

**Additive manufacturing:
Towards alloys with spatially defined tribological
properties**

by

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Abstract

The tribological properties of metals produced by additive manufacturing (AM) techniques are not well understood. Recent literature has produced contradictory findings on the wear and friction performance of AM steel compared to conventionally manufactured steel. Beyond tribology, scientists and engineers observed that AM produces alloys with unconventional microstructures, in turn giving rise to unconventional properties. Furthermore, they observed that the resultant microstructure is a direct function of the AM process parameters. In this thesis, a bottom-up, experimental approach is used to uncover the complex role the AM-induced microstructure plays in the tribological response of steel (first aim of the thesis). Several tribological set-ups (reciprocating sliding, in-situ/ex-situ SEM micro-scratching, macro-scratching) and imaging techniques (SE, BSE, ECCI, EDS, EBSD, white-light interferometry) revealed a great deal of anisotropic tribological behavior ('tribological anisotropy') ensues from the microstructure. Dislocation density and arrangement, low-angle grain boundaries, high-angle grain boundaries, crystallographic orientation all contribute to the tribological anisotropy. Based on this knowledge, the AM process parameters were controlled to produce steel tribosurface with spatially defined microstructure design (referred to herein as 'multigrade tribosurface'). These multigrade tribosurfaces in turn exhibit multigrade tribological properties (second aim of the thesis) not achievable via conventional manufacturing routes. In particular, it was revealed that the Taylor factor (accounting for orientational stiffness of the underlying crystal) correlates with the coefficient of friction during sliding over a wide load range. Furthermore, it was revealed that the presence of strong crystallographic texture, or lack thereof, correlates with the susceptibility to generating wear debris. The results in this thesis, by and large, explain the tribological nuances of AM alloys and demonstrate the AM technology's potential to furthering the field of tribology.

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Statement of originality

I, the author of this thesis, declare that the work herein is produced by my own effort and writing at the time of my PhD project at Imperial College London. Where the work was not my own, I made explicit references citing the work and/or ideas of others. Published parts or manuscripts ensuing from this work were explicitly mentioned at the title page of each applicable chapter.

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Dedication

To my family, parents, wife, and son.

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Acronyms

ρ dislocation density.

$\perp$ single dislocation.

τ_o shear stress to move a dislocation in the absence of dislocations.

σ_o frictional stresses.

τ_{flow} flow shear stress.

σ_y yield strength.

A fully austenitic solidification mode.

AF austenitic-ferritic solidification mode.

A^* effective Hertzian contact area.

Cr_{eq} chromium equivalent.

Cr_{eq}/Ni_{eq} equivalent chromium-to-nickel ratio.

D grain size.

E elastic modulus.

E_l linear energy density.

E_v volumetric energy density.

F fully ferritic solidification mode.

FA ferritic-austenitic solidification mode.

HV Vickers hardness.

M Taylor orientation factor.

N normal load.

Ni_{eq} nickel equivalent.

P_o applied pressure.

SD_{total} total sliding distance.

V_{total} total wear volume.

W^* minimum conformal scratch width.

W_{rate} wear rate.

d_t transition depth.

h hatch spacing/distance.

k' shear flow equation constant.

k_y Hall-Petch coefficient.

r radius.

t hatch spacing.

ν poisson's ratio.

w_{total} total scratch width.

AM additive manufacturing.

BD building direction.

CoF coefficient of friction.

EBSD electron backscatter diffraction.

FCC face-centered cubic.

GND geometrically necessary dislocations.

HAGB high-angle grain boundary.

LAGB low-angle grain boundary.

P laser power.

SD scanning direction.

SLM selective laser melting.

TD transverse direction.

V laser scanning speed.

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Chapter 1

Introductory chapter

1.1 Problem statement and research aims

The advent of metal additive manufacturing (AM) technologies has enabled various industries to tap into new performance domains. These domains were unlocked by AM's ability to produce parts with complex geometries thereby reducing weight and simultaneously maintaining the structural integrity. Considering the rapid incorporation of AM components across several critical industries (e.g., aerospace, automotive, and healthcare), tribologists need to examine the tribological properties of these components. To date, tribological properties of AM metals remain poorly understood. Several studies (detailed in 2.4) reported contradictory conclusions on the wear performance of AM metals compared to conventionally manufactured metals. A similar issue concerning friction is also found in literature.

One definite feature of an AM metal is its unusual microstructure when compared with a conventionally manufactured metal of the same kind. In fact, the resultant microstructure is a function of the exact process parameters selected by the operator of the AM-machine at the time of fabrication. The mechanical properties, including the tribological ones, are known to be affected by the underlying microstructure. Therefore, it is logical that a relationship between the microstructure and tribological properties of an AM metal must first be established. After doing so, we may find it possible to induce certain tribological properties by microstructure control. Indeed, the thesis further aims to show that producing single-object steel with spatially defined tribological properties (i.e., a multigrade tribosurface) is viable. The author hopes the results in this work would provide indispensable insights to researchers and practitioners in the field of additive manufacturing and tribology.

1.2 Thesis structure

Chapter 1 introduces the problem and the research aims. Chapter 2 provides necessary background information and overview on the literature to justify the research. Chapter 3 specifies the experimental methods and design for the entire project. Chapters 4-6 are the results chapters. In particular, Chapter 4 offers experimental evidence on the anisotropic wear mechanisms arising from the microstructure. Chapter 5 ties those findings with a visual examination through in-situ tribological experiments. Finally, Chapter 6 exploits the true potential of additive manufacturing in controlling the microstructure to induce site-specific tribological properties. Chapter 7 offers concluding remarks and recommendations for future work.

Chapter 2

Background and literature review

2.1 Chapter introduction

Centuries of metallurgical experience has taught us that the properties of an alloy are a function of the underlying microstructure's constituents (e.g., grain size, chemical composition, etc.). These properties are exhibited in the form of mechanical, electrical, chemical, and magnetic performance of the alloy. We translated this knowledge into calculable steps of alloying, cold/hot working, and heat-treating to achieve a predictable microstructure aimed at certain properties. However, these efforts have also introduced us to a dilemma known as the strength-ductility trade-off, a natural phenomenon which stipulates that strengthening of an alloy must be accompanied by a drop in useful ductility, and vice-versa [1]. Quite remarkably, it was recently discovered by Wang et al. [2] that metals produced by selective laser melting (SLM) – a type of metal additive manufacturing (AM) techniques – can simultaneously exhibit strength and ductility, therefore allowing them to break off this trade-off curve. Wang et al. attributed this quality to a heterogeneous hierarchical microstructure consisting of high-density dislocations, sub-cellular structures, solute segregation, and low-angle grain boundaries. This unconventional microstructure, which gives rise to such unconventional material properties, is not readily available in alloys manufactured via conventional routes.

The unconventional microstructure of the SLM-produced components is linked with the inherently unconventional manufacturing process that SLM employs. Unlike conventional manufacturing routes, SLM manufactures components by directing a high-energy beam onto a bed of metallic powder (see Figure 2.1). As the beam passes through select regions of the powder, these regions instantaneously melt and solidify. In fact, Liu et al. [3] reported that the cooling occurs at a rate of 10^3 - 10^8 °C/s compared with traditional quenching which occurs at a rate of 10^2 °C/s. Such rapid cooling results in an unconventional microstructure (and therefore unconventional material properties) which is unattainable via traditional manufacturing routes. In addition, the local heat flux induced by the laser can influence the so-called crystallographic texture (covered in section 2.2.1) which bears impact on the anisotropy of the produced part. Apart from light-weighting and freedom of design, the aforementioned attributes play a significant role in motivating SLM-based research across various fields today.

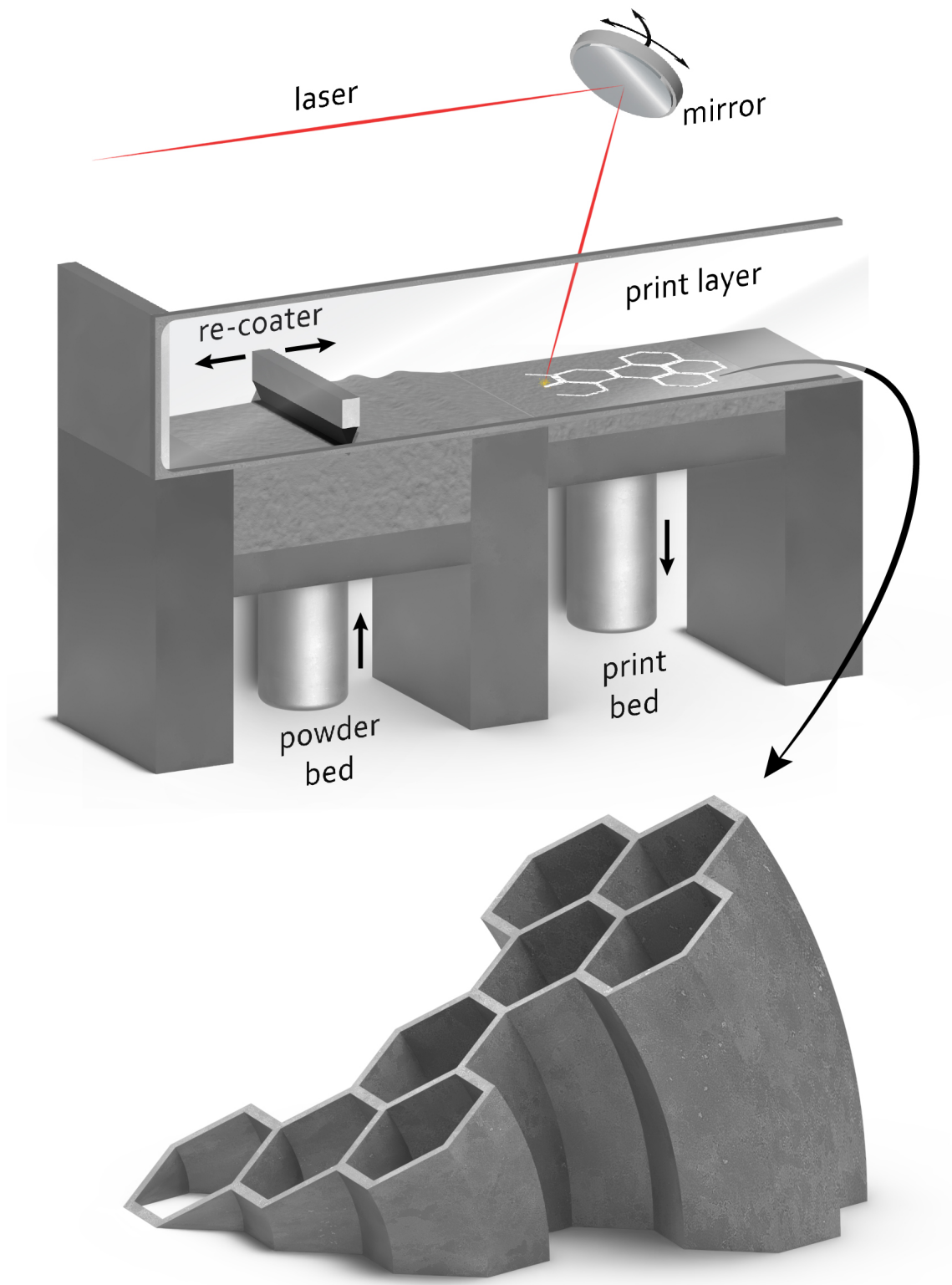


Figure 2.1: Additive manufacturing via laser powder-bed-fusion technology, also commonly referred to as selective laser melting.

From a tribology perspective, studies concerning metal AM components have emerged just over a decade ago [4]. However, research output in this area has been rather limited and focused mostly on comparing the tribological performance of SLM- against conventionally manufactured alloys, with little insight into the underlying mechanisms. In those comparisons, it was generally believed that SLM components offers superior wear resistance, due to ‘finer microstructure’ [5]. However, this notion became quite hard to justify in light of following studies that observed quite the opposite result [6]. Quite naturally, these discrepancies diminish our confidence in utilizing these new materials, especially in critical applications where component failure can lead to devastating consequences (e.g., aerospace, rail, medical implants, etc.). Therefore, it is imperative that the nuances which give rise to these performance discrepancies are understood before any wide-spread tribological implementation of SLM components can be achieved.

The following sections of this chapter will cover the necessary background and literature review to ensure the essential concepts are unambiguous to the reader and to set the stage for the work in this thesis, respectively. Note that the remainder of this chapter is best read in the sequence presented by the author. If the reader prefers to read sections selectively, they may find it helpful to go back to earlier sections in case they find certain concepts or terminologies ambiguous.

2.2 Microstructure and properties of polycrystalline materials

2.2.1 Overview of the microstructure constituents

Polycrystalline materials comprise the vast majority of inorganic solids in existence. The term ‘poly-’ denotes the presence of ‘many’ crystals (i.e., grains). A single grain composes of atoms arranged in a periodic order in the 3D space, which conveniently can be defined by a single unit cell. To fully define a unit cell, we must know its six lattice parameters (i.e., the lengths and angles of and between their perpendicular axes: a , b , c , α , β , and γ), in addition to the composition and position of atoms along its structure (Figure 2.2).

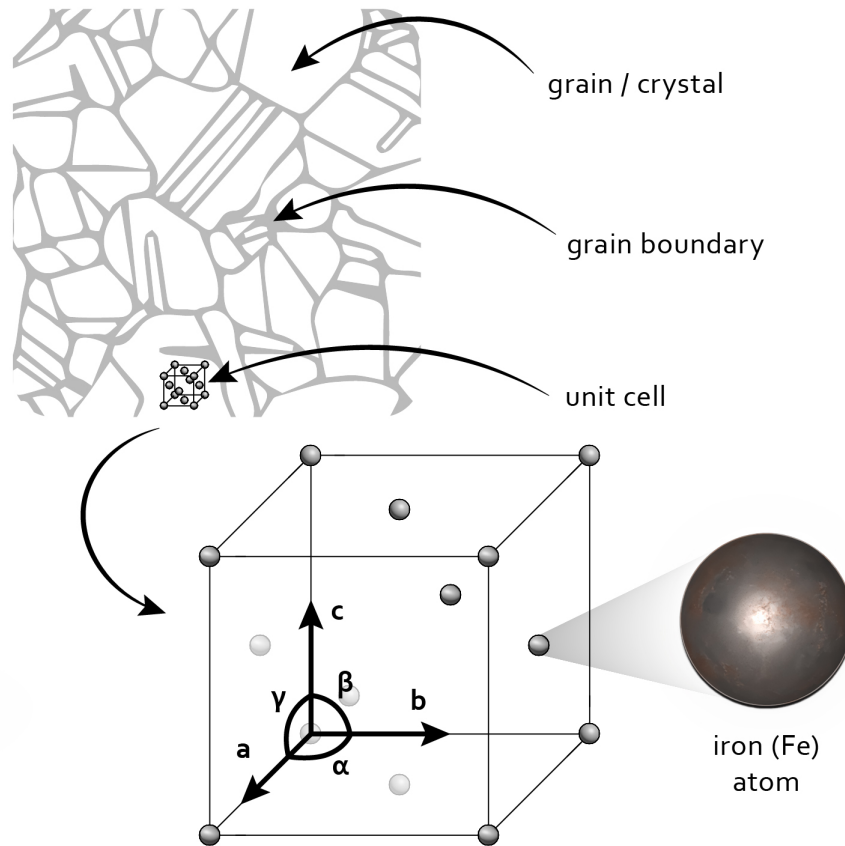


Figure 2.2: Grains/crystals are separated by grain boundaries. Within each grain boundary lies a characteristic unit cell which is defined based on its chemical composition, atomic arrangement, and lattice parameters a , b , c , α , β , and γ .

A variation in the lattice parameters results in a change in the crystallographic structure, whereas a variation in the position of atoms results in a change of the so-called Bravais lattice. Table 2.1 shows seven crystal structures which in total possess 14 Bravais lattices. The simplest of all is the simple cubic structure shown in Figure 2.3a. In the cubic unit cell, all of its axes' lengths are identical and the angles between them are equally set to 90° . In this case where the Bravais lattice is 'simple', all the atoms exist only on the corners of the cube. By comparison, the body-centered form of the cubic structure has its atoms positioned on the corners as well as one in the body center Figure 2.3b. Further, the face-centered form has atoms on the corners as well as all the face centers Figure 2.3c. These three types are referred to as simple cubic (SC), body-centered cubic (BCC), and face-centered cubic (FCC), respectively. In nature, SC is rarely found due to its instability; though an example of an SC is the α allotrope of Polonium (α -Po). Examples of BCC include chromium and tungsten, while examples of FCC include aluminum and copper. Some metals such as iron (Fe) can exhibit different Bravais lattices (i.e., phases) depending on the processing conditions. For instance, α -Fe (ferrite) and δ -Fe (delta-ferrite) are BCC allotropes of iron, while γ -Fe (austenite) is the FCC allotrope of iron [7]. Some alloys are engineered to exhibit dual phases such as duplex stainless steel (DSS) which consists of both ferrite and austenite.

Table 2.1: Seven common crystal structures, their unit cell lattice parameters, and their Bravais lattices

	Crystal structure	Lattice parameters	Bravais lattices
1	Cubic	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$	Simple [†] ; body-centered; face-centered
2	Tetragonal	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	Simple [†] ; body-centered
3	Orthorhombic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	Simple [†] ; body-centered; face-centered; base-centered*
4	Hexagonal	$a = b \neq c$ $\alpha = \beta = 90^\circ; \gamma = 120^\circ$	Simple [†]
5	Trigonal	$a = b = c$ $\alpha = \beta = \gamma \neq 90^\circ$	Simple [†]
6	Monoclinic	$a \neq b \neq c$ $\alpha = \gamma = 90^\circ; \beta \neq 90^\circ$	Simple [†] ; base-centered*
7	Triclinic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	Simple [†]

* Base-centered has lattice points on corners plus on a pair of parallel face centers.

† The simple (cubic) is also known as primitive (cubic).

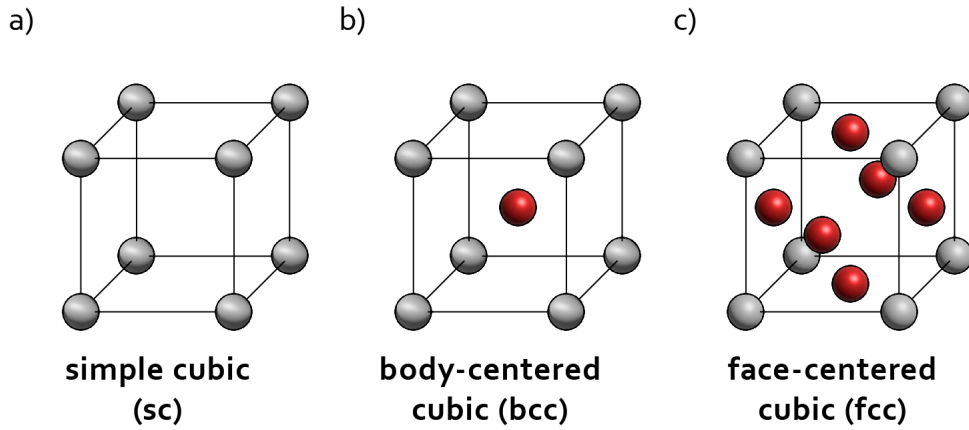


Figure 2.3: Comparison between three different Bravais lattices, (a) simple cubic (SC), (b) body-centered cubic (BCC), and (c) face-centered cubic (FCC).

Upon inspecting a polycrystalline material under the right conditions, the grains will appear to be separated by grain boundaries. The grain boundaries emerge due to a mismatch in the crystal orientation between two (or more) neighboring crystals (Figure 2.4a). Even if the two neighboring crystals are compositionally identical, a mismatch in their orientation angle will result in a grain boundary interface. Depending on the misorientation angle (Figure 2.4b), these grain boundary interfaces can either be high-angle grain boundaries (HAGBs) or low-angle grain boundaries (LAGBs). The cut-off angle between a LAGB and a HAGB is typically in the range of $10\text{--}15^\circ$. Although a HAGB constitutes a crystal of a specific orientation, this condition is considered ideal; in reality, a crystal typically contains local misorientations (below $10\text{--}15^\circ$) which lead to a so-called local misorientations occurring within the area bounded by the HAGB. Nonetheless, due to the low misorientation angle of LAGBs, they often do not have much visual prominence and thus are not as distinct as HAGB. Both HAGB and LAGB are considered as types of defects of a polycrystalline material.

HAGBs/LAGBs, amongst others, are considered as planar-type defects (i.e., defects that occur across a plane). Other types of planar defects are stacking faults (SFs) and boundary twinning. The former occurs when the order of atom stacking is interrupted as shown in Figure 2.5a (e.g., ‘ABCABCABC’ becomes ‘ABCABAABC’), while the latter occurs when a crystal mirrors its neighboring crystal as shown in Figure 2.5b (e.g., ‘CBA—ABC’). But there can be other types of defects such as line- or point-type defects. For example, dislocations (Figure 2.5c) are line-type defects, while single-atom impurities (Figure 2.5d) are point-type defects. Dislocations occur when an extra half-plane of atoms exists between two planes of atoms or when two sets of lattices are offset/sheared from one another, or when a mixture of both occurs. These three dislocations are known as edge-, screw-, and mixed-dislocation, respectively. On the other hand, point defects can exist when an atom is missing (i.e., vacancy defect), when an atom occupies a space where typically no atom exists (i.e., interstitial defect), or when an atom with different structure or chemical composition substitutes one of the bulk atoms (substitutional defect). While many other types of defects exist, this thesis chooses to introduce only those that are crucial or relevant to the understanding of the work in the body of the thesis.

