outlook

The versatility of additive manufacturing techniques to produce novel bearing surfaces with tailored microstructure is promising. This can be achieved by varying the build orientation and/or the scan strategy to alter the grain arrangement and the crystal texture of the tribological surface [15]. Even for surfaces with a degree of porosity, microstructure can have knock-on effects capable of alleviating the wear rate (cf. T3 and T2 at 4.9 N). For this reason, it cannot be simply stated that the wear resistance of L-PBF components is better or worse than a conventional one, as this not only depends on porosity, but also on the microstructure and its response to external conditions (cf. T1 and B). Our work demonstrates that wear mechanisms can be controlled based on initial microstructure of the surface, provided that the desired microstructure of the tribological surface is carefully considered in the design phase of the component and the work hardening effects are taken into consideration. This work encourages a multi-

disciplinary research in the field of metallurgy, additive manufacturing, and tribology to produce bearing surface with excellent wear properties.

Author contributions

C.W.M., T.R., and M.B. designed the experiment. M.B. led the project, conducted the experiments, and wrote the manuscript. M.S.P. provided a substantial revision to the work. All co-authors contributed to the data analysis and discussion.

Declaration of competing interests

The authors declare no competing financial interests nor personal relationships that could have appeared to influence the work.

Data availability

The raw is available to download from <http://dx.doi.org/10.17632/xd3xrxggtk.1>. The EBSD raw data cannot be shared at this time as the data also forms part of an ongoing study.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.matdes.2020.109076>.

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