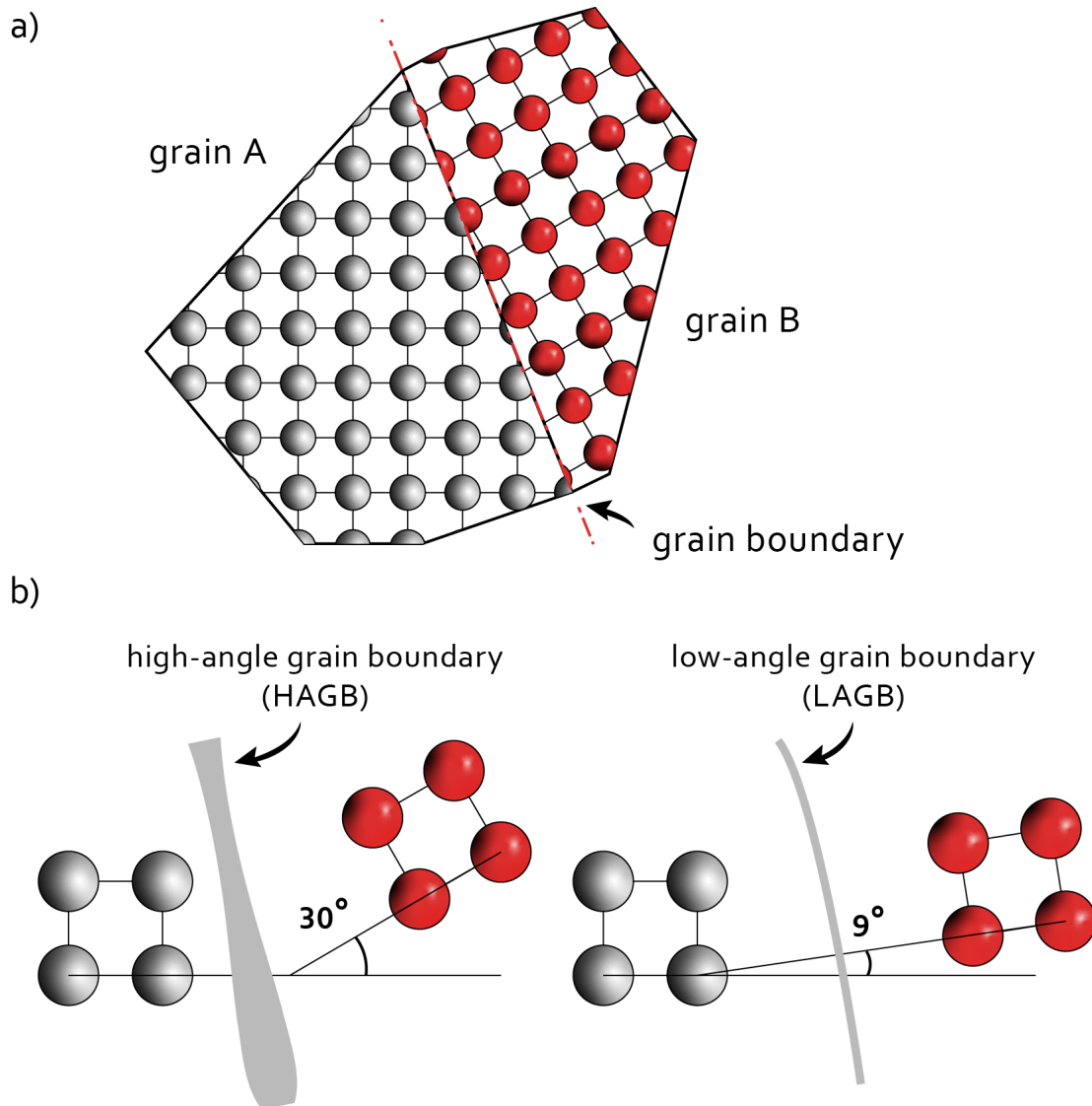


Figure 2.4: Schematics showing the grain boundary interface which emerges due to (a) misorientation between two neighbouring crystals/grains. (b) High-angle grain boundaries are typically defined by a misorientation angle greater than $10\text{--}15^\circ$. Whereas the low-angle grain boundaries are typically defined by a misorientation angle of less than $10\text{--}15^\circ$.

The morphology of the grains (shape, size, aspect-ratio, etc.) can significantly vary depending on the material and the processing conditions. In single-crystal materials, such as quartz, the grain is visible to the naked eye because the entire macroscopic object consists of a single grain (hence the name single-crystal). However, most polycrystalline materials require a closer investigation of the microstructure to observe the grain morphology. Generally, grains assume their form during the transition from liquid to solid, which is known as the solidification stage. During this stage, grains solidify through five different solidification modes: 1) planar, 2) cellular, 3) cellular-dendritic, 4) columnar-dendritic or 5) equiaxed-dendritic, shown in Figure 2.6a-f [8]. For a particular alloy, the solidification mode is dictated by two main parameters, the thermal gradient (G) and the solidification rate (R). The ratio of G/R can be used to determine the solidification mode (Figure 2.6a) [9].

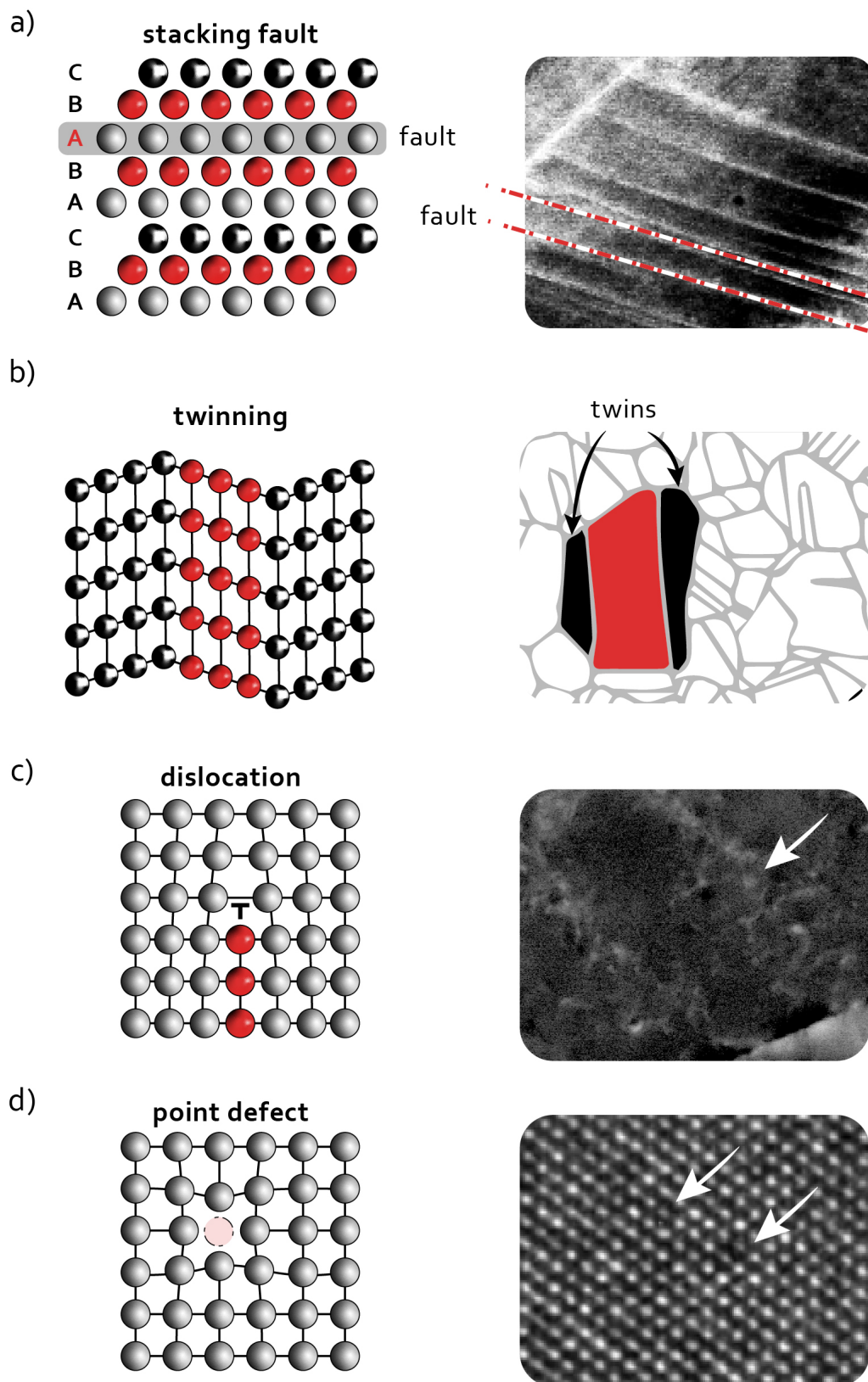


Figure 2.5: Different types of crystallographic defects. (a) stacking fault, (b) twinning, (c) dislocation, and (d) point defect.

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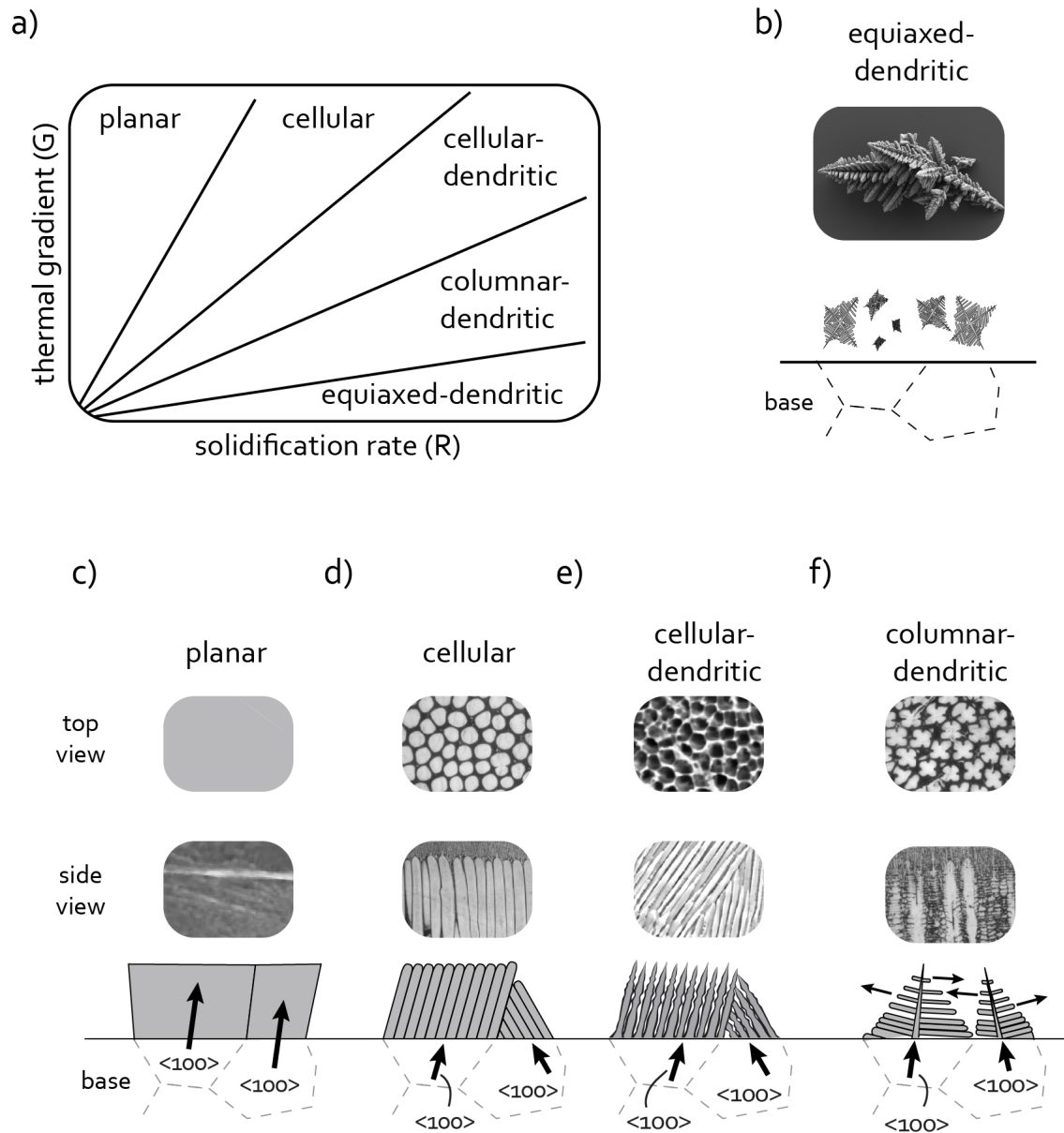


Figure 2.6: (a) The solidification mode is dictated by the ratio of thermal gradient (G) to the solidification rate (R). Schematics and actual images of the (b) equiaxed-dendritic, (c) planar, (d) cellular, (e) cellular-dendritic, and (f) columnar dendritic are presented. Note their growth direction with respect to the base crystals/grains.

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A simple scenario which can be used to conceptualize G and R is to consider a stationary laser beam with a circular profile. Let us assume its power (P , in Watts) and speed (V , in m/s) are equal to p and 0 , respectively. We will melt a solid object by projecting this beam perpendicularly onto the object's surface. Observing the surface from a top view will show a melt pool of a circular profile (Figure 2.7a). For the sake of simplicity, we will assume that only one of two states can exist at a time, solid (S) or liquid (L) (i.e., no mushy zone). The center of the melt pool is where the maximum temperature ($T_{\max}$) will be. At a certain radial distance (r) away from the melt pool center, the temperature reaches an infinitesimal value between the melting and solidifying state which we will denote as the solid-liquid (S-L) interface (Figure 2.7a). Upon switching off the beam ($P = 0$), the S-L interface will transform into a fusion line (FL) from which the solidification will initiate (Figure 2.7b). Note that due to symmetry, the fusion line's thermal gradients and solidification rates (G_{FL} and R_{FL}) are constant. In other words, the solidification mode and rate are uniform along the FL. As a result, the solidification will initiate orthogonal to the FL and towards the melt pool's center (Figure 2.7c) [17].

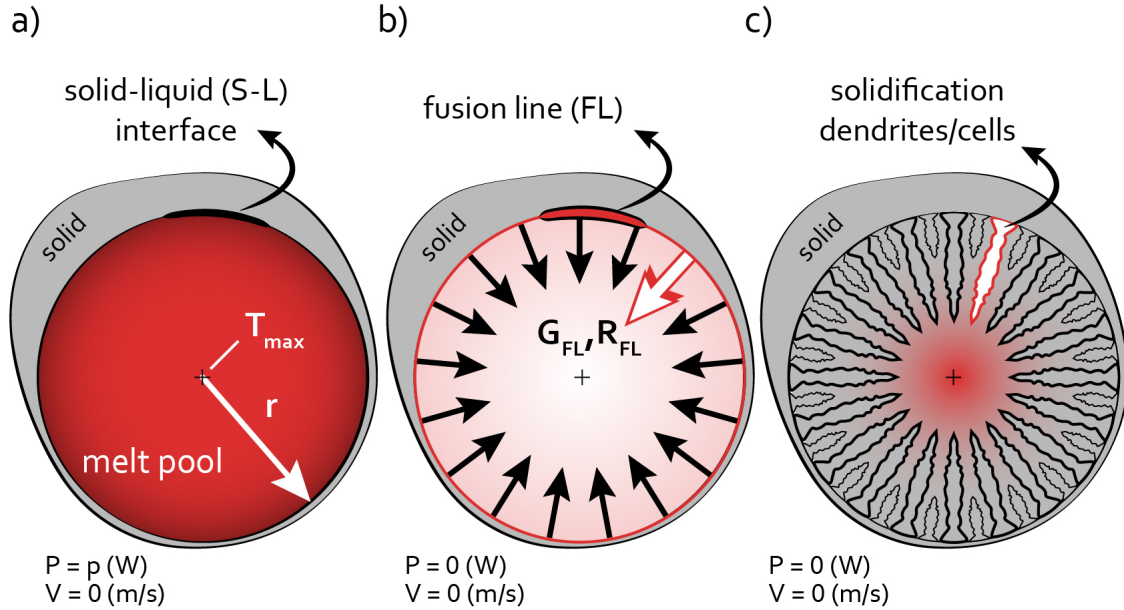


Figure 2.7: Top view of a welding spot ($V = 0$ m/s and $P = p$ W) showing (a) melt pool, (b) thermal gradient (G) and solidification rate (R) directions, and (c) the resultant solidification cellular dendrites. In this example, the solid-liquid (S-L) interface (a) is transformed into the fusion line (b) from which the solidification perpendicularly stems.

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Now let us consider a dynamic situation where the laser is moving. For arbitrary non-zero values p and v , a resultant melt pool can be observed in Figure 2.8a. The resultant two-dimensional morphology of the solidified metal is shown in the wake of the spot. As in the previous example, the S-L interface separates the liquid from the solid state.

The moving melt pool leaves a trace of the FL (the two tangent lines to the oval shape). The center of the melt pool leaves a trace of the centerline (CL). The figure shows that the temperature profile of the CL is always greater than FL, given that CL has a closer proximity to the heat source. But unlike the previous example, the thermal gradient (G) and solidification rate (R) are not constant. In this case, if we traced G along the S-L interface, we would observe that it is smallest where the S-L intersects the CL (G_{CL}) and greatest where the S-L intersects the FL (G_{FL}) (see Figure 2.8b). On the other hand, R has a maximum value where the S-L intersects the CL ($R_{CL} = v$). Moving along the S-L towards the fusion line will gradually decrease R until it reaches an infinitesimal value at the intersection between S-L and FL ($R_{FL} \approx 0$) [8]. Changing the speed will change the melt pool shape, and therefore change G and R , which will impact the morphology of the grains in its wake [18, 19].

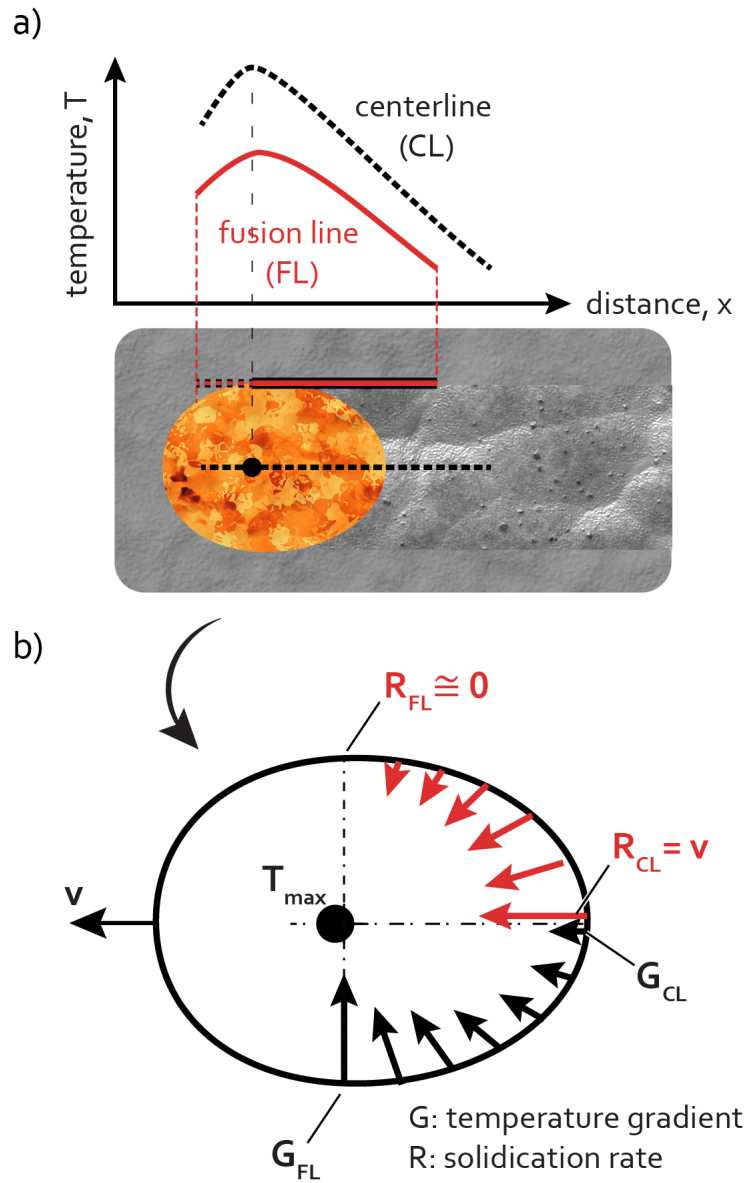


Figure 2.8: A welding spot of a moving laser beam ($V = v$ m/s and $P = p$ W) showing the temperature profiles of the fusion line and the centerline (a). The magnitudes of the thermal gradients (G) and solidification rates (R) are presented in (b). Where the maximum and minimum G occur are the fusion line and the centerline, respectively. Whereas the maximum and minimum R occur along the centerline ($R_{CL} = v$) and the fusion line ($R_{FL} = 0$) respectively.

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Apart from the grain morphology, the orientation of the crystals or grains can vary depending on the chemical composition and the process variables. Thus, even if two grains are geometrically identical, their characteristic crystallographic orientation need not be identical. The crystal orientation is often characterized in the three-dimensional space by means of the Euler angles (ϕ_1 , ϕ , ϕ_2). Modern techniques such as electron backscatter

diffraction (EBSD) can resolve the Euler angles of a cross-sectional surface (more on the EBSD technique in Chapter 3), after which we are able to visually discern the orientation of individual crystals. One method is called inverse pole figure (IPF) mapping, which consists of a color key, a reference frame, and the mapped grains Figure 2.9. First, a reference frame has to be selected, say the z-axis (out of screen) which we would name as IPF-z or $\text{IPF}\parallel z$. The color key is then used to indicate which direction of the crystal is parallel to the reference frame z. For example, the red-colored grains have a $[001]$ direction parallel to the z-axis, while the blue-colored grains have a $[111]$ parallel to the z axis. In addition to the crystal orientation, EBSD data provide quantitative information on the (two-dimensional) size of grains, shape of grains, grain boundaries, local misorientation angles, phase identification, etc.

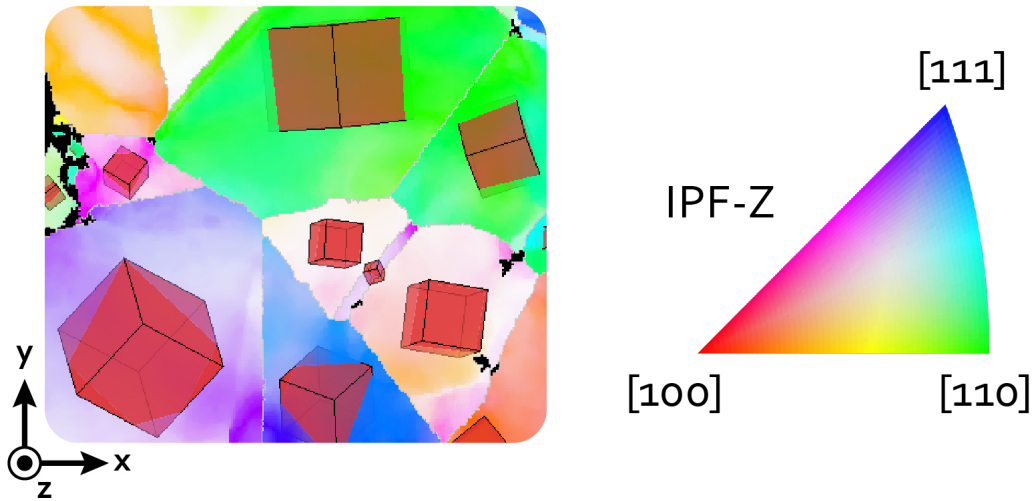


Figure 2.9: An inverse pole figure (IPF) map showing the crystallographic orientation. This map is presented with the reference frame along the z axis (into/out of screen). The orientation of the cubes (overlaid on the crystals) can be determined via the IPF color key on the right-hand side.

When a material has the bulk of its crystals oriented similarly, that material is said to exhibit a (crystallographic) texture. Materials can naturally exhibit texture or have randomly oriented crystals. Depending on the need, texture can be induced or reduced by certain treatment or processing techniques. Crystal texture can be observed in a number of ways, one of which is using the so-called pole figures. Unlike the IPF, a pole figure uses a predefined crystal's plane normal (denoted by the miller 'HKL' notation) to determine its orientation. The frequency of observed planes appear as dots in the pole figure, which correspond to a two-dimensional projection of an intersection between a line projected from the specified plane normal and a three-dimensional hemisphere [20]. The dots can then be represented in the form of contours to generate the 'contour' pole figure shown in Figure 2.10.

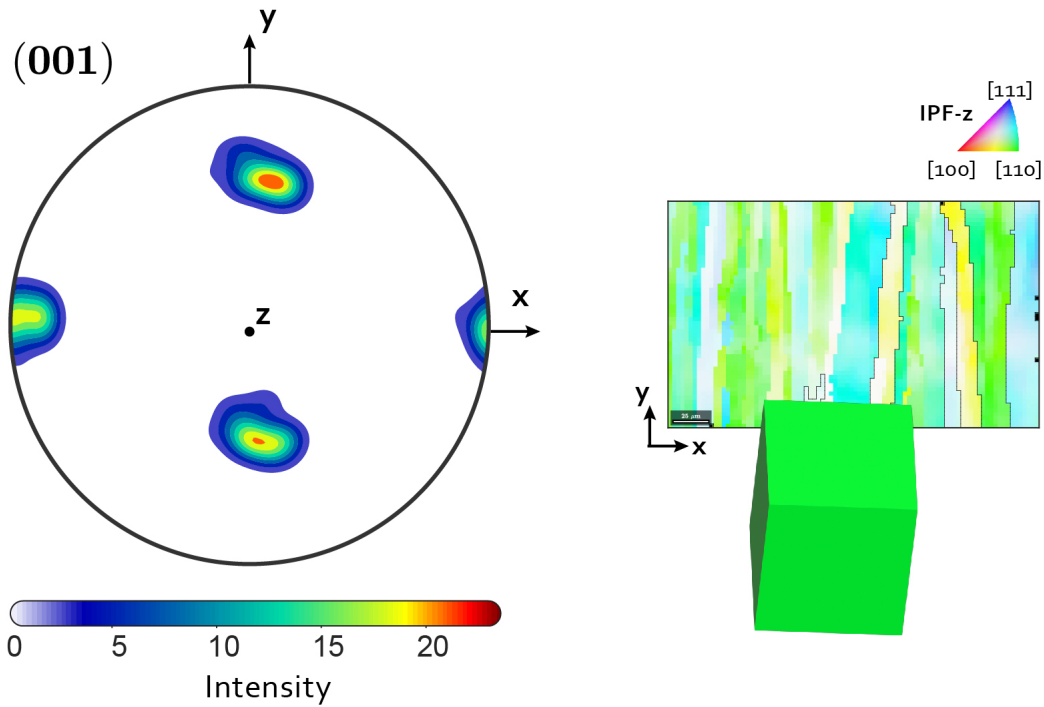


Figure 2.10: Contour pole figure map. This map can conveniently show the frequency of any desired planes (e.g., (100)) with respect to the sample's reference axes. The map was generated of a 3D-printed 316L stainless steel sample with strong $\langle 110 \rangle$ (green) texture.

2.2.2 Overview of the microstructure-property relationships

Not only do the microstructure constituents play a significant role in the overall mechanical performance of the material, they also contribute to the electrical, magnetic, thermal, and chemical properties of the material [21]. Therefore, it is crucial to understand the underlying microstructure and its impact on the relevant material properties. For instance, certain nuances of the microstructure may promote or inhibit corrosion, and thus can impact the corrosive (or oxidative) wear of the material. Similarly, other microstructure constituents may increase or decrease the hardness of the material, which can impact its wear behavior in a tribological setup. This section will cover as many relevant topics as possible drawn both from existing literature and the author's own knowledge of the topic.

One of the most basic concepts related to a material's strength is its yield strength (σ_y), the point at which the material undergoes plastic deformation. Plastic deformation in metals is typically accommodated by the so-called slip mechanism and the motion of dislocations. When the stress resulting from the external forces exceeds σ_y , the metal is coerced to adapt as to accommodated for such external forces. This adaptation occurs in

the form of slipping of atoms over one another in a certain plane and direction. However, slipping occurs in an energy-efficient way, i.e., by the motion of dislocation (rather than a sudden slip of bulk crystals). During slip, the atomic bonds of the moving atoms and its neighbors are broken and reformed successively which results in a travel of the dislocation along a particular slip direction and a particular slip plane. The combination of slip direction and slip planes are referred to as the slip system. For example, FCC crystals have 12 slip systems characterized by the miller notation $\{111\}\langle 110 \rangle$ shown in Figure 2.11 (where the $\{111\}$ denote the family of planes and the $\langle 110 \rangle$ denote the family of directions). Understanding these concepts is important because in order for a material to plastically deform, there needs to be a way for dislocations to move. If dislocations are harder to move, then the material is considered ‘stronger’ because it would require more energy to induce plastic deformation. Noteworthy, as dislocations move, they can impinge upon a grain boundary or other dislocations. Such events can facilitate the motion of dislocation, impede the motion of dislocation, or outright annihilate the dislocation. These complex interactions play a significant role in material strengthening and thus has spurred waves of interests from material scientists and engineers since the 1930’s [22].

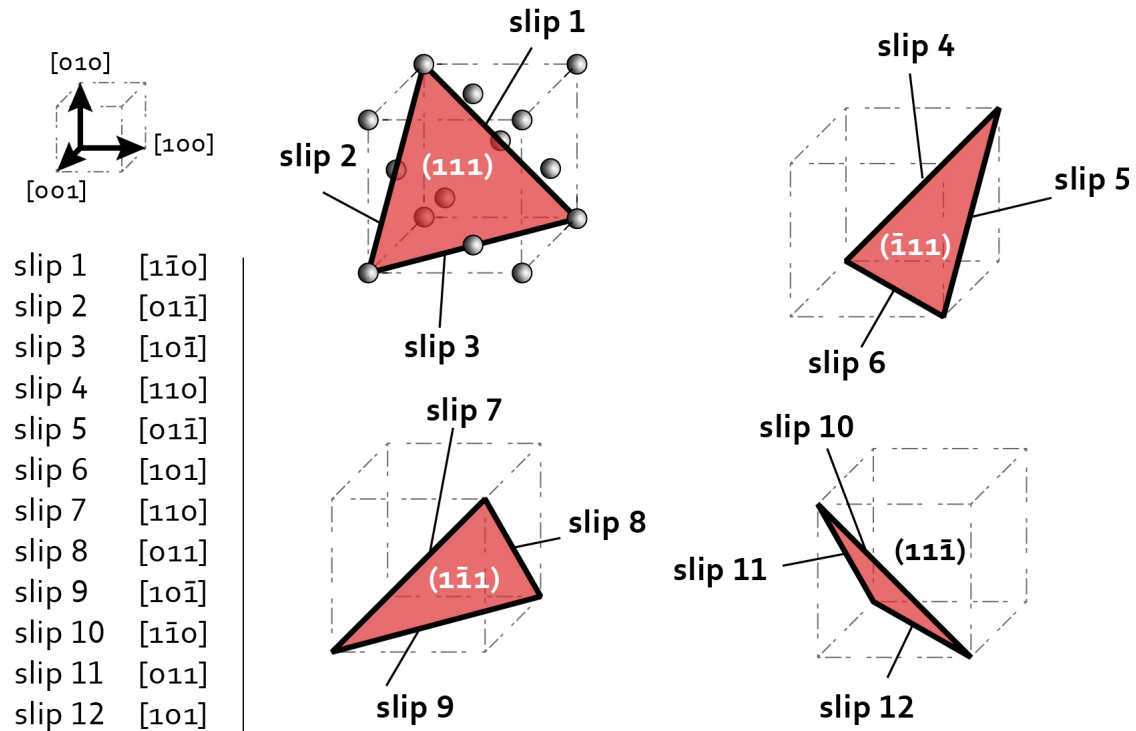


Figure 2.11: Twelve slip systems of the FCC Bravais lattice. In FCC, slipping occurs on the $\langle 011 \rangle$ family of directions which lie on the family of $\{111\}$ planes.

Over the years, practitioners devised four key material-strengthening mechanisms [21]:

- (i) work hardening;
- (ii) grain size reduction;
- (iii) solid solution strengthening; and
- (iv) precipitation hardening.

Strengthening via work (or strain) hardening occurs through various means such as hammering, rolling, or drawing. Such processes increase the dislocation density (ρ) over the unit area (ρ /m²) by several orders of magnitudes. For example, ρ of well-annealed metals typically falls in the range 10^{10} - 10^{12} /m², while in heavily cold-rolled metals, it typically falls in the range of 10^{14} - 10^{15} /m² [23]. The increase in dislocation density is typically accompanied by an increase in σ_y of the metal; because dislocations can serve as sites for dislocation pile-up, which stop other moving dislocations and thus impede slipping. As a result, plastic deformation becomes more difficult to achieve. The first dislocation-strengthening theory was that of Taylor, proposed in 1934 [24]. Taylor postulated that the flow shear stress (τ_{flow}) required to move a dislocation is related to the total density of dislocations (ρ /m²) present in the material. This theory has been verified experimentally at least since the early 1960's [25, 26]. Taylor's equation became widely used given its important mechanistic insight into the material's mechanical strength. The Taylor equation (also referred to as Taylor forest hardening equation) for the τ_{flow} is given by:

$$\tau_{flow} = \tau_o + k' \sqrt{\rho} , \quad (2.1)$$

where τ_o is the required shear stress to move a dislocation in the absence of dislocations, and k' is often treated as a constant. For convenience, we may also relate τ_{flow} to the applied normal yield stress (σ_y) as:

$$\sigma_y = \frac{\tau_{flow}}{\cos\psi \cos\zeta} \quad (2.2a)$$

$$= \frac{\tau_o + k' \sqrt{\rho}}{\cos\psi \cos\zeta} \quad (2.2b)$$

$$= \sigma_o + k'' \sqrt{\rho} , \quad (2.2c)$$

where ψ is the angle between the slip plane's and applied force's normal and direction, respectively; and ζ is the angle between the slip's and applied force's directions. For convenience, ψ and ζ may be treated as constants and thus be incorporated within τ_o and k' , hence becoming a constant for frictional stresses (σ_o) and a second term constant (k''), respectively. It thus follows that as ρ increases, σ_y increases, which increases the material's strength to plastic deformation. However, this strengthening mechanism is also associated with a drop in useful ductility, which has been a long-standing dilemma for material scientist. As a result, an 'optimal' balance between strength and ductility is often decided based on the ultimate application in which the material is utilized.

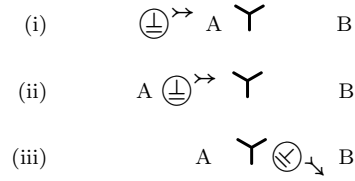
The second mechanism of strengthening previously mentioned was by changing the average grain size (D) of the material (i.e., grain size reduction). Hall and Petch [27, 28] observed that σ_y increases when D decreases. Their experiments led to the now well-known Hall-Petch relationship:

$$\sigma_y = \sigma_o + \frac{k_y}{\sqrt{D}} \quad (2.3)$$

In Eq. 2.3, σ_o denotes the frictional stress (same as 2.2c) and k_y denotes the Hall-Petch coefficient. This inverse-proportionality between σ_y and D can be explained by how grain boundaries (generally the HAGBs) act as barriers to the motion of dislocations. To illustrate, consider two neighboring grains, (A) and (B), separated by a HAGB:



Let us now consider a single dislocation ($\perp$) moving from within (A) towards (B), and give it the notation $\perp_A$. As depicted below this paragraph, the moving $\perp_A$ will need to change its course of motion just as it crosses the HAGB. This change is necessary because the active slip systems of (B) are not the same as those in (A). Which means that by the time $\perp_A$ has crossed the boundary, it has changed its direction of motion, becoming as so: $\bowtie_B$. This process requires more energy and thus makes dislocation motion, and specifically plastic deformation, harder to occur.



Now consider two separate moving dislocations, $\perp_{100}$, and $\perp_{1,000}$, both traveling the same distance x . The subscripts $_{100}$ and $_{1,000}$ denote the number of HAGB each dislocation has to cross in order to reach its terminal destination. Becuase of the shear number of HAGB, $\perp_{1,000}$ would require much more force to reach its desintation than $\perp_{100}$ would. This situation becomes more pronounced in multi-phase materials, where the phase of (A) $\neq$ (B). Due to the heterogeneity of grains, a traveling $\perp$ may encounter a completely different crystal structure and slip systems when it crosses a HAGB.

Given that the material's strength depends on both the D and ρ , the Hall-Petch (Eq. 2.3) and Taylor (Eq. 2.2c) terms can be combined to describe the material's σ_y as follows:

$$\sigma_y = \sigma_y^{dislocations} + \sigma_y^{grain-size} \quad (2.4a)$$

$$= \sigma_o + k'' \sqrt{\rho} + \sigma_o + \frac{k_y}{\sqrt{D}} \quad (2.4b)$$

$$= 2\sigma_o + k'' \sqrt{\rho} + \frac{k_y}{\sqrt{D}} \quad (2.4c)$$

$$= \sigma_o + k'' \sqrt{\rho} + \frac{k_y}{\sqrt{D}}, \quad (2.4d)$$

where $2\sigma_o$ (in Eq. 2.4c) was combined into a single term σ_o (in Eq. 2.4d), representing the frictional stresses. Nonetheless, Eq. 2.4d will be further expanded later in this section as to account for one particularly important constituent of the k'' coefficient.

The third mechanism of strengthening, i.e., solid solution strengthening, involves a chemical variation of the material by replacing one type of atom in the structure by another (substitutional solid solution) or by adding atoms to sites in the structure where normally no atoms exist (interstitial solid solution). In both cases, the introduction of the alloying (solute) atom to the bulk atoms strengthens the material through a chemical or an elastic process. In the former, the difference in chemical bonding between the alloying atom and the bulk atoms changes the dislocation-atom interactions. In the latter, a larger size substitutional/interstitial atom creates a strain field which acts as a barrier to dislocation motion. In the case where the introduced atom is of the same size as the bulk atoms, then no strain field is created.

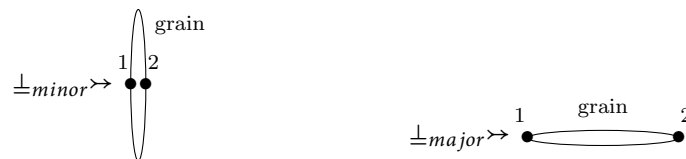
The last mechanism of strengthening, i.e., precipitation hardening, involves a heat treatment process that leads to change in solubility of certain alloying elements. This technique results in the emergence of precipitates within the original microstructure which grow over time. Initially, the growth of these precipitates is accompanied by a strengthening of the material, as they act as obstacles to dislocation motion. Nonetheless, as the precipitates continue to grow, the material starts losing the attained strength, as the precipitates at that size tend to facilitate dislocation motion. Thus, practitioners use different techniques to halt this growth before the material loses the attained strength.

Defects are among the constituents that play a significant role in the mechanical strength materials. Although the role of HAGBs in material strengthening has been introduced, grain boundaries, in general, are known to influence the corrosion [29–32], creep [33, 34], electrical [35] and, thermal [36] performance of the material. The other planar-type defect, i.e., stacking fault, is strongly correlated with the twinning deformation mechanism (twinning is another competing deformation mechanism to slipping). Different metals

possess different typical ranges of stacking faults, which are normally characterized by the stacking fault energy. In practice, lower stacking fault energy is associated with higher susceptibility of twinning in face-centered cubic (FCC) metals and alloys [37–39]. Advantageously, the alloying process allows for inducing different stacking fault energy to control the susceptibility to deformation twinning. Apart from being associated with low stacking fault energy, deformation twinning typically occurs at high-pressure, high-strain, low-temperature deformation [40–42]. The last type of defect-type, i.e., vacancies, lead to a reduction in the density of the material due to missing atoms. Furthermore, they also contribute to an increase in the yield strength of the material, accompanied with a drop in ductility [43].

Alteration of the chemical composition of the alloy has been effectively used to combine or even induce new properties in materials. For example, titanium alloys such as Ti-6Al-4V typically comprise HCP and BCC Bravais lattices denoted by α - and β -phase, respectively. Alloying elements (α -stabilizers: Al, Sn, Ga, Zr, C, O, and N; and β -stabilizers: V, Mo, Nb, Ta, Cr, Fe, Cu, Ni, Si, and B) are added or subtracted to alter the ratio between the α - and β -phase [44]. Decades of experimentation gave rise to tens of grades of titanium alloys that suit variable applications from biomedical to aerospace. Analogously, the addition of Cr and Ni to austenitic steels (e.g., grade 316L) has proven to greatly enhance their corrosion resistance.

The grain morphology can have significant impact on the mechanical properties of a material. One example that has been already covered was the strengthening associated with grain-size reduction (see Eq. 2.3). In this examples, the grains were assumed to be equiaxed (e.g., Figure 2.6b). But what happens when a grain's spatial dimension is elongated (e.g., Figure 2.6e)? To illustrate, consider an elliptically shaped grain, depicted below this text, whose major and minor axes' aspect ratio is 10:1. Now consider an externally moving $\perp$ that is heading towards the grain. Let us assume two scenarios: one in which the $\perp$ motion is along the minor axis ($\perp_{minor}$), and another where the $\perp$ motion is along the major axis ($\perp_{major}$). In both scenarios, $\perp_{minor}$ and $\perp_{major}$ will eventually cross from point 1 and 2 on the grain's boundary. However, the time it takes $\perp_{minor}$ to cross from 1 to 2 is one tenth of the time it takes $\perp_{major}$ to cross from 1 to 2. The implication of this is that $\perp_{minor}$ would encounter a resistance to motion significantly earlier than $\perp_{major}$ would. And this cycle can repeat if the next grains along the $\perp$'s path exhibit the same morphological arrangement. In other words, plastic deformation is significantly harder in the scenario where the distance from 1 to 2 is shorter. This form of relative strength (mechanical anisotropy) stems from the morphological arrangement of grains.



The solidification stage can cause grains to solidify in high aspect-ratios, causing many forms of anisotropic behaviors. For example, Vandersluis et al. observed that hardness improved for aluminum alloy (A319) when they deliberately altered the solidification rate (R). They attributed this strength to the refinement of grain boundaries, harder second phase, and stress-concentrating particles [45]. In another example, Hofweber and Fiore reported that the cellular solidification mode of Ni-base superalloy is weaker and more brittle than the dendritic mode, which they attributed to trapped impurities between the grain boundaries [46].

Another major contributor to the properties of a polycrystalline material is its crystallographic texture (i.e., the bulk crystal orientation). Texture can trigger anisotropic behavior across a wide range of properties such as yield strength [47], corrosion resistance [48], electrical [49], and thermal conductivity [50]. In fact, there is a direct (positive) correlation between the degree of anisotropy of the material and its texture strength. Upon achieving a perfectly textured material, a polycrystalline effectively transforms into a monocrystalline material, and thus the entire material behaves as a single crystal. And since there is a direct correlation between anisotropy and texture, it follows that a single crystal (i.e., the perfect texture) must be intrinsically anisotropic. One simple way to elucidate the single crystal anisotropy is to look at the underlying unit cell of the associated Bravais lattice (e.g., FCC, BCC, HCP, etc.). A Bravais lattice exhibits anisotropic behavior due to the nature of arrangement between atoms and their bonds. For example, the pure Fe BCC unit cell shown in Figure 2.12a exhibits different Young moduli along the [001] (228 GPa), [011] (296.5 GPa), and [111] (319.3 GPa) directions. Whereas an austenitic (FCC) alloy (Fe-12Cr-18Ni) in Figure 2.12b exhibits Young moduli of 198 GPa, 283.5 GPa, and 312 GPa along the same respective directions (values calculated based on elastic coefficients found in [51] for BCC and [52, 53] for FCC). To account for texture in a materials' strength, the coefficient k'' of the Taylor dislocation term in Eq. 2.4d can be expanded as follows [54, 55]:

$$k'' = M\alpha\mu b \quad (2.5)$$

where the first term (M) is the Taylor orientation factor, which varies based on a crystal's orientation relative to the loading direction. α is the constant accounting the interactions of dislocations in different slip systems, μ is the shear modulus, b is the Burgers' vector magnitude. Hence, Eq. 2.4d may be finally re-written to accommodate for the grain sizes (D), dislocations (ρ), and crystal orientations (M) as:

$$\sigma_y = \sigma_o + M\alpha\mu b\sqrt{\rho} + \frac{k_y}{\sqrt{D}} \quad (2.6)$$

The value of M can vary over a certain range based on the crystal's orientation and its Bravais lattice. For example, in FCC lattices M can typically vary between 2.449 for the [001] direction to 3.6 for the [111] direction [56]. As a result, the [111] direction is 'harder'

than the $[001]$ direction in a single FCC unit cell. This variation in M is used in the determination of the stiffness moduli for various loading directions (revisit 2.12). Due to this unit-cell anisotropy, a pronounced texture would propagate the anisotropy towards the macro scale. However, on the macro scale, a material is observed to behave isotropically the more randomly oriented its crystals are. Primarily because the anisotropic effects from crystals tend to ‘cancel out’ one another, thus, resulting in an overall isotropic behavior. Due to the wider impact on material properties (mechanical strength, electrical/thermal conductivity, corrosion resistance, etc.), crystallographic texture continues to be an active area of research in materials science.

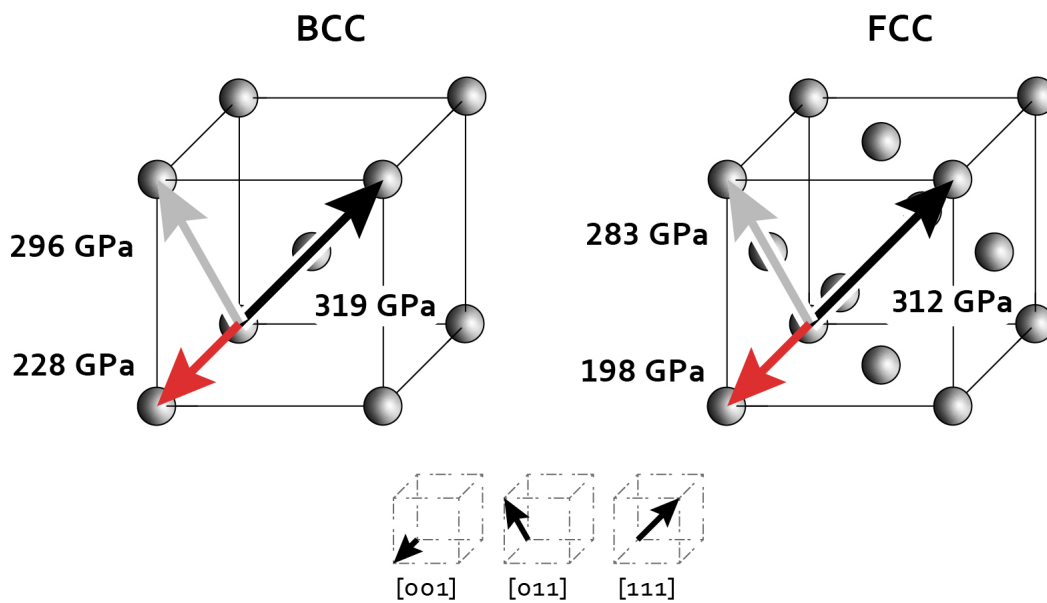


Figure 2.12: The effect of Taylor orientation factor (M) on the stiffness of two BCC and FCC Bravais lattices. It can be seen that in all cases, the $\langle 111 \rangle$ directions are the stiffest, whereas the $\langle 001 \rangle$ is the least stiff.

2.3 Selective laser melting (SLM)

2.3.1 History, principles, applications, and materials

SLM was invented in the 1990's by a German company (Fraunhofer ILT) [57]. As with any AM technology, SLM first relies on the creation of a computer-aided design (CAD) model which is subsequently converted to its triangular representation known as standard tessellation language (STL). The computer uses the STL file to create two-dimensional slices that collectively form the complete three-dimensional geometry. Each of the two-dimensional slices contains a set of instructions for the three-dimensional printer stating where and how ‘printing’ should occur (Figure 2.13). The printing part contains a wide

range of possible parameters (covered later in this section) that vary based on the user choice and the type of printer and its capabilities. In essence, SLM employs a laser to selectively melt specified regions of a powder bed in successive two-dimensional slices (i.e., after a powder layer is selectively melted, a fresh layer of powder is laid on top). The melting of powder clusters induces a melt pool which upon cooling down transitions into a unified solid body as previously shown in Figure 2.1. One benefit of manufacturing in this additive manner is the ability to fabricate parts with complex geometries which cannot be done with molds or conventional manufacturing routes.

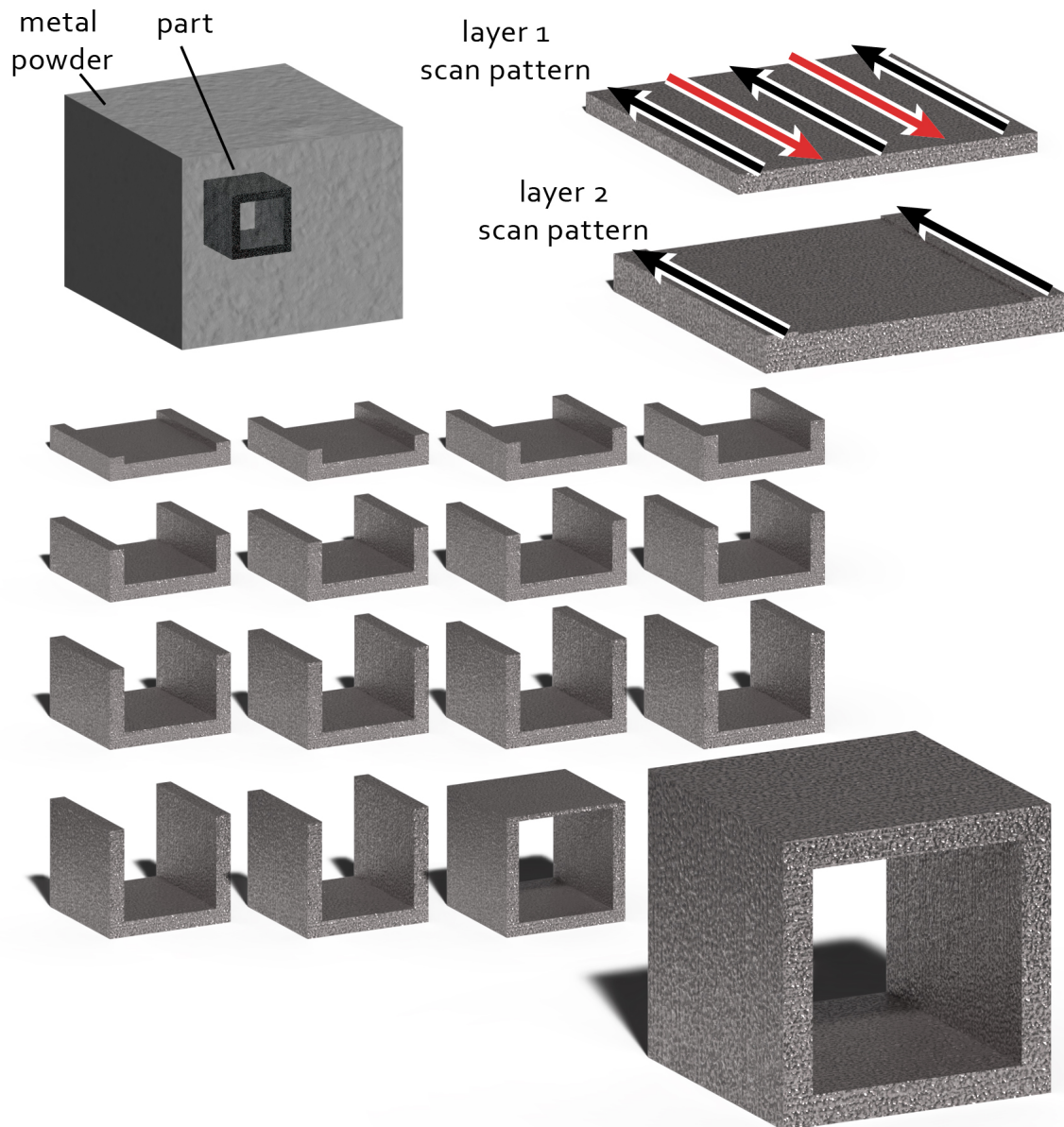


Figure 2.13: A schematic showing layer-by-layer fabrication of the selective laser melting (SLM) process. The laser scanning patterns are shown for layers 1 and 2.

Many materials can be used with SLM such as, metals, ceramics and, composites. According to Wohlers et al.'s [58] survey on SLM materials from 1999 to 2014, nearly 32.3% of published research on SLM utilized steel and iron-based alloys. Next was titanium-based and nickel-based alloys at 24.1% and 8.6%, respectively. Other metals constituted 19.3%. Finally, composites and ceramics amounted to 10.5% and 5.2%, respectively.

Yap et al. [59] discussed the most prominent applications for SLM in published research. They reported that Fe-alloys (e.g., steel) were mostly used in medical and dental applications, heat exchangers, light-weight structures, porous structures. Whereas Ti-alloys (e.g., Ti6Al4) were mostly used in medical and dental implants due to their superior biocompatibility and lightweight. It was also reported that Ti-alloys were used in the development of light-weight automotive and aerospace components. Ni-based alloys (e.g., Inconel superalloy) were used for applications involving extreme environmental conditions. Research in these alloys mostly covered high-temperature applications such as aircraft engine components, gas turbine blades, and turbocharger rotors. Other types of metal groups such as copper, aluminum, magnesium, tungsten, gold, and cobalt-chrome were marginal with applications mostly covering biomedical, dental, heat-exchangers, and jewelry. As for SLM-based composites (e.g., metal matrix composites (MMCs)), applications were largely dependent on the desired material properties. This field continues to grow rapidly due to the limitless room of possibilities of developing new SLM materials. For example, SLM was used to investigate the capability of producing composite materials for medical implants, aerospace, automotive, machining tools, molds creation, and heavy-duty electrical contacts. On the other hand, SLM research in ceramics was reportedly limited due to the poor flowability of ceramic powder on the SLM bed (because ceramics are thinner than metals, evenly-spreading them on the power bed presents a current challenge). Nonetheless, published SLM research included a variety of ceramics such as silica, alumina, yttria-stabilized zirconia, tri-calcium-phosphate, dental porcelain, alumina-silica mixture, silicon carbide, and silicon monoxide. The applications of ceramic-based SLM were mostly in medical and dental uses. However, according to Yap et al., there has been other published research focusing on creating molds by melting silica as well as creating thin wall structures for turbine blades and nozzles.

According to Van-Elsen [60], the SLM process has over 50 parameters, which makes it a rather complex process. These parameters varied between those which are controllable (e.g., laser speed, laser power, etc.) and those which are predefined (e.g., powder's diffusivity or morphology). Regardless of the controllability, the parameters were categorized into four groups: 1) Laser and scanning parameters, 2) powder material properties, 3) powder-bed properties, and 4) recoat parameters, and build environment parameters. The controllable parameters are listed in Table 2.2.

Table 2.2: List of controllable SLM parameters according to Van-Elsen [60].

Classification	Parameters
Laser and scanning parameters	Laser power Laser spot size Scan velocity Hatch spacing Scan strategy
Powder-bed properties and recoat parameters	Deposition system parameters Layer thickness Powder-bed temperature
Build environment parameters	Oxygen level Pressure Gas flow velocity Ambient temperature

The combined effects from the predefined 38 parameters and from changes made to the controllable 12 parameters will ultimately dictate the quality of the produced part. In fact, changes in input parameters can have a large impact on the part's quality [61, 62]. However, with the large number of parameters involved, isolating the impact of a single parameter is quite difficult given the complex parameter-interaction effects that come into play. Regardless, efforts were made to aggregate some of the most prominent controllable parameters into a series of functions in an attempt to correlate input parameters with produced part's qualities (e.g., defects, microstructure, residual stresses, etc.). One such parameter is the linear energy density (E_l) function [63]:

$$E_l = \frac{P}{V} \quad , \quad (2.7)$$

where P is laser power, V is the laser speed. E_l is in J/m. However, due to E_l 's oversimplification of the process parameters, many other researchers [64–77] use a more encapsulating expression referred to as the volumetric energy density (E_v):

$$E_v = \frac{P}{V \, h \, t} \quad , \quad (2.8)$$

where h is the hatch distance, t is the powder layer thickness. E_v is in J/m³. Despite their reasonable utility, E_l and E_v have limitations; many researchers who relied on these two energy expressions reported contradictory findings in the final parts' quality [67, 73, 77–79]. Shipley et al. [78] argued that neither the energy density function nor the laser power and scanning speed relationship is alone sufficient to predict defects such as porosity. Although E_v contains more parameters than laser power-scan speed, it lacks other possibly significant attributes such as the laser type, powder-bed temperature, and focal offset distance. Finding a better correlation function remains a current challenge in the field.

As covered previously in 2.2.1, the heat flux which is aligned with the direction of a laser beam plays a significant role in the solidification process. This correlation means that the choice of laser scanning pattern in the SLM process impacts solidification process and, as a result, the anisotropy of the material. One simple way of demonstrating this effect is by arbitrarily fixing the laser scanning direction (SD) and producing geometrically identical samples with different orientations with respect to the build plate. This approach has been documented in lattice structures [80], dog-bone tensile test specimens [81], and tribology [82]. All of these studies reported some level of variation in the anisotropic properties by merely rotating/tilting the specimens on the build plate. However, the scanning patterns, also known as scan strategies, can be deliberately chosen to induce a different microstructure even without the need to rotate/tilt the specimens. This feature alone has spurred a wide interest from many researchers – which is well documented in Jia et al.’s excellent review paper on the topic [83]. Their summary involved 14 different SLM scan strategies shown in Figure 2.14. The arrows on the figure indicate the SD for the corresponding scan strategy. It can be seen that the SD can be manipulated not only in single layers, but in sequential layers. Such examples of flexibility offered by SLM (and other AM processes) will certainly sustain research interest for years to come.

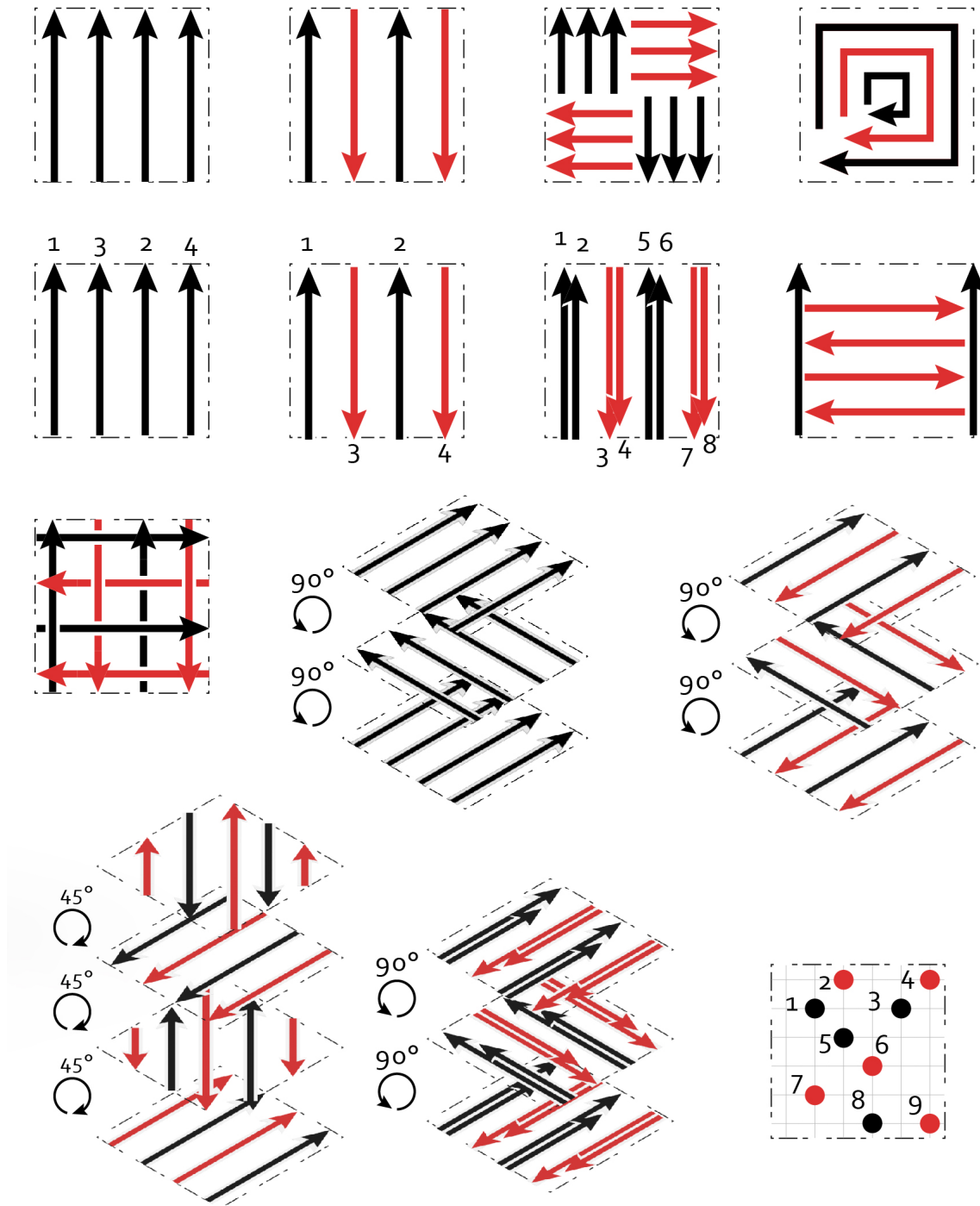


Figure 2.14: Various laser scan strategies available in SLM.

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2.3.2 Austenitic 316L stainless steel via SLM: a detailed inspection

We previously discussed the ubiquity of Fe-based alloys in SLM research, and in fact, between 2003 and 2013 there were more than 100 publications involving SLM-manufactured Fe-based alloys [59]. However, based on a more recent review by Fayazfar et al. [84], one particular Fe-based alloy dominated the powder-bed-fusion research field (accounting for 41%), namely 316L stainless steel. The interest in this particular alloy is owed to its excellent corrosion resistance, high ductility, and biocompatibility. This Fe-based alloy is part of the 300 (austenitic) series and is typically further characterized by its Cr-Ni ratio (although other elements do exist). Furthermore, the “L” suffix signifies the low carbon content, which is typically below 0.03 wt.%. It is often found that this series alloy is accompanied by the attribute “austenitic” stainless steel for being primarily composed of single-phase austenite (λ -Fe). Nonetheless, in reality this alloy can exhibit a range different phase-compositions depending on its chromium equivalent (Cr_{eq}) and nickel equivalent (Ni_{eq}), which are calculated as follows [85, 86]:

$$Cr_{eq} \text{ (wt.\%)} = Cr + Mo + 1.5 Si + 0.5 Nb \quad (2.9)$$

$$Ni_{eq} \text{ (wt.\%)} = Ni + 0.5 Mn + 30 C + 30 b \quad (2.10)$$

In the 1940's, Schaeffler [87] developed a reasonably accurate phase-transition diagram based on these two element-equivalents for various 300-series steel (308, 309, 309 Nb, 310, 312, 316, 317, 318) [87, 88]. His remarkable experimental work distilled the solidification modes, phase transitions, and final phase composition to the four shown in Table 2.3. The letters in the first column denote the fully austenitic solidification mode (A), austenitic-ferritic solidification mode (AF), ferritic-austenitic solidification mode (FA), and fully ferritic solidification mode (F), respectively. The middle column shows the equivalent chromium-to-nickel ratio (Cr_{eq}/Ni_{eq}) for which the corresponding solidification applies. The last column shows the sequence of phase transitions via arrows where λ , δ , L denote austenite, delta-ferrite, and liquid, respectively. Depending on the Cr_{eq}/Ni_{eq} ratio, the final composition (i.e., after solidification) comprise either single phase λ or dual phase $\lambda + \delta$, or single phase δ . Ni is known as the a ‘ λ -stabilizer’ and Cr is known as ‘ δ -stabilizer’, and hence their ratio determines the ‘stable’ phase(s) at the end of solidification. Thus, for a conventionally manufactured 316L alloy with a Cr_{eq}/Ni_{eq} ratio of 1.35, the expected final composition would comprise $\lambda + \delta$. However, in practice it was found that at high cooling rates, the suppression of solute distribution results in a transition that is fully ferritic or fully austenitic [89]. In fact, many SLM-based research on this alloy only report observing a λ -Fe phase [16, 90–93].

Table 2.3: The solidification modes, equivalent Cr_{eq}/Ni_{eq} ratios, and phase transitions in 300-series steel based on Schaffeler’s investigation [87]. It can be seen that depending on the Cr_{eq}/Ni_{eq} ratio, the solidification modes can vary between fully austenitic (A), austenitic-ferritic (AF), ferritic-austenitic (FA), or fully ferritic (F).

Solidification mode	Cr_{eq}/Ni_{eq}	Phase transition
A^*	≤ 1.25	$L \rightarrow (L + \lambda) \rightarrow \lambda$
$AF^\dagger$	$1.25-1.48$	$L \rightarrow (L + \lambda) \rightarrow (L + \lambda + \delta) \rightarrow (\lambda + \delta)$
$FA^\P$	$1.48-1.95$	$L \rightarrow (L + \delta) \rightarrow (L + \delta + \lambda) \rightarrow (\delta + \lambda)$
$F^\S$	≥ 1.95	$L \rightarrow (L + \delta) \rightarrow \delta$

* Fully astenitic

† Austenitic and ferritic

¶ Ferritic and austenitic

§ Fully ferritic

The microstructure constituents of SLM-manufactured 316L are easily distinguished from traditionally manufactured 316L due to the striking dissimilarity. To be precise, the SLM-manufactured 316L exhibits a hierarchical microstructure whose length scale spans six orders of magnitude [2] (i.e., includes solute segregation, solidification structures decorated with high density dislocations and nano-inclusions, LAGBs and, HAGBs). Thanks to this unconventional microstructure, SLM-manufactured 316L exhibits greater strength while also sustaining ductility, allowing it to break away from the traditional strength-ductility banana curve (see Figure 2.15).

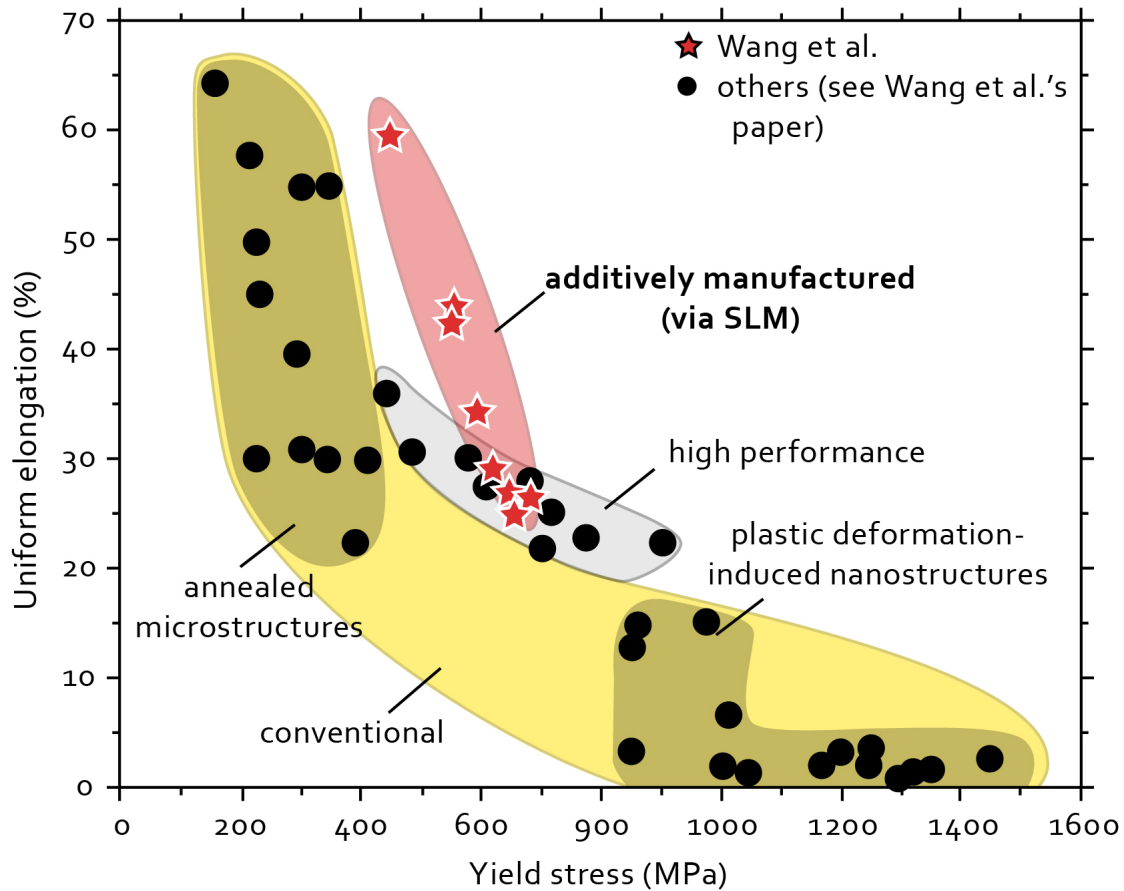


Figure 2.15: SLM-manufactured 316L stainless steel breaks the strength-ductility trade-off (banana) curve.

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Although not exclusive to 316L stainless steel, alloys manufactured via SLM tend to exhibit significant anisotropic behavior. This anisotropy stems from two components: 1) the morphology of grains and their underlying solidifications modes and 2) the crystallographic texture. Both of these can vary significantly based on the employed SLM parameters and the chemistry of the utilized alloy (phases, Bravais lattices, etc.).

It is only logical that the solidification in SLM-manufactured 316L would be different than its conventional counterpart due to the high cooling rates in SLM [3]. In fact, Wang et al. [94] reported two simultaneous solidification modes in SLM-manufactured 316L which are caused by the instability in thermal gradients and heat flux directions. They observed that the bottom of the melt pool exhibits a planar mode which quickly transitions into a cellular-dendritic mode upon moving along the building direction (BD) (see Figure 2.16). Furthermore, they reported that the cellular dendrites at the top of the melt pool re-align themselves along laser scanning direction (SD) (i.e., the direction of maximum heat flux). The complex solidification that occurs in SLM-manufactured 316L gives rise to non-equiaxed grains (i.e., grains whose size vary depending on the section with respect to the melt pool) (see Figure 2.16)

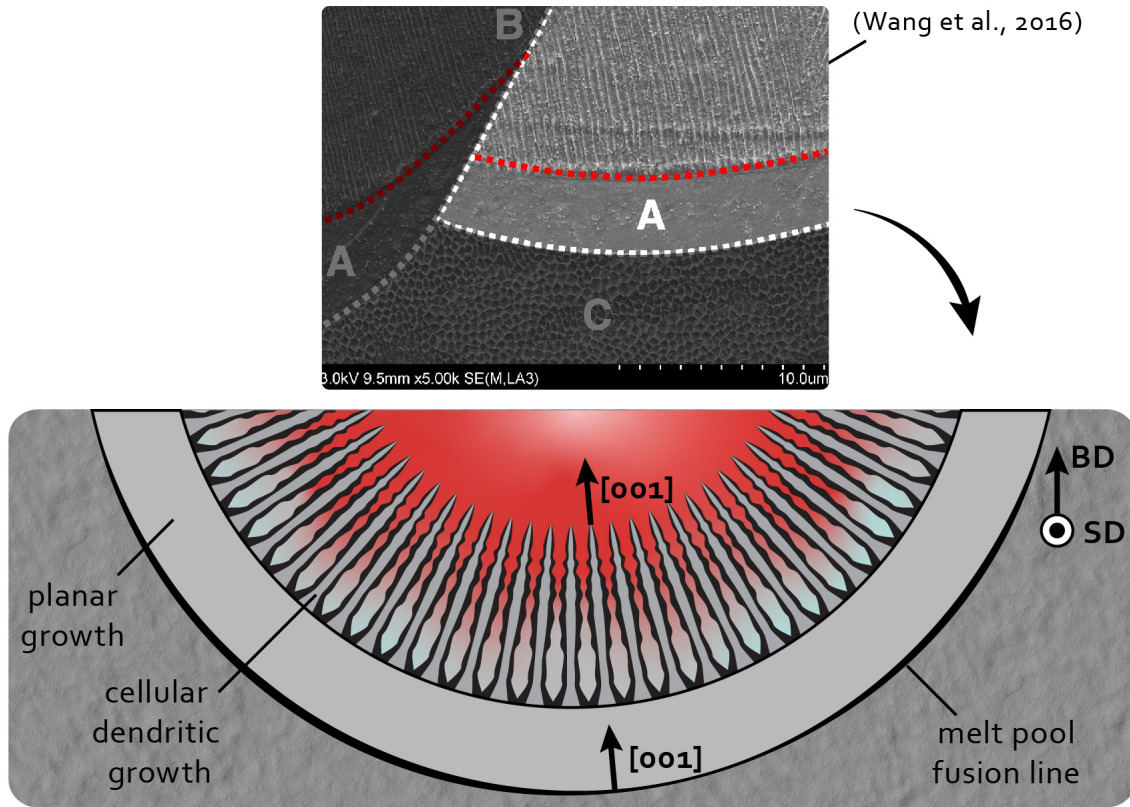


Figure 2.16: A cross-sectional view of the melt pool showing two solidification growth modes that occur in selective laser melting: planar (bottom section) and cellular-dendritic (top section).

Permissions: Top SEM micrograph was resued with permission from Elsevier: Materials & Design, Ref. [94], © 2016.

The preferred solidification growth direction in the FCC austenite occurs along the $\langle 001 \rangle$ edges of the unit cell [8]. Because the thermal gradient is perpendicular to the curved melt pool boundary [95], a set of $\langle 001 \rangle$ align themselves with that direction (Figure 2.17). The other set of $\langle 001 \rangle$ directions are aligned with the maximum heat flux (the laser SD, i.e., into the screen). The curved edges along the sides of the melt pool cause the G-aligned $\langle 001 \rangle$ cellular dendrites to have a 45° angle with the BD's axis (Figure 2.17). In other words, these inclined cellular dendrites have a (110) plane parallel to the BD axis. This is a well-known component referred to as the Goss component which is defined by the miller notation as $(110) \parallel \text{BD} \langle 001 \rangle \parallel \text{SD}$. On the other hand, the slender region confined to the center of the melt pool typically has its G closely aligned with the BD's axis, and thereby has a set of $\langle 001 \rangle$ directions aligned in that direction (Figure 2.17). The other set of $\langle 001 \rangle$ directions for this slender region are, similar to the previous one, aligned with the laser SD). In other words, these cellular dendrites have a (100) plane parallel to the BD axis. This (100) is a well-known component referred to as the Cube component which is

defined by the Miller notation as $(001) \parallel \text{BD}$ $\langle 001 \rangle \parallel \text{SD}$. It is typically found that SLM-manufactured 316L exhibits a strong Goss texture and a weak Cube texture [90]. Pham et al. attributed the Cube texture to the continuous epitaxial growth which occurs near the melt pool's centerline, whereas they attributed side-branching (of cellular dendrites) for the broadening of the Goss-texture grains [16]. Nonetheless, they also acknowledged that epitaxial growth may be interrupted due to perturbations caused by changing heat flux [16]. Thus, the SLM process parameters which impact the thermal profiles can significantly impact the strength of these observed textures.

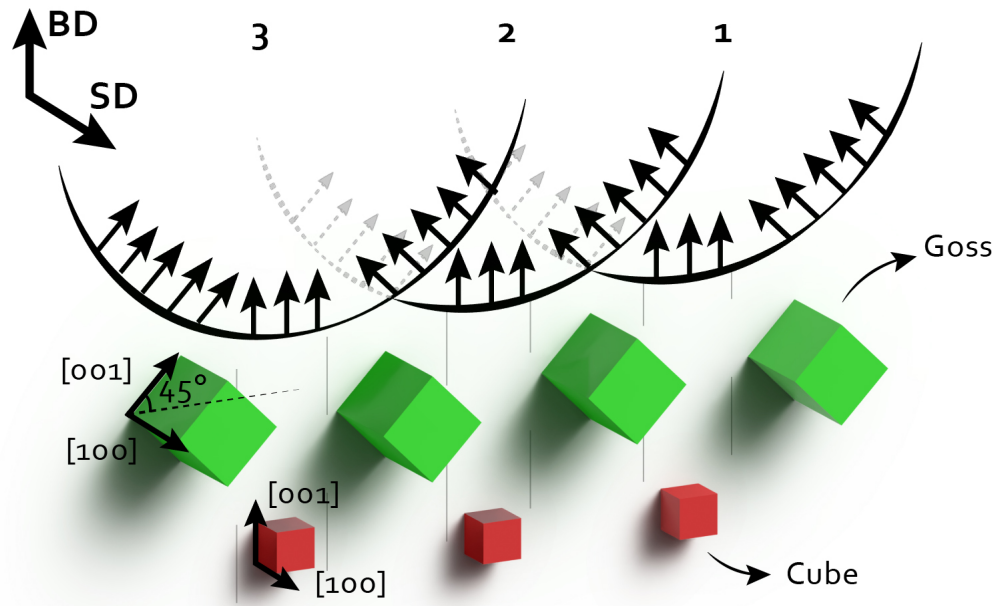


Figure 2.17: Texture development as a result of sequential laser scans (1 to 3). It can be seen that a Goss component develops along the curved parts of the melt pool, whereas a cube component develops in the narrow area along the centerline. The distance which separates the centerlines of two melt pools is known as the hatch spacing distance. This is one of the parameters that can be controlled to influence the texture development. In this example, it is assumed that the hatch spacing, along with other parameters, do not result in disrupting the Goss/cube texture components.

With careful control of the SLM parameters, (e.g., laser scanning strategy, speed, power, etc.) the texture(s) can be modulated accordingly [90, 96–98]. This modulation has a significant impact on the anisotropic behavior of the material. In fact, recent papers [99, 100] reported a successful single-crystalline-like, SLM-manufactured 316L obtained via precise control of the SLM process parameters. Other fascinating contributions such as Sofinowski et al.'s [101] involved adjusting the SLM process parameters mid-print to deliberately induce sequential single-crystalline-textured layers in 316L component via a technique they coined as layer-wise engineering of grain orientation (LEGO). From these efforts it became evident that SLM-based manufacturing has much more to offer beyond just design freedom; rather, this technology paves the way for producing materials with engineered material properties through a deliberately designed microstructure.

2.4 Additive manufacturing in tribology of metals

2.4.1 Broad view on the field

Assessing the progress of AM-based tribological research can be an overwhelming task for a number of reasons. First, metal-based AM alone consists of 14 different technologies [102]. Each of these technologies uses different means to achieve the same end; for example, some are powder-bed systems, others are powder-feed systems, and others are wire feed systems. As such, their alloy manufacturing capabilities differ based on their inherent set up. Nonetheless, even when two of these technologies produce the same alloy, one cannot simply infer the tribological properties of one from the other, because the various means of manufacturing produce different microstructures whose impact on the tribological properties cannot be underestimated. Another complicating factor is the tribological setups themselves (pin-on-disc, cylinder-on-block, block-on-block, etc.) as well as the conditions (load, speed, environmental conditions, temperatures, etc.). These setups and conditions promote different wear mechanisms, which typically impact the tribological performance. In fact, even swapping the tribological pair in a pin and disk (from A on B, to B on A) can significantly change the wear behavior [103]. Simultaneously, the wear mechanisms are correlated with the underlying microstructure which is impacted by the type of AM technology and the processing parameters. Thus, although the first article on the tribology of an AM metal was published more than 15 years ago [4], the field is still considered nascent as researchers' efforts scatter amid all the aforementioned complexities.

Figure 2.18 shows the number of publications since the inception of the field to 2018, totaling 48 publications. Noting that the scope of literature gathered covered all tribological studies conducted using any AM technology involving metal alloys and metal matrix composites (MMCs). The number of publications has seen a sudden increase in 2018 and continues to grow today, which indicates that tribologists have started to acknowledge the merits of AM within their field. Nonetheless, a closer look into the intricacies reveal that the efforts scattered across multiple AM technologies, alloy-classification, and materials – See Table 2.4. Furthermore, researchers utilized various tribological set-ups and conditions which also add up to the complexities (the reader may wish to consult the individual papers in Table 2.4 for more details on this aspect). Nonetheless, the table also shows that SLM-related papers are more prominent than other AM technologies.

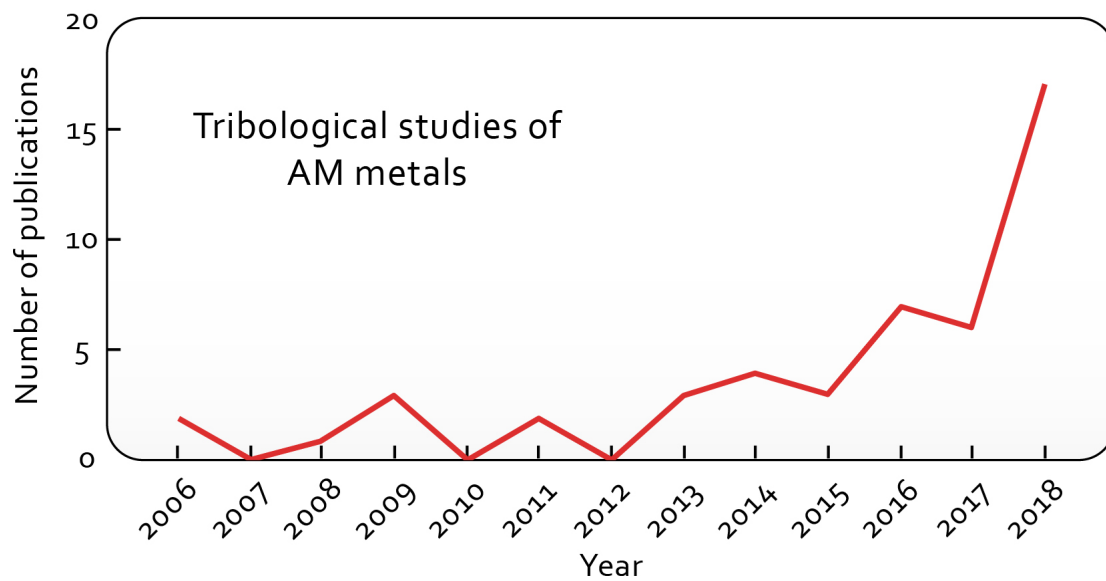


Figure 2.18: Published papers on the tribology of AM metals since inception (2006) to take-off year (2018).

Of the SLM-based research, the largest portion of publications (44%) was on Fe-based alloys, followed by MMCs (25%), and finally all others (31%) (Figure 2.19a). Scrutinizing the Fe-based publications reveals that 79% of which is on 316L stainless steel (Figure 2.19b). This breakdown shows that tribology research on 316L additively manufactured by selective laser melting is indeed the most prominent compared to all the other technology-material combinations.

The following subsection will serve as an in-depth investigation of the literature on SLM-manufactured 316L within the context of tribology.

Table 2.4: Survey of AM technologies, alloy classification, and material type used for tribological research papers between 2006 and 2018.

Technology	Classification	Material(s) [reference]
DMLS [¶]	MMC	(Fe, Ni)-TiC [4]; Fe-Si [104]; Fe+SiC [105]; WC [106]; AlSi10Mg-TiB ₂ [107]
	Ti-alloy	Ti6Al4V [108]
EBM [¶]	CoCrMo-alloy	CoCrMo [109]
	Ti-alloy	Ti6Al4V [108, 109]
LENS [¶]	CoCrMo-alloy	CoCrMo [110]
	MMC	CaP-Ti6Al4V-N ₂ [111]
	TiAl-alloy	TiAlCrNb [112]
	Ti-alloy	Ti6Al4V [113]
LFR [¶]	Fe-alloy	34CrNiMo6 [114]
LMD [¶]	CrNiSi-alloy	Cr13Ni5Si2 [115]
	CuAl-alloy	CuAl10Fe1 [116]
	MMC	MLG+Ni3Al [117]; Ni-Cr-Si-B-Fe [118]
	Ni-alloy	IN718 [119]
SLM	Al-alloy	AlN/AlSi10Mg [120]; AlSi12 [121]
	CoCrMo-alloy	CoCrMo [122]
	Fe-alloy	1.2709 tool steel [122]; 316L [5, 64, 122–128] [*] ; 316L+C, B, V, Nb [129]; M50 steel [130]; Ni-steel [131]; Fe(?) [132] [†]
	MMC	Ti [133]; TiC/Ti [63, 134]; SiC/AlSi10Mg [135]; TiC/AlSi10Mg [136]; WC-reinforced Ni-steel [137]; Al [138]; Graphene/Inconel 718 [139]
	Ti	commercially-pure Ti [140]
	Ti-alloy	Ti6Al4V [122, 141, 142]
	TiC-alloy	TiC [64]
	W-alloy	TiC-reinforced W [143]
SLS [¶]	Fe-alloy	DirectSteel (EOS GmbH) [144]
	MMC	SiC/Carbon Fiber [145]; Polymer-coated steel [122]

[¶] The reader may wish to consult the glossary at the end of the thesis for the precise definition of this term.

^{*} Although reference [128] reports employing "SLS", the technology used was in fact SLM.

[†] Note: Although reference [132] reports employing "DMLS", the technology used was in fact SLM.

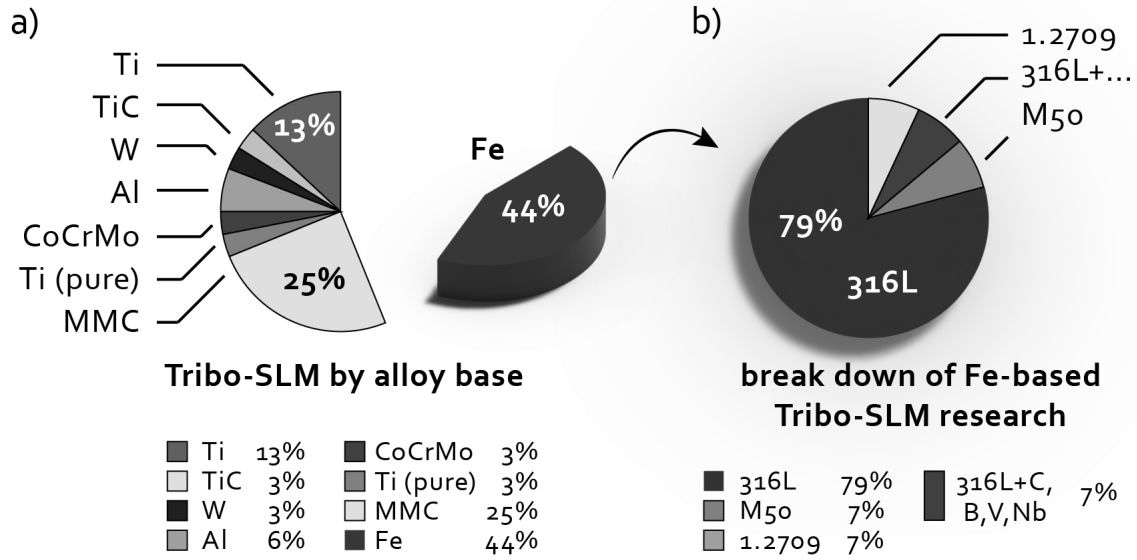


Figure 2.19: Breakdown of Tribo-SLM research by base alloy shows that Fe is the most popular, comprising 44% (a). Majority of the Fe-based research (79%) was conducted using 316L stainless steel (b).

2.4.2 Tribology of SLM-manufactured 316L stainless steel

In the previous section we saw that 316L stainless steel is the most prevalent material in tribological research using SLM technology. This section will provide an in-depth discussion on the tribological research related SLM-manufactured 316L. For the sake of readability, this subfield will be referred to as ‘Tribo-SLM316’ from here onward.

The author examined the literature since the inception of Tribo-SLM316 to when the field started to take-off, specifically covering 2013 to Q3-2020 as shown in Figure 2.20. The number of Tribo-SLM316 papers published in 2020 alone was almost equal to the sum of those published in the preceeding seven years.

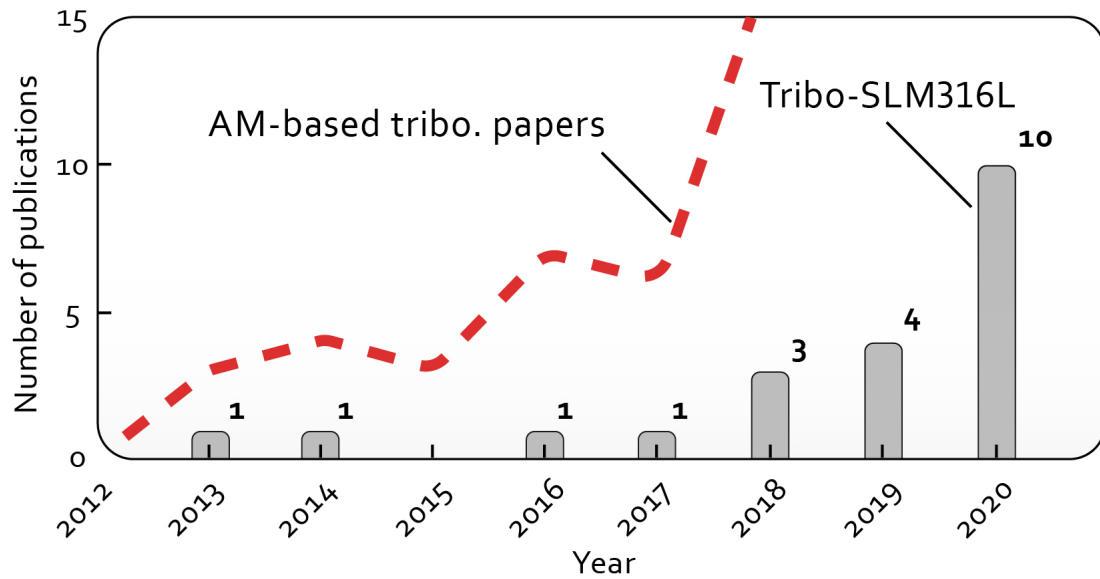


Figure 2.20: Tribo-SLM316 research since the first published paper (2013) to take-off year (2020).

The most important experimental conditions have been extracted from the Tribo-SLM316 papers and collated in Table 2.5 below. The table compares various experimental aspects, for example, highlighting the studies in which conventional- and SLM-manufactured 316L were investigated side-by-side, the studies in which the tested SLM-manufactured 316L was in its as-built condition, and even the studies' choice of material for the counter body. These parameters are not comprehensive, as they do not reveal some intricacies such as the contact stresses, speeds/frequencies, etc. The author left it to the reader to extract such data if they wish. Nonetheless, if we were to rely on the available literature to understand the tribological behavior of SLM-manufactured 316, then it would be crucial to piece together any relevant knowledge to help reach this understanding. Equally important is the ability to distinguish between which of the available literature is pertinent to achieve this understanding. One of the commonly used practices to understand a new material is to compare its behavior against a reference material with well-known properties. Therefore, the author deliberately chose this criterion as the first category upon which the literature can be divided. The following three categories (as-built comparison, ambient, and temperature) are also crucial for comparison as the addition of lubricant, modification of the alloy, and the use of high/low temperature environments add significant complexity to the problem at hand. The remaining categories (motion, configuration, and counter body) may be selected based on which is the closest approximation to the real-life tribological set-up, or purely based on the available resources.

Table 2.5 shows that almost half of the published literature did not compare the tribological behavior of SLM-316L side-by-side with conventionally manufactured 316L. Those that did compare the two materials under dry, as-built, and room temperature conditions are [6, 82, 126, 146–150]. Nonetheless, the more we move toward the right side of the table, the more the experimental parameters deviate from one another. In fact, none of the published work used the same material for their counter body (let alone the parameters which the author did not present in the table such as speeds, hertzian contacts, etc.). Adding to the complexity are the variation of SLM process parameters and the specific manufacturing route for the conventional 316L (e.g., forged, rolled, cast, etc.). Thus in reality, none of these studies' experimental conditions were identical, but some do share more similarities than others.

Table 2.5: Comparing the experimental parameters used by various references on tribological research concerning SLM- and conventionally manufactured 316L. The table provides an appreciation of the breadth of parameters used, despite the field's nascency.

SLM vs. conv.	As-built compar- ison	Ambient	Temp. (°C)	Motion	Configu- ration	Counter body	Ref.
Compared	Yes	Dry	Room temp.	Linear	Ball-on-plate	Steel (100Cr6)	[147]
						WC	[150]
					microscratch	Diamond	[82]
				Rotary	Ball-on-disk	Stainless steel (?)	[126]
						Steel (52100)	[6]
						Steel (G10450)	[148]
			Room temp., 200-600	Linear	Ball-on-plate	Al ₂ O ₃	[149]
			Room temp., 300-600	Rotary	Ball-on-disk	Al ₂ O ₃ , Si ₃ N ₄	[146]
		Lub.	37	Linear	Ball-on-plate	Al ₂ O ₃	[5]
			Not specified	Rotary	Ring-on-disk	38CrMoAl, Brass (H68)	[127]
		No	Not specified	Rotary	Ring-on-disk	PEEK	[151]
Not compared	N/A	Dry	Room temp.	Linear	Ball-on-plate	Steel (?)	[128]*
						Steel (E-52100)	[123]
						Zirconia	[152]
				Rotary	Microscratch	Diamond	[153]
							[154]
					Ball-on-disk	Al ₂ O ₃	[129]
				Linear	Flat-on-disk	Steel (EN32)	[155]
			Room temp., 200-600		Ball-on-plate	Steel (E-52100)	[124]
		Dry, Lub.	18, 37-38	Rotary	Ball-on-disk	Al ₂ O ₃	[156]
		Lub.	Not specified	Linear	Spool valve	38CrMoAl	[157]

* Note: Although reference [128] reports employing "SLS", the technology used was in fact SLM.

All of the following papers [6, 82, 126, 146–150] compared the dry wear performance (in specific wear rate) of the SLM-manufactured 316L to the conventionally manufactured 316L. However, the results varied between those who observed a better performance for the SLM-manufactured [82, 149], those who observed better performance for the conventionally manufactured [6, 147, 150], and those who observed similar performance [146, 148]. Noteworthy, these inconsistencies remain unaddressed, which makes it unclear whether SLM is a viable means for tribological applications. Based on the author’s review of the literature, these inconsistencies remain unaddressed because there is a lack of mechanistic understanding of how the underlying microstructure impacts the tribological behavior.

It is evident that attempts have been made to explain some of the differences in tribological performance between the two manufacturing routes. For example, Tascioglu et al. [150] argued that the net effect porosity has on the wear resistance is greater than that of the hardness. Akin to Tascioglu et al.’s thought, Sun et al. [126] argued that if porosity is eliminated, then the wear resistance of SLM-manufactured 316L can be as good as the conventionally manufactured 316L. Contrarily, Lanzutti et al. [149] believed that hardness was the reason why SLM-manufactured 316L exhibited better wear resistance than conventionally manufactured 316L. In addition, Yang et al. [82] observed that anisotropy plays a role in the wear resistance of SLM-manufactured 316L. And other researchers [6, 146, 148, 149] noted different wear mechanisms varying between adhesion, abrasion, and plastic deformation across the SLM- and conventionally manufactured specimens. These commendable efforts allude to the complexity of the problem at hand, and thus give rise to the need for developing a mechanistic understanding of the role of microstructure on the tribological behavior of SLM-manufactured 316L. With the limited number of available literature and the continuous branching into specialized research areas which raise the problem complexity (e.g., high-temperature [146, 149], lubricated [5, 127, 151], post-treatment [147, 150])), such mechanistic understanding remains lacking.

Developing a mechanistic understanding entails that we forfeit much of the existing focus on the performance and instead shift attention to relationships between the microstructure and the tribological behavior. This shift becomes especially important given the evidence that at least some level of tribological anisotropy exists in SLM-manufactured 316L [82]. Knowing that anisotropy is a by-product of the microstructure which, in and of itself, is a by-product of the SLM process parameters, means that a relationship exists between tribological anisotropy and the SLM process parameters. In other words, the SLM process parameters can be carefully selected to ‘adjust’ the anisotropic behavior of SLM-manufactured components; nonetheless, it is worth noting that the extent of such impact remains unclear. Deliberate tweaking of the microstructure to produce a multi-graded tribological surface would be a unique advantage of utilizing AM technologies over conventional routes for specialized tribological applications.

2.5 Remarks on the complexity of wear and friction in metal-metal sliding pairs

In light of the inconsistent findings surrounding Tribo-SLM316 (see section 2.4.2), the author believes that there is a genuine need to embark on studies that focus on mechanistic aspects of wear and friction. These mechanistic studies are especially important when comparing two different techniques such as SLM and any other conventional technique, as the nuances can help better explain the inconsistencies.

In a continuous effort to design better tribological systems, tribologists have spent decades relying on discerning the mechanisms by which wear ensues. In 1933, Ricardo [158] postulated a theory of corrosive wear. Then, the adhesive wear theory put forth by Archard in 1953 [159] has become widely accepted (although it is argued that Reye [160] had conceptualized a similar idea in 1860, which was lost in translation). In 1959, Goddard et al. [161] introduced the theory of abrasion of solids. And in 1973, Suh [162] produced an alternative to the adhesive model which he coined as the delamination wear theory. Today, additional mechanisms are reported in literature, such as erosion, surface fatigue, plastic deformation, etc. There are many instances across literature that the author can cite where one mechanism's name is used to describe another. Furthermore, there are plenty of examples highlighting lack of consensus on what scholars accept as the main mechanisms in wear. Perhaps these examples are indicative of some complex aspects in the fundamental understanding of wear that still linger to date.

Rigney [103] provided an excellent commentary on the sliding wear of metals as part of a special issue in *Tribology International*, highlighting the progress in the 50 years preceding 1997. In his paper, he noted how the development of one wear mechanism over another as well as the behavior of friction may drastically vary based on the composition of the tribo-pair, set-up, and environment. Therefore, he argued that linear models (e.g., Archard's [159]) constitute a vast oversimplification of a complex problem; or as he nicely put it, "... it is unrealistic to expect that a single wear equation can be developed to cover all cases - unless it contains so many parameters that it becomes impractical to use it".

Certain aspects of the microstructure (covered in 2.2.2) may lead to a sudden transition in wear or friction which is not captured via linear equations. Rigney [103] exemplifies using three types of stainless steel: 304, 310, and 316. Each one of these steels exhibits a different tribological behavior which is owed to the underlying microstructure. Despite being austenitic steels (i.e., 300-series), the Schaeffler diagram indicates (based on the wt.% of Ni_{Eq}) that the phase transitions for the three alloys are not identical. In a sliding experiment between tool steel and 310, the debris detected after the trial consisted of 310. Whereas in an experiment with tool steel and 304, the detected debris consisted of tool steel. While this may be counterintuitive given that tool steel is significantly harder than

304 (due to higher carbon content), the phase transition caused by sliding explains this result. As with any other alloy, sliding causes the 304 alloy to work-harden. This work hardening is exhibited in the form of recrystallization (by forming nano-crystallines in grain sizes as small as 3 nm) of the surface and near surface layers. However, due to 304's chemical composition, its worked interface, i.e., the nano-crystallines, suddenly experiences a phase shift to a martensitic (α' -Fe) BCC. The debris generated is mechanically mixed between the tribo-pair and is hard enough to abrade tool steel. The result of which may be experienced as a sudden shift in the coefficient of friction or the wear rate in any of the tribo-pair. Noteworthy, this α' -Fe transition can be inhibited if the temperature of the interface is increased. As for 316 (not to be confused with 316L which has a low carbon content), this sort of α' -Fe transition can occur after long sliding distances. These insights, which are essential in developing a predictable tribological system, are only captured if the nuances of the material are incorporated into the equation. Thus, it is only logical that linear wear equations fail to explain much of these phenomena.

A material's hardness is commonly used by tribologists to compare different materials and sometimes as means to explain certain tribological behaviors. Rigney discussed the role of hardness in sliding behavior more elaborately in a self-contained paper [163]. He argued that microcutting and smooth sliding are two extremes with a continuous spectrum in-between. The former is associated with abrasive wear where the latter is associated with sliding wear. Hardness in microcutting has a direct role if the hardness ratio between the tribo-pair is significant and the angle between them is convenient for establishing penetration. In this mode, the debris generated (usually in the form of chips or sheets) is mostly identical in composition to its parent material. On the other hand, hardness' role in sliding is elusive. Nonetheless, Rigney provided a valuable insight based on experimentally obtained correlation pertaining to the hardness of the tribo-pair. In pin-on-disk setups, the hardness ratio of the disk to the pin (H_{disk}/H_{pin}) plays a significant role in determining the severity of wear. He stated that mild wear and smooth sliding are dominant when H_{disk}/H_{pin} is greater than one throughout the experiment. Whereas severe wear and rough sliding are dominant when H_{disk}/H_{pin} is less than or equal to one, whether at the beginning or at a sudden transition during sliding. Bowden and Tabor [164] suggest that the critical ratio close to 0.8. While these hardness-ratio insights are remarkably useful, the author believes that they should be used with care. Because the hardness ratio is susceptible to sudden change due to many factors (e.g., work-hardening, mechanical mixing, or environmental changes [163]), the author notes that this ratio should always be used within the context of the whole tribological system and the microstructural features of the tribo-pair.

The environment of a tribological system is known to influence the wear and friction behaviors. Rigney's [103] examples from literature showed that an experiment in vacuum produces entirely different results than in atmospheric conditions. He provided several material examples such as dual-phase steels, Nickel aluminide, and Pb-Sn. The examples

demonstrated that the coefficient of friction is often lower in vacuum than in air. Even wear can exacerbate in two orders of magnitude in air than in vacuum. Similarly, the author notes that variations in temperature, humidity, lubricant composition, and lubrication regimes can significantly impact the tribological behavior.

The aforementioned complexities serve to highlight the need for contextualizing the individual factors (hardness, environment, material's composition, etc.) into the entire tribological system/study. The interplay between these complexities is crucial in developing key mechanistic insights into how the Tribo-SLM316 is different than its conventionally manufactured counterpart.

2.6 Chapter conclusion

It is undoubtable today that metal additive manufacturing (AM) technologies such as selective laser melting (SLM) can produce materials with unconventional properties. These material properties are directly correlated with the constituents of the underlying microstructure. The high cooling rates and SD-dependant solidification in SLM give rise to a heterogeneous hierarchical microstructure with controllable crystallographic texture, respectively. Among the SLM materials that quickly rose through the ranks of academic research is 316L stainless steel due to its excellent corrosion resistance, high ductility, and biocompatibility. As a result, the SLM-induced microstructure of this alloy been intensively studied (compared to many other SLM alloy choices) to a point where the crystallographic texture can be modulated to mimic the highly anisotropic single-crystal. In addition, this alloy (unlike many others) consists of a simple fully austenitic single-phase, face-centered cubic lattice. For all these reasons, 316L is an ideal alloy choice for venturing into nascent research areas involving complex interactions. It was covered that the tribology of SLM-manufactured components is one such nascent area where our understanding is lacking. This lack of understanding is noticeable among inconsistencies in the limited existing literature comparing the tribology of SLM to conventional routes. Some studies claim that SLM's tribological performance is superior, while other studies claim the opposite. In addition, other experiments showed that the difference between the two routes is marginal. These inconsistencies call for developing a mechanistic understanding into the relationship between SLM-induced microstructure and the tribological behavior. In order to achieve this understanding, the complexities induced by the tribological systems and their nuanced interaction with the microstructure must be acknowledged and accounted for into the analysis of the results. This may mean that we should rather focus on simplifying the experimental set-up as much as practically possible. If this key insight becomes clearer, then it would be possible to exploit SLM's ability of controlling the microstructure to produce a multigrade tribological surface which exhibits site-specific tribological properties for specialized applications.

Chapter 3

Experimental methods and design

3.1 Chapter introduction

This chapter is devoted to the experimental methods and design used to meet the research objectives of developing 1) a mechanistic understanding of the microstructure's role on the tribological behavior of SLM alloys, and subsequently 2) a novel multigrade tribological surface via SLM. The experimental designs are elaborated upon in sections 3.3 (first objective) and 3.4 (second objective). For context, the reader is encouraged to read those two sections first; and use section 3.2 as supplementary, in case much detail about a specific technique or equipment is sought.

3.2 Equipment and methods

3.2.1 SLM machines

Two SLM machines were employed for this project, the first is located at Imperial College London and the second is located at Nanyang Technological University (NTU). The Imperial-machine's model is Mlab Cusing R, manufactured by Concept Laser (Figure 3.1a). This machine is capable of printing many alloys including stainless steels and titanium alloys. Nonetheless, this machine is strictly fed with 316L alloy powder to minimize the likelihood of cross contamination across subsequent builds. On the other hand, the NTU-machine's model is SLM 500, manufactured by SLM Solutions (Figure 3.1b). Unlike the previous machine, this one is equipped with a twin laser by default with higher power output. The key attributes of both machines are listed in Table 3.1 below (full technical specifications for the printers can be found in Appx. 1 and Appx. 2).

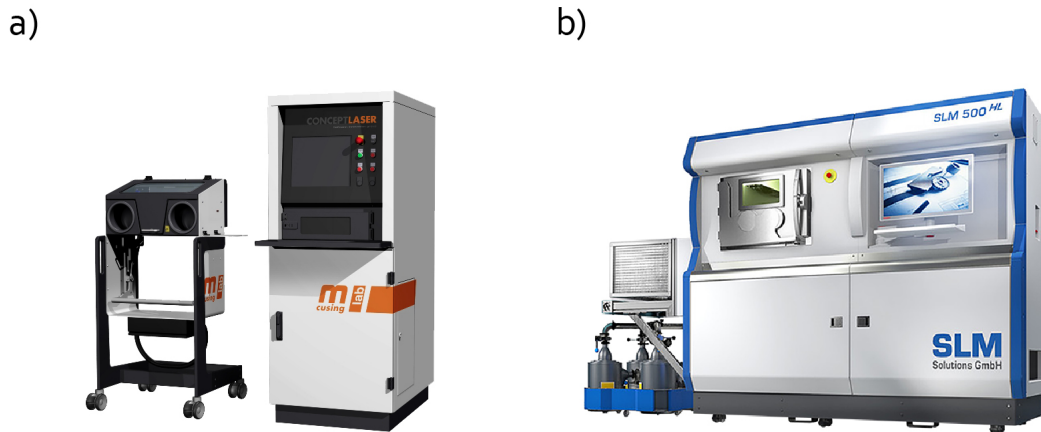


Figure 3.1: The SLM equipment used in the project. (a) concept machine, and (b) SLM 500 machine.

Table 3.1: Technical specifications of the utilized SLM equipment.

	Mlab Cusing R	SLM 500
Laser type	fiber (continuous)	fiber (continuous)
Max. laser power (W)	100	400
Max. laser speed (mm/s)	7,000	10,000
Layer thickness (μm)	15–50	20–90
Spot diameter (μm)	≈ 50	≈ 80 –115
Inert gas type	Argon*	Argon

* This attribute is for the sourced machine, although other options are available

3.2.2 Scanning electron microscopy techniques

Although the project initially utilized a scanning electron microscope (SEM) by Hitachi (S-3400), this SEM was later replaced/upgraded with two field emission gun SEMs (FE-SEM) by TESCAN: Mira 4 and Clara 4. Unlike its predecessor, the FE-SEM has a resolution of 1.2 nm at 30 keV in high vacuum mode. Furthermore, the FE-SEM is equipped with a wide range of detectors which were employed throughout this project, each making a valuable contribution to the characterization. These detectors are scanning electron (SE), electron backscatter diffraction (EBSD), energy-dispersive x-ray (EDS/EDX), and backscattered electron (BSE). The high-resolution spot size offered by the FE-SEM was utilized in the BSE mode to achieve electron channeling contrast imaging (ECCI). All these techniques, beside the conventional SE, are further elaborated upon below.

EBSD plays a pivotal role in this project as it provides a quantitative crystallographic analysis, such as, crystal orientation (texture), grain size, phase identification and distribution, boundary characterization, and localized strains. The advantage of this technique over X-ray crystallography (XRD), is that it provides site-specific data by scanning a grid within the field of view. This technique relies on transcoding a certain type of electrons (namely, the diffracted backscatters) into crystallographic data. The way it works is by tilting the sample with respect to the incident electron beam to allow a characteristic electrons to be captured by the detector (Figure 3.2a). The characteristic electrons are a subset of backscattering (escaping) electrons that satisfy the Bragg condition; these electrons are referred to as the diffracting electrons. Because a crystal has many (hkl) planes whose angles with respect to the beam incident are different, some will satisfy the Bragg condition, and some will not. A plane that satisfies the Bragg condition will diffract electrons in a conical shape rather than a single point. This diffraction occurs orthogonally on both sides of the plane, forming a top (excess) and a bottom (deficit) cones. The sections of the cones that intersect the phosphor screen form what is known as the Kikuchi band (Figure 3.2b). This Kikuchi band has a width equal to two times the incident angle [165]. In a similar fashion, all other Bragg-satisfying planes will form Kikuchi bands, making the pattern seen in Figure 3.2c. Together, the bands can be indexed to give crystallographic in-

sight such as phase, orientation, etc (Figure 3.2d). Noteworthy, EBSD requires a carefully prepared smooth surface in order to increase the intensity of the Kikuchi bands. Throughout this thesis, the 316L samples were prepared for EBSD following the protocol in Appx. 3.

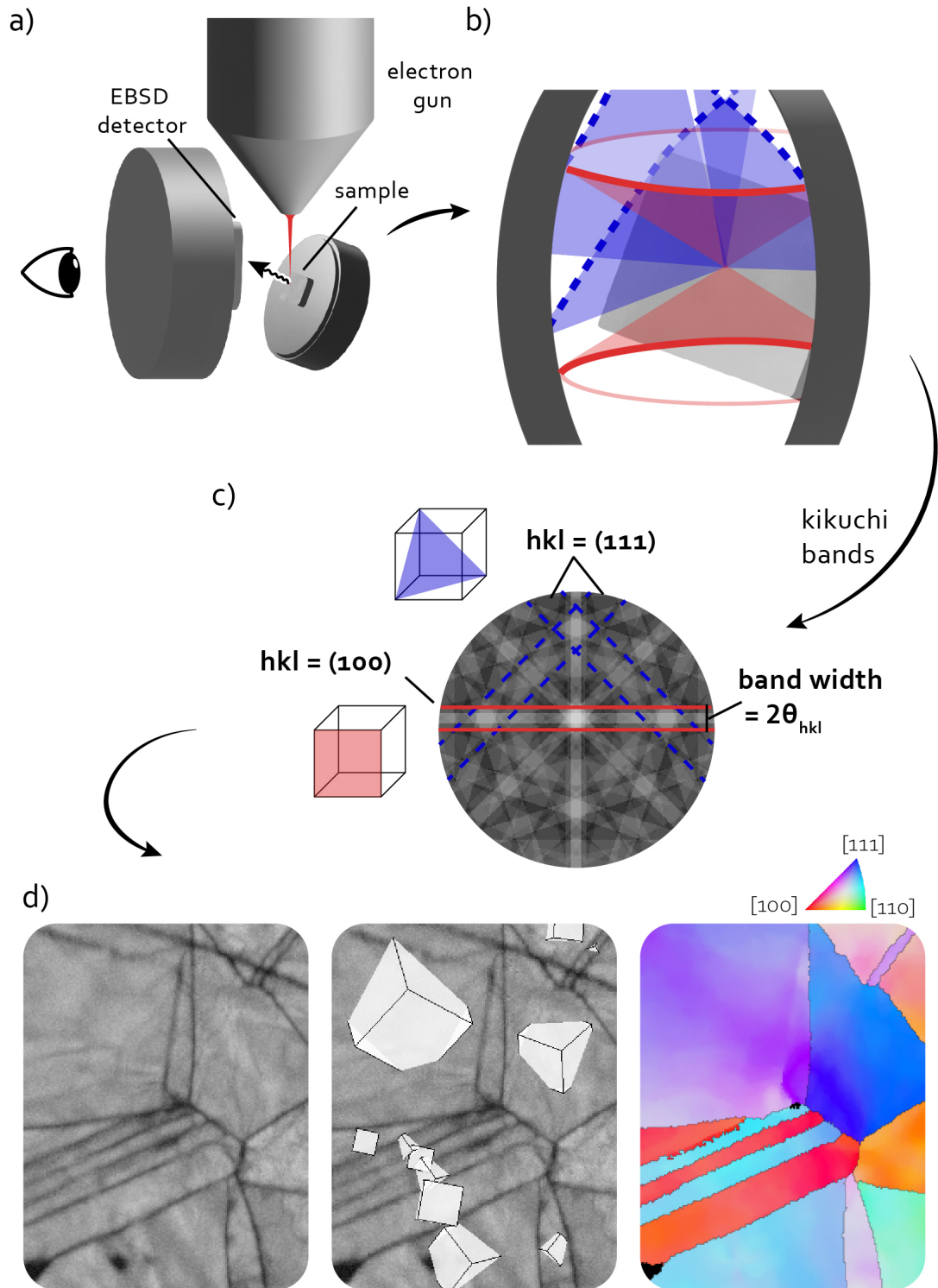


Figure 3.2: Working principle of the EBSD technique. (a) Sample position inside the SEM chamber in relation to the EBSD detector and the electron gun (i.e., at 70° tilt). (b) View of the sample from the perspective of the EBSD detector shows the Kossel cones of Bragg-condition satisfying planes. The red cones correspond to the excess and deficit cones of the (100) planes, whereas the blue cones correspond to the (111) planes. (c) As each pair of cones intersect the phosphor screen of the detector, Kikuchi bands appear which collectively help index the specific crystal.

EDS (or EDX) provides an important insight into the elemental composition of features such as wear track or microstructure constituents. This technique relies on detecting the characteristic energy associated with the chemical elements comprising the specimen. Unlike in the EBSD technique, the specimen in this technique does not need to be tilted, as the EDS detector is conveniently inserted at an angle between the specimen's surface and the gun's axis (Figure 3.3a). The technique works by exciting the specimen's electrons via the incident beam's high energy. The excitement process will 'knock off' electrons from their orbital/shell, creating an empty space and a positive charge. To balance this charge, electrons from other orbitals will occupy this empty space, resulting in a release of a characteristic energy. With a continuous bombardment of electrons, the EDS detector collects characteristic energies associated with the chemical elements comprising the specimen (Figure 3.3b).

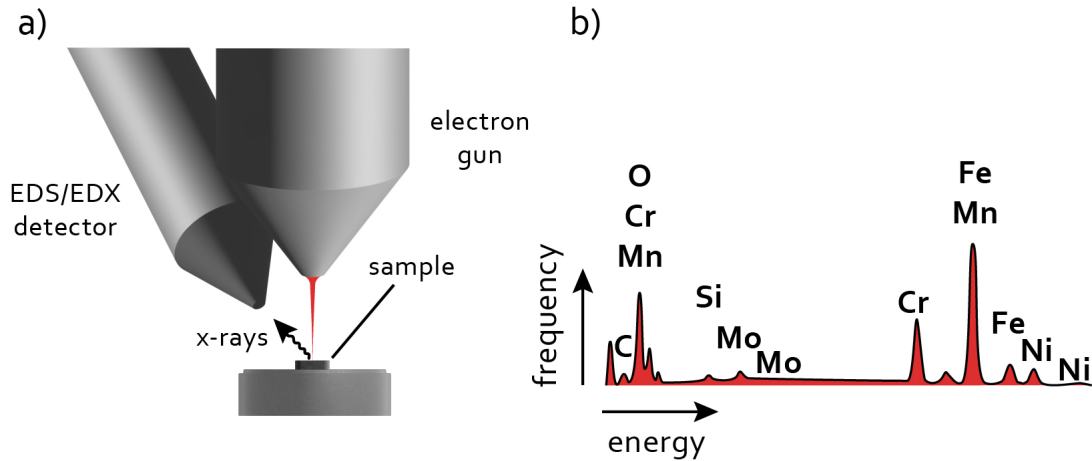


Figure 3.3: Working principle of EDS/EDX technique. (a) Sample position inside the SEM chamber with respect to the EDS detector and the electron gun. (b) the energy released by each element present in 316L stainless steel alloy.

BSE was used primarily for performing ECCI technique to visualize defects at high resolution. ECCI is a powerful SEM technique which can give some functionality of transmission electron microscopy (TEM), but without the need for destructive preparation of the sample (i.e., extracting a thin foil). Through this technique, dislocations and stacking faults can be observed [166]. However, this technique is limited to near-surface defects. Apart from BSE, a decent ECCI requires a beam with high brightness, and a small beam convergence (spot size), which is available in the FE-SEM [167]. The technique works by 'channeling' electrons through a crystal lattice as shown in Figure 3.4. Channeling is synonymous with establishing a Bragg condition for a specific lattice plane (hkl) (also referred to as two-beam condition). Upon doing so, a striking contrast is visible on the BSE image between the grain and the near-surface defect(s). Primarily because channeled electrons are can hardly backscatter due to the alignment with the incident beam compared with the (misaligned) defect. Nonetheless, establishing a deliberate (hkl) Bragg condition is difficult without certain capabilities such as beam or stage rocking (which not available in Mira 4). In the absence of such capability, channeling is possible via trial-and-error.

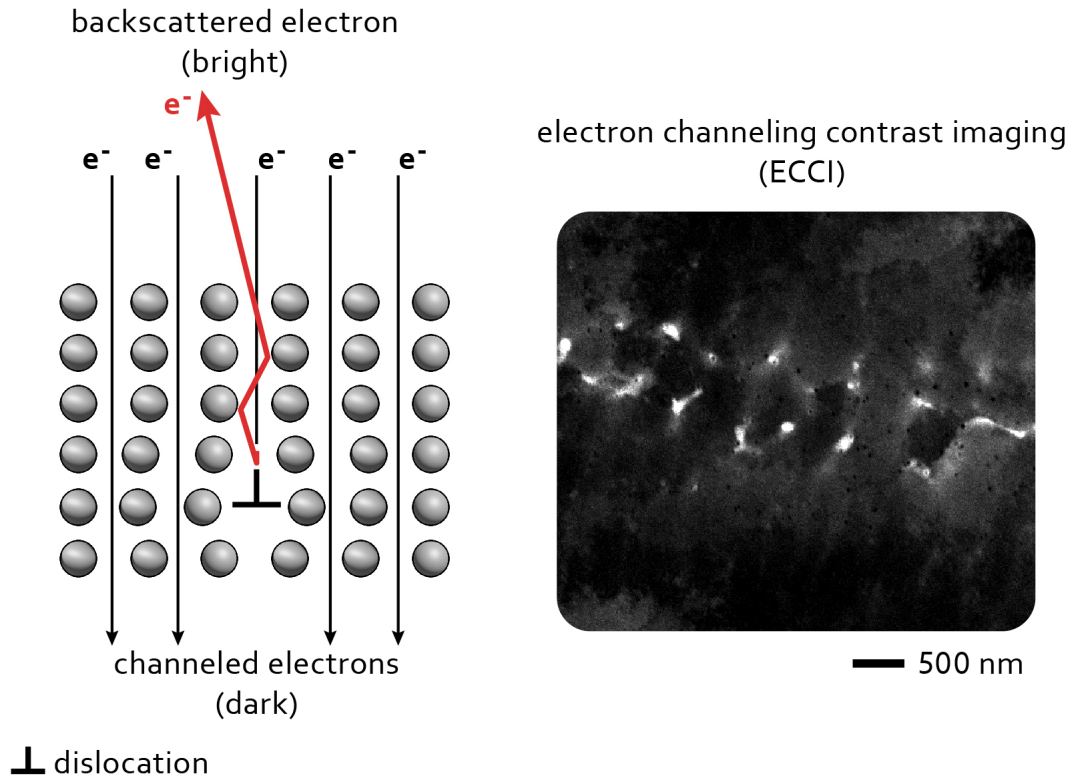


Figure 3.4: The working principle of the ECCI technique. Crystals which are aligned in their Bragg condition absorb majority of the incident beam's electrons and thus appear dark. Whereas dislocations cause a portion of the beam's electrons to backscatter (escape) and thus emit bright contrast.

3.2.3 Surface topography techniques

To characterize the specimens' surfaces and wear tracks, a quantitative three-dimensional optical microscope was used (also known as non-contact profilometer). The equipment's model is Wyko NT9100 by VEECO, whose working principle is white-light interferometry (WLI). WLI relies on LED sources, optics and a beam splitter, a camera, and a microscope objective to establish interferometry. Using these elements in conjunction with a motorized stage, accurate vertical and horizontal maps of the surface can be constructed. This model has a vertical and optical resolution of ≤ 0.1 nm and $0.49 \mu\text{m}$, respectively. The full technical specifications for this machine can be found in Appx. 4.

3.2.4 Tribometers

Several tribometers were employed for this project, located at Imperial College London and the National Physical Laboratory (NPL). The Imperial-tribometer is a linear high-frequency reciprocating rig (HFRR) by PCS instruments (Figure 3.5). The HFRR establishes point contact via a ball-on-flat set-up, wherein the upper specimen has a diameter of 6 mm and the lower specimen has a diameter and height of 3.5 mm and 10 mm, respectively. Furthermore, this HFRR has a frequency and stroke ranges of 10-200 Hz and 20-2000 μm , respectively. On the other hand, one of the NPL-tribometers (Figure 3.6a) is a custom-designed rig for performing in-situ SEM microscratch tests [168]. The benefit of performing in-situ microscratch testing is the ability to capture successive SE snapshots which show the damage build-up and microstructural evolution in multi-scratch tests. In addition, the NPL-tribometer can measure the applied normal force and the coefficient of friction (CoF) with the aid of two capacitance plates and a flexure element (designed by Smith et al. [169]). The former allows for accurate control of the applied force via a feedback loop, while the latter allows for correlating the damage build-up with the CoF measurements. The scratcher employed for this project consisted of a sphero-conical (blunt cone) tip half-apex angle of 45° (Figure 3.6b). Scratchers came with two options of radii, 1 μm and 5 μm . Another NPL-tribometer, used for macro-scratch testing, was manufactured by Teer coatings ltd. (model ST-3001). This tribometer employed a Rockwell C-type diamond tip (Figure 3.7).

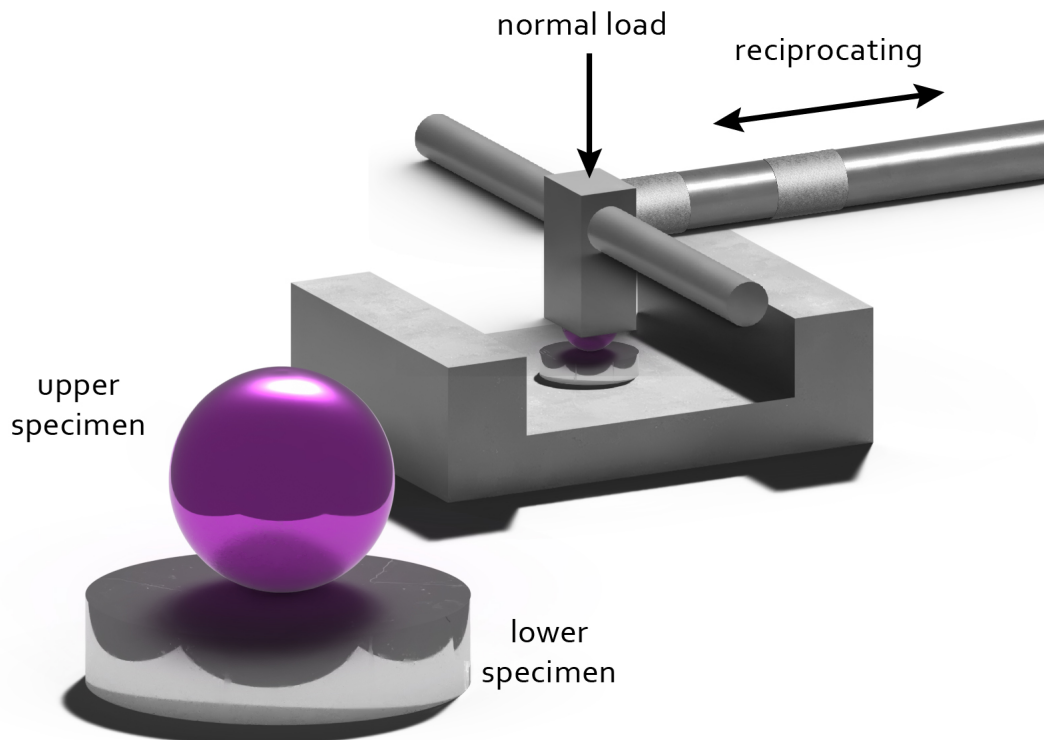
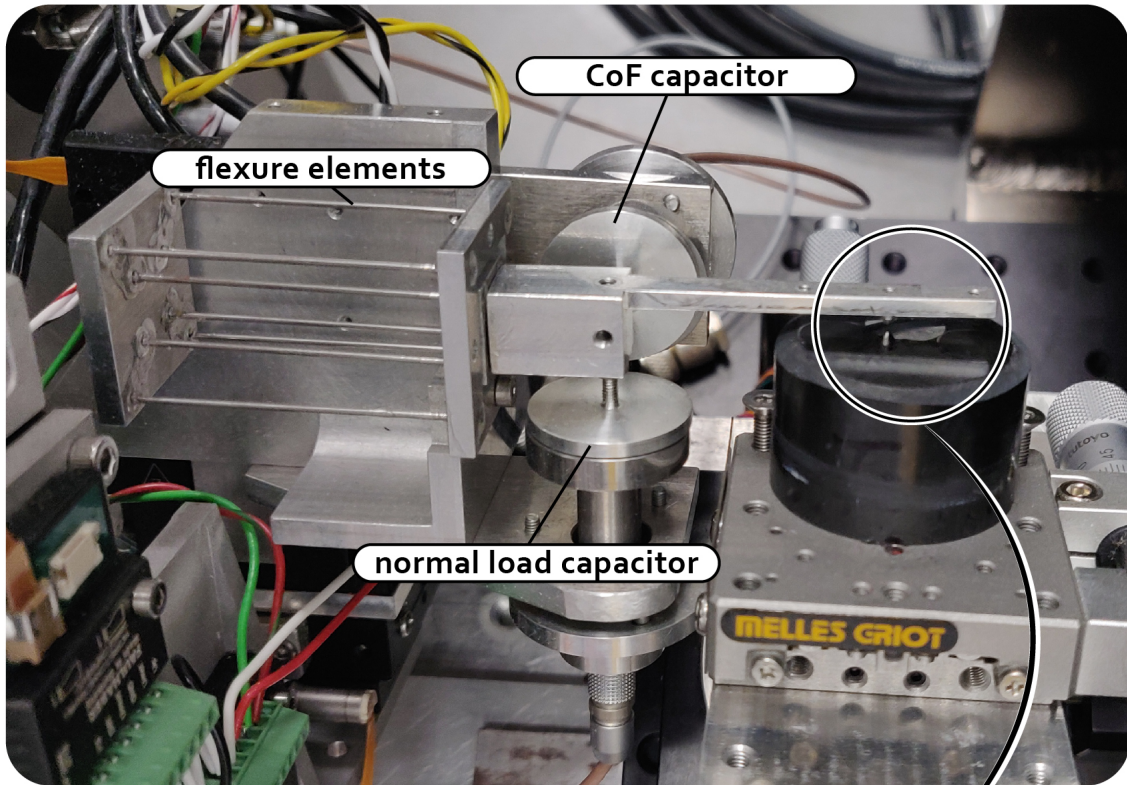


Figure 3.5: Illustration of the high-frequency reciprocating rig (HFRR) highlighting the upper specimen (ball) and lower specimen (disc/flat). Linear reciprocating motion with applied normal load are used to induce wear.

a)



b)

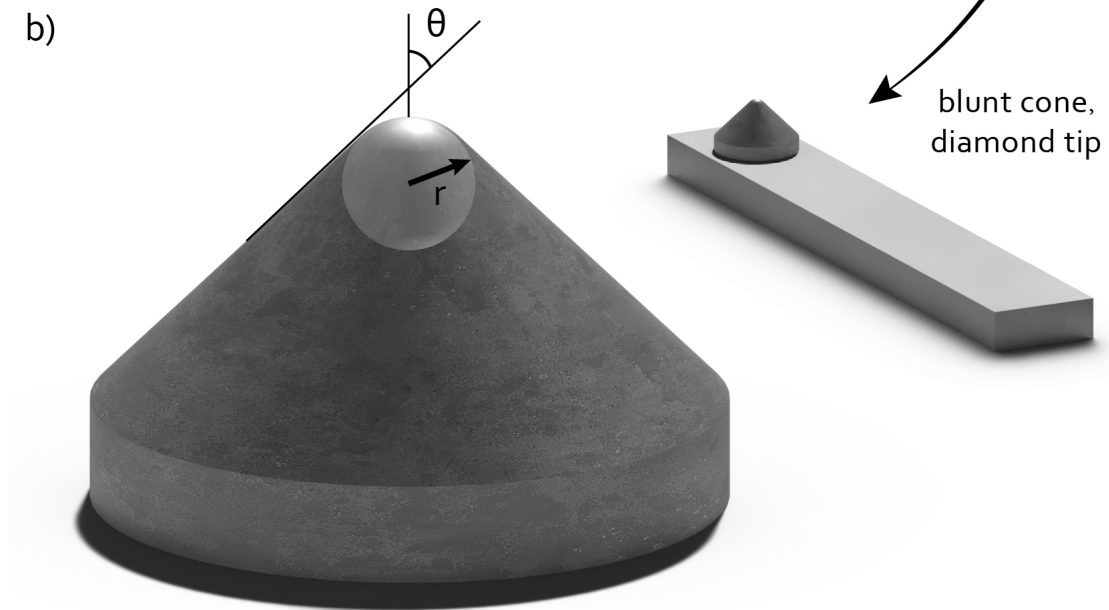


Figure 3.6: The NPL-designed and -manufactured tribometer. (a) Core components of the tribometer (i.e., flexure elements, load and friction capacitors, and scratcher). (b) A detailed illustration of the utilized scratcher, which has a sphero-conical (blunt cone) shape with a diamond tip. The half-width angle of the scratcher's tip is 45° . Two available options of tip-radii, $1\ \mu\text{m}$ and $5\ \mu\text{m}$.

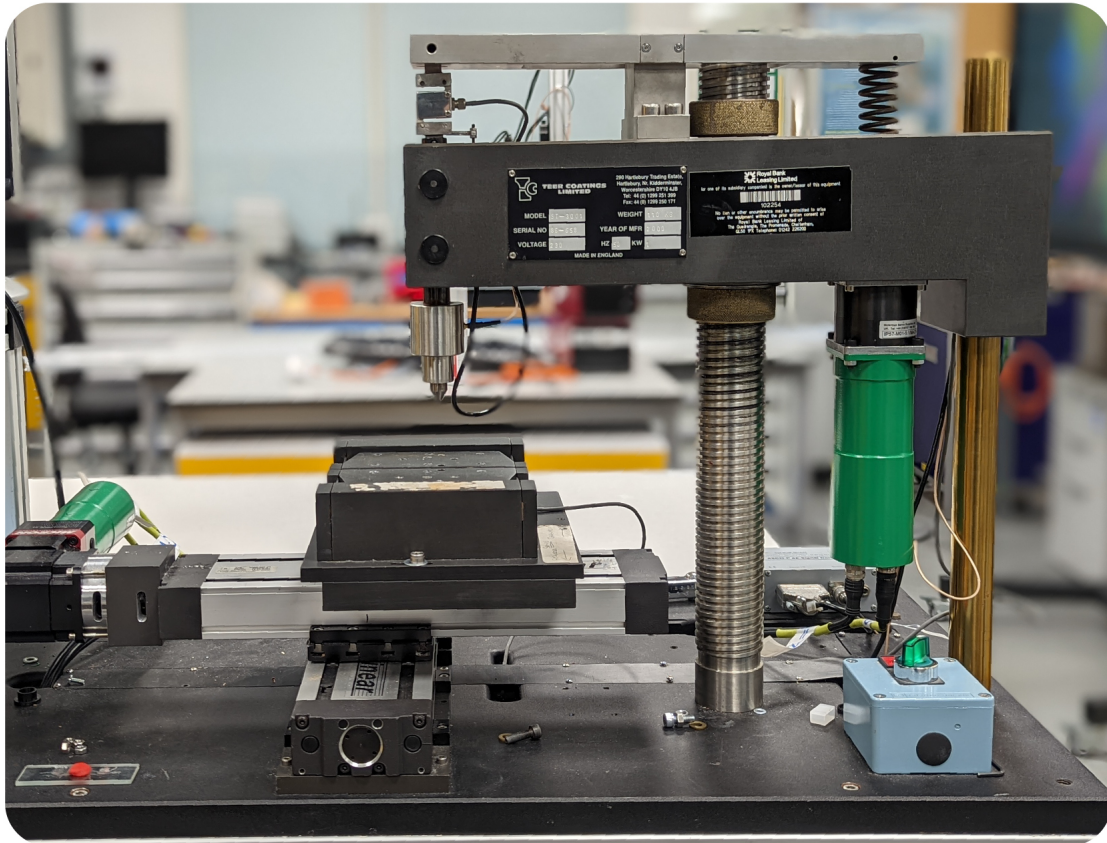


Figure 3.7: Teer coatings ltd. macro-tribometer (model ST-3001).

3.2.5 Software

The analyses of data and generation of charts were primarily performed with the aid of MATLAB. For the handling of EBSD data, an open-source, command-line MATLAB toolbox known as MTEX was used [170]. Despite the steep learning curve associated with this toolbox, it offers superior versatility for analyzing EBSD data compared with the graphical user interface software provided by EBSD manufacturers. Registration for SE-captured images (mentioned in 3.2.4) was performed with an open-source toolbox ImageJ – Fiji plugin [171]. The registered images were transcoded into video format with a custom MATLAB script. A MATLAB-based software, Anisovis[172], was used for generating single crystal, three-dimensional stiffness maps.

3.3 Correlating the microstructure and tribology of SLM-manufactured 316L

As highlighted in the review of literature (2.4.2), there is no consensus on whether SLM-manufactured steel is suitable for tribological applications. Amidst this uncertainty, there remains a lack of studies, and especially those that focus on the mechanistic aspects. This section comprises two experimental studies that focus on understanding the tribological

behavior of SLM-manufactured 316L. The first study focuses on highlighting the role of microstructure on the anisotropy of wear mechanisms in sliding contact. As such, this study pays less attention to the performance aspect but more to revealing relationships via several characterization techniques. The second study focuses on visual observation of the tribological contact evolution by mimicking single-asperity contact. In this study, the role of microstructure on the anisotropic tribological behavior can be visually appreciated. In both studies, conventionally manufactured 316L is used as a reference material.

3.3.1 Wear mechanisms anisotropy

Sample fabrication, preparation, and characterization

HFRR specimens were fabricated by SLM and conventional methods from 316L material. The SLM specimens, totaling of 47, were fabricated from a single batch using the Mlab Cusing machine with the parameters shown in Table 3.2 below. The selection of these parameters (with exception of the scan strategy) was based upon the manufacturer's recommended parameters to minimize the porosity. The choice of uni-directional scan strategy was to simplify the thermal history of the melt pools.

Table 3.2: SLM parameters used to fabricate the 316L specimens using the Concept machine.

Parameter	Selection
Scan strategy	Fixed uni-directional
Laser power	90 (W)
Scan speed	600 (mm/s)
Hatch spacing	77 (μm)
Layer thickness	20 (μm)

The SLM axes comprise the SD, BD, and transverse direction (TD). To validate and examine the tribological anisotropy, three different-specimens types whose tribological surfaces were deliberately aligned with each respective reference plane (i.e., SD-BD, SD-TD, TD-BD). In other words, the SLM parameters (including scan strategy) were kept constant, but the fabricated specimens were tilted 90° on the build plate (see Figure 3.8a-c). Consequently, the samples were named SD-BD, SD-TD, and TD-BD as to indicate the respective SLM plane to which the tribosurface is parallel. On the other hand, the conventionally manufactured specimens were fabricated from wire-drawn 316L rod (denoted as WD). The elemental composition for each manufacturing route is shown in Table 3.3 below. Noting that the WD specimens were annealed (4 hours at 1060°C and water quenched) while the SLM specimens were not treated as to examine their as-built properties. Nonetheless, all samples were mirror polished (by Watson Metal polishing, UK) as to achieve a consistent surface roughness. The R_a was consistently below 800 nm as measured by WLI. The surface roughness measurements were taken after ultrasonication

baths in toluene and isopropanol to remove any loose debris or polishing compounds. At this stage, the samples were fully prepared for the tribological testing.

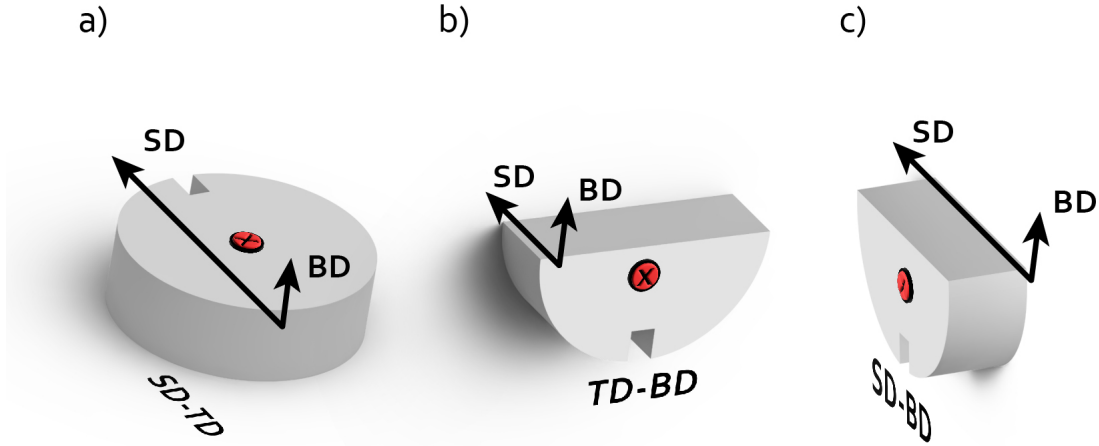


Figure 3.8: Illustrations of the three types of SLM-manufactured specimens. The “X” marks indicate the tribological surface and below each specimen is the plane corresponding to that tribological surface. The tribological surface in (a) is SD-TD, (b) is TD-BD, and (c) is SD-BD.

Table 3.3: The elemental composition of the powder and the wire-drawn 316L. Values shown are based on data obtained from suppliers of each material. Noting that the wire-drawn specimen was annealed for 4 hours at 1060 °C and subsequently water quenched.

	wt. %												
	Fe	Cr	Ni	Mo	C	Mn	P	S	Si	Cu	N	Co	O
SLM	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

EBSD Characterization

A subset of the SLM and conventional samples were taken out of the group for EBSD analysis. These samples underwent further metallurgical preparation such that the EBSD can be carried out. EBSD was performed on the Hitachi S-3400 SEM. The conditions for the EBSD analyses of samples were identical. The conditions were a sample tilt of 70°, an accelerating voltage of 25 keV, and a step size of 2 μm . The final map size was 1.5 mm x 1.2 mm. The analysis was carried out with the MATLAB toolbox MTEX [170]. The segmentation angle for high-angle grain boundaries (HAGB) was set to 15°. The maps were slightly post-processed by removing grains with size less than 10 pixels and performing a Laplacian smoothing with an iteration rate of 2. Grain sizes were calculated using the diameter function in MTEX.

Taylor factor calculation

M was calculated based on a custom-written script which utilized the *calcTaylor()* function in MTEX. A plane strain with symmetrical flow was assumed in which the loading is set perpendicular to the sliding surface. The same post-processing used in the EBSD maps was also performed on the Taylor maps.

Hertzian contact and HAGB intersections calculations

The Hertzian contact radius, a , was estimated based on Eq. 3.1 for the elastic contact of a sphere-on-flat:

$$a = \left(\frac{2rN}{3E^*} \right)^{1/3}, \text{ where} \quad (3.1)$$

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

r is the radius of the ball in mm, N is the applied normal load in Newtons, E_1 and E_2 are the elastic moduli of the specimen and ball (respectively) in MPa, and ν_1 and ν_2 are the Poisson's ratios of the specimen and ball (respectively). E_1 and E_2 were assumed to be 220,000 MPa, while ν_1 and ν_2 were assumed to be 0.27 for 316L stainless steel. The radius, r , was set to 3 mm. A custom-written script was used to determine the intersection between the Hertzian contact and the HAGB of the EBSD map. The script utilized the *boundary.intersect()* function in MTEX. Intersections were calculated for an arbitrary distance of 1000 μm for each EBSD map.

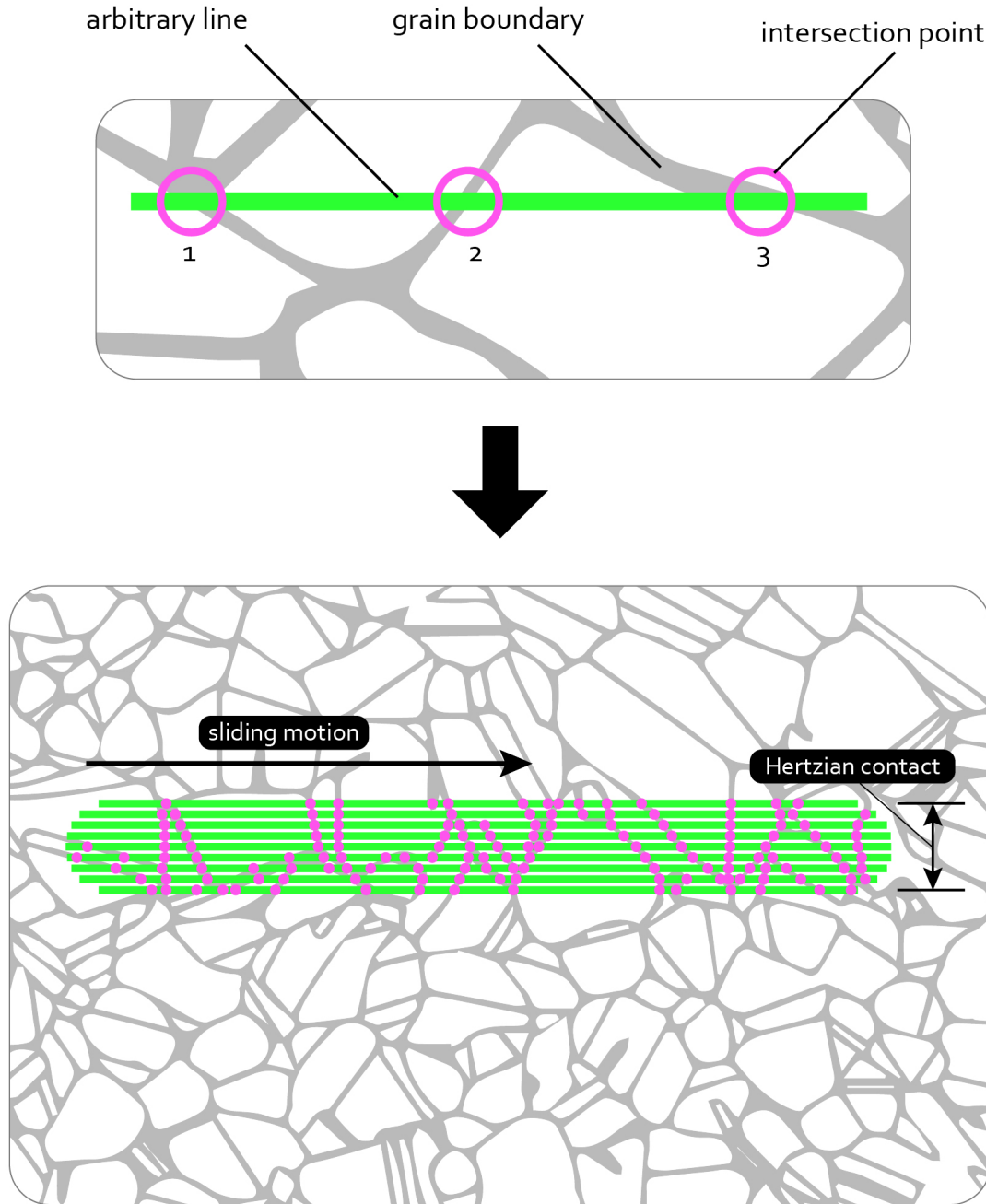


Figure 3.9: Hertzian contact and HAGB intersection method. MTEX's `boundary.intersect()` function was used to visualize intersection grain boundary intersections with of a sliding Hertzian contact. The magenta-colored dots resemble these intersection points.

Hardness measurement

Vickers hardness (HV) was determined using a universal hardness tester ZHU 187.5 (Zwick, Germany). A force and hold time of 3 kgf and 10 s were used, respectively (i.e., HV3/10). For each specimen, six hardness measurements were taken and averaged along with the corresponding standard errors for each.

Wear test, measurement and analysis

Dry sliding, reciprocating wear tests were conducted using the HFRR tribometer. The balls (upper specimens) were made of 316L as to have the hardness and composition as close as possible to the lower specimens. Using the hardness measurement protocol above, the measured hardness of the 316L balls was 232.5 ± 8.2 . The stroke length, reciprocating frequency, and trial time were set to 1.5 mm, 50 Hz, 30 min, respectively. Five different loads were applied: 0.98, 1.96, 2.94, 3.92, 4.9 N. For the sake of accuracy, the intention was to perform three trials for each load. However, considering the number of specimens available after performing EBSD and hardness measurements, some specimens possessed only two trials. The average CoF for each load and specimen type was calculated along with the standard error. The specimens were subsequently cleaned in sonicated baths for surface and wear characterization.

The wear tracks were analyzed using WLI. Due to the large area of the wear track, multiple small 3D maps were collected in batches and stitched using the VEECO's software. The wear track maps were subsequently post-processed to remove noise and align the images. The wear volume was calculated based on the total displaced volume from specimen's flat surface down to the deepest point of the wear track. The wear rate (W_{rate}) (in $\text{mm}^3/\text{N.m}$) was calculated based on Eq. 3.2 below:

$$W_{rate} = \frac{V_{total}}{N SD_{total}} \quad (3.2)$$

where N is the normal load, SD_{total} is the total sliding distance, and V_{total} is the total wear volume. Further analysis of the wear tracks was performed using a combination of SE and EDS. The SE accelerating voltage varied between 15-25 keV based on the features being inspected. On the other hand, the EDS accelerating voltage was 20 keV. The location of EDS mapping for all samples was fixed at the center of the wear track using the same magnification.

3.3.2 In-situ observation of tribological anisotropy

Sample fabrication

This experiment used one specimen from each type described in the previous experiment. The reader may wish to revisit the fabrication process in 3.3.1.

Preparation, and characterization

The specimens in this experiment (SD-TD, SD-BD, TD-BD, and WD) were prepared for EBSD prior to the scratch testing as to allow for post-scratch EBSD analysis. The conditions for EBSD were similar to those used in 3.3.1. In addition, ECCI was performed on the TESCAN Mira 4 SEM with the accelerating voltage, beam current, and working distance of 10 kV, 1 nA, and 9 mm, respectively.

In-situ scratch testing

The NPL tribometer was used for conducting high-vacuum, in-situ SEM scratch testing (Figure 3.6). Due to the sphero-conical shape of the scratcher, a certain transition depth (d_t) dictates mode of contact. d_t was calculated as 0.293 μm based on the following expression [173]:

$$d_t = r(1 - \sin\theta) \quad (3.3)$$

where r is the tip's radius (1 μm) and θ is the half-apex angle (45°). Therefore, depending on the actual value of d_t , the effective Hertzian contact area (A^*) can be one of two:

$$A_{spherical-cap}^* = \pi \frac{w^{*2}}{4} \sqrt{2} \quad , \text{ or} \quad (3.4)$$

$$A_{sphero-conical}^* = \pi \frac{w^{*2}}{4} \sqrt{2} - 0.3811 \quad (3.5)$$

where W^* is the minimum conformal scratch width in μm . The difference between minimum conformal scratch width (W^*) and the total scratch width (w_{total}) is shown in Figure 3.11a,b. It can be seen that W^* can be equivalent to w_{total} (Figure 3.11a), or less than w_{total} (Figure 3.11b). If we only consider w_{total} , then we ignore any complexities that occur inside the larger wear track. Whereas considering W^* has the advantage of tracking the conformity variations of the wear track across cycles. The calculation of W^* was done by taking eight measurements from two-dimensional SEM images. The standard deviation for those measurement was calculated. Lastly, the applied pressure (P_o) was calculated as was done in previous studies [174, 175]:

$$P_o = \frac{2N}{A^*} \quad (3.6)$$

where N is the normal load. As for the area, note that it was divided by a factor of 2. The reason being that as the scratcher plows forward, only half of its area physically comes into contact with material from the specimen being scratched (see Figure 3.10).

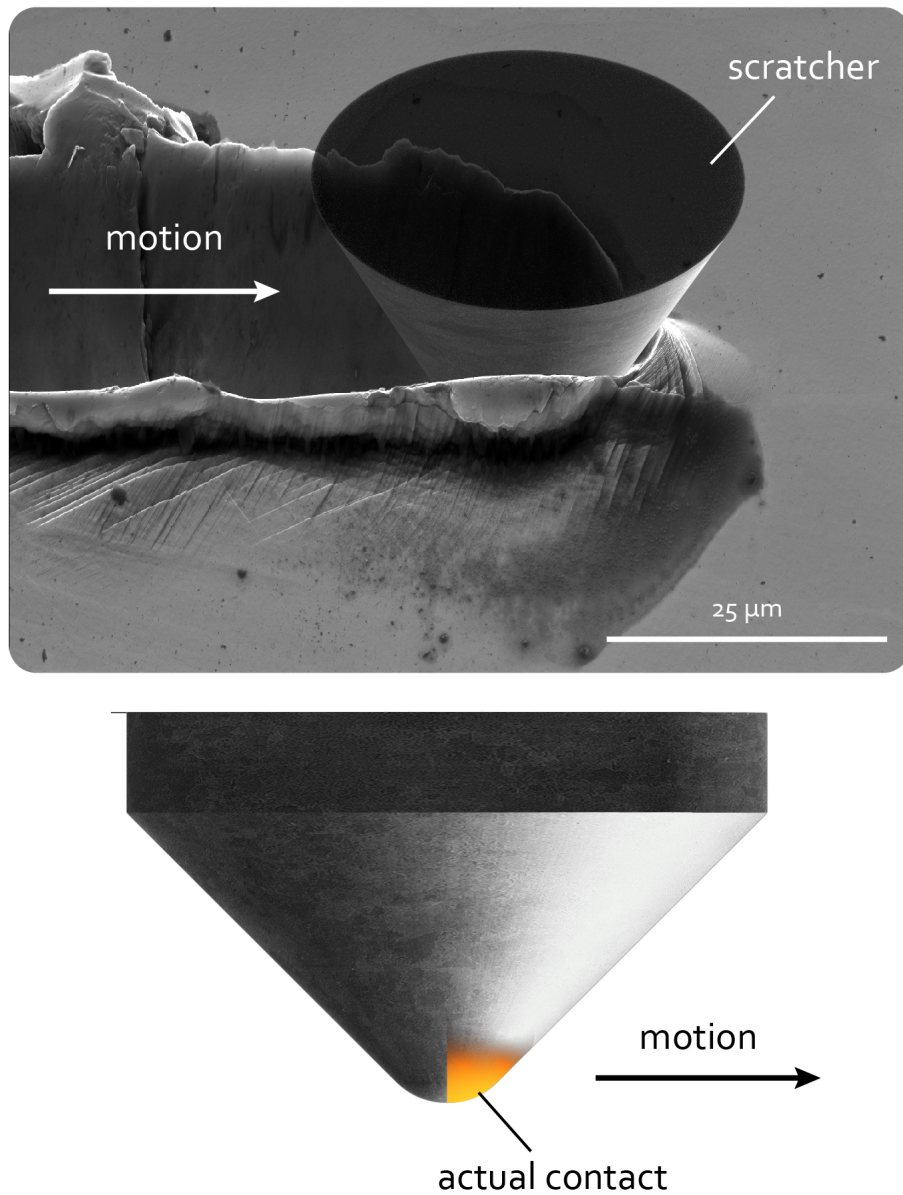


Figure 3.10: SEM micrograph of a micro-scratched specimen with the indenter superimposed on top. The schematic below the SEM micrograph shows the actual contact surface compose only halve of the idealized Hertz contact.

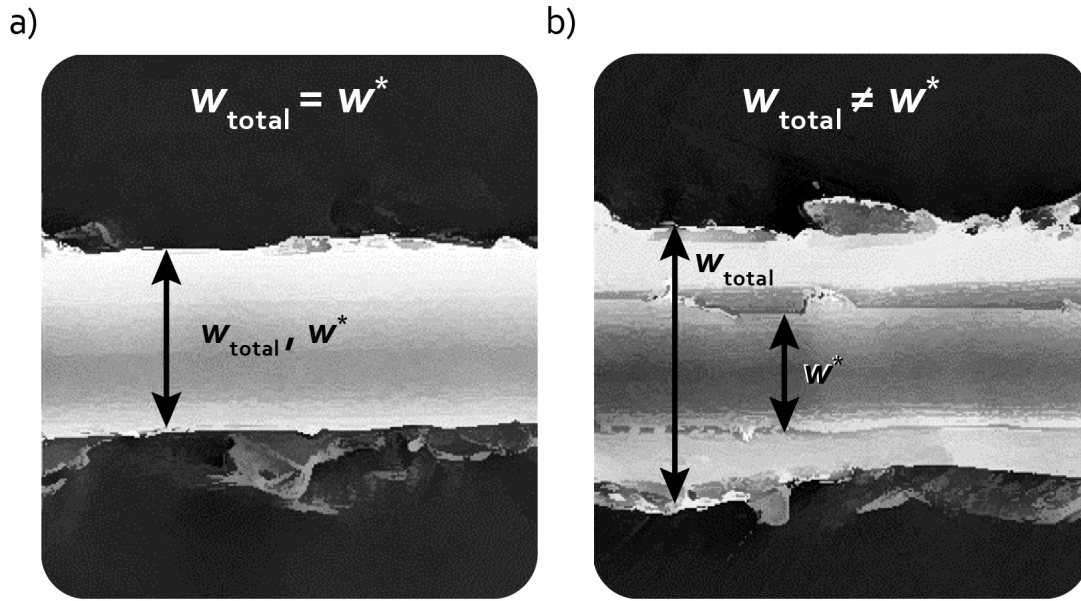


Figure 3.11: Schematics to explain the difference between the total scratch width (w_{total}) and the minimum conformal scratch width (W^*). (a) shows a scenario where w_{total} is equal to W^* , whereas (b) shows a scenario where w_{total} is greater than W^* .

Two types of scratch tests were performed, a single- and a multi-pass test. In both tests, the normal load was 100 mN, the scratch distance was 1 mm, and speed was 300 mm/s. The single-pass was used to study the onset of wear and friction, whereas the multi-pass was used to study the evolution of the two.

In the multi-pass test, each SEM image corresponded with a cycle of two scratches performed in the opposite directions. Table 3.4 below shows the equivalent scratch number for each cycle. Note that the first SEM image consists of a single scratch (i.e., half a cycle) while the remainder are all double-scratch cycles. The scratching was interrupted once a significant build-up of damage was noticed. Any scratches continuing after built-up damage were extra. At the end of testing, the total scratch cycles for SD-TD, SD-BD, TD-BD, and WD were 55, 55, 54, and 28, respectively.

Table 3.4: The corresponding cycle number to each scratch. Given that each cycle comprises two scratches in opposite directions, the first scratch has a cycle of 0.5.

Cycle number	0	0.5	2	3	4	...	54	55
Scratch number	0	1	3	5	7	...	107	109

The captured images were registered and transcoded into video with the ImageJ – Fiji plugin [171] and MATLAB, respectively. Damage assessment was performed using a combination of WLI, SEM, and EBSD.

3.4 Developing a multigrade tribological surface via SLM

To date, producing an alloy with multigrade tribological properties has not been achieved. One reason, which has already been covered, is because the tribology of SLM-manufactured components is poorly understood. Another reason is because the area of ‘tailoring’ the microstructure is, in and of itself, quite complex. It requires a careful understanding and modulation of the SLM parameters to achieve the desired attributes, such as a multi-textured, single component. This section is devoted to the experimental design and techniques used to develop and test a multigrade tribological surface of SLM-manufactured 316L.

Sample fabrication

Multiple multigrade tribosurfaces were produced in collaboration with Sofinowski et al.[101] using their layer-wise engineering of grain orientations (LEGO) approach. Their novel method works by taking advantage of the directional solidification along the SD. One of the $\langle 001 \rangle$ directions is closely aligned with the SD. As such, the $\langle 001 \rangle$ follows changes in the SD as well. By changing the SD mid-print (via specifying the angle of rotation), grains can be rotated along the BD’s axis for a pre-defined set of layers. However, this can only be achieved by first optimizing the other SLM process parameters for texture control. The process of optimization, which falls beyond the scope of this PhD project, was successfully implemented by Sofinowski et al. [101]. The optimization process involves controlling the energy input to achieve a consistent thermal profile which maintains the dominant texture. The SLM parameters which were used (following Sofinowski et al.’s paper [101]) are shown in Table 3.5. Noting that these ‘optimized’ parameters are machine specific. In fact, the same parameters were used with the Renishaw AM250, but yielded a weak texture, and specimens were subsequently discarded. These parameters were held constant for all of the produced multigrade tribological surfaces. The elemental composition of the gas-atomized 316L powder is shown in Table 3.6

Table 3.5: SLM parameters used to fabricate the 316L specimens using the SLM500 machine.

Parameter	Selection
Scan strategy	Bi-directional
Laser power	240 (W)
Scan speed	600 (mm/s)
Hatch spacing	100 (μm)
Layer thickness	40 (μm)

Table 3.6: 316L-powder composition used for SLM500-specimens

wt.%									
Fe	Cr	Ni	Mo	C	Si	Mn	P	S	N
Bal.	16-18	10-14	2-3	0.03	<1	2	0.045	0.03	0.1

Three multigrade tribosurfaces were produced, each with a different crystallographic texture configuration. The first tribosurface comprised a repeating set of three grades of crystallographic texture. The grades were produced by selecting one of three rotation angles for each grade. These angles were 0° , 90° , and 55° . As the schematic in Figure 3.12 shows, these angles should produce a $\langle 001 \rangle \parallel z$, $\langle 011 \rangle \parallel z$, and $\langle 111 \rangle \parallel z$, respectively. For simplicity, the ‘ $\parallel z$ ’ suffix will be omitted hereinafter. The different grades were arranged along the axis perpendicular to the BD’s axis (i.e., the TD axis). Each grade segment was 1-mm thick along the TD axis.

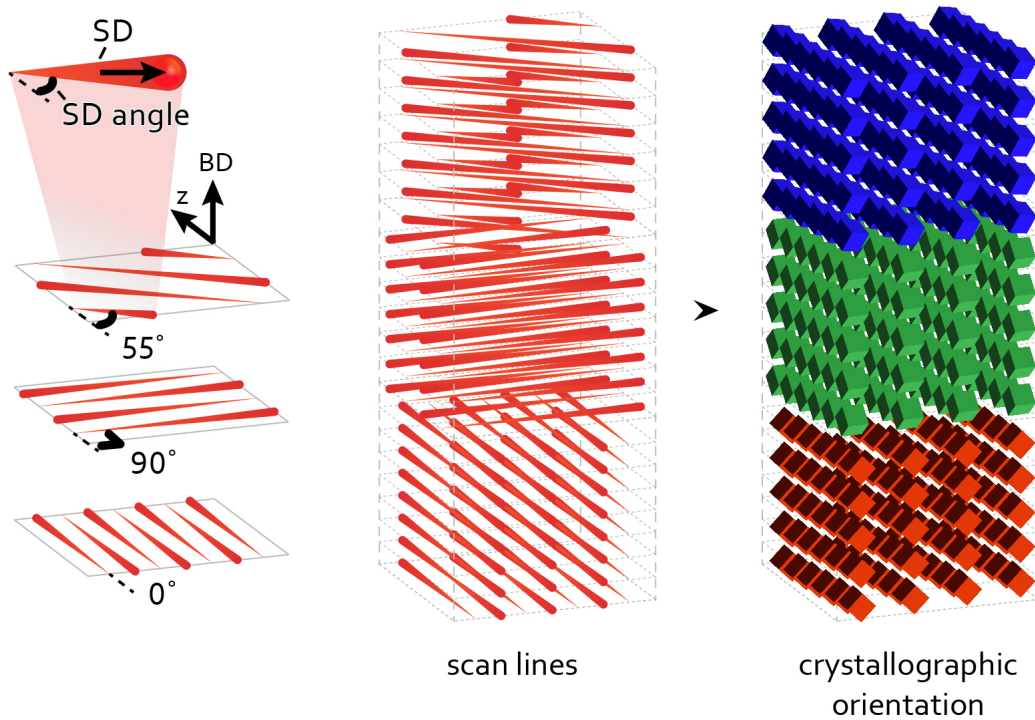


Figure 3.12: Layer-wise grain orientation (LEGO) technique [101].

The second tribosurface also comprised a repeating set of three grades of crystallographic texture. However, these grades were arranged along the BD’s axis. The same three rotation angles were used as in the previous tribosurface. Considering a contact diameter of approximately $3.5 \mu\text{m}$ which revealed upon scratching a scrap specimen, the transition between the texture blocks needs to be large enough to allow sufficient data

generated for each texture. Simultaneously, because texture typically develops only after a certain number of layers were overlaid (typically spanning a distance of 0.5 mm based on experience gathered by our collaborator Sofinowski), it was safest to select 1 mm thickness for each texture block. Thus, by dividing the thickness of each texture block (1 mm) by the thickness of each layer (25 μm), we arrive at a total of 25 layers per texture block.

The third tribosurface comprised two grades of crystallographic texture, a $\langle 001 \rangle$ and a $\langle 111 \rangle$. These were produced by selecting a rotation angle of 0° and 55° , respectively. The two grades were arranged along the BD's axis and were not set to repeat. As such, the entire tribosurface was equally split into two regions, a $\langle 001 \rangle$ and a $\langle 111 \rangle$. The specimen was designed in a disc shape such that it can optionally fit into the HFRR lower specimen holder. However, for the sake of this thesis, the disc shape of the specimen bears no relevance.

Sample characterization

Sample characterization was performed using TESCAN Clara 4 SEM, which carries a e-Flash1000 EBSD detector and a low energy, retractable BSE detector. For EBSD, the accelerating voltage and working distance were 20 kV and 15-18 mm, respectively. The step size varied between 9 nm to 9 μm based on the feature of interest. To visualize dislocations, the BSE was configured to perform ECCI. The accelerating voltage, working distance, beam current for ECCI were, 20 kV, 9 mm, 1 nA, respectively. To enhance ECCI visualization, brightness was lowered to 0% (i.e., using only contrast). Subsequently obtained images were image-processed to increase sharpness and contrast.

Tribological testing

Tribological testing consisted of micro- and macro-scratch testing. The micro-scratch testing was conducted using the NPL tribometer (described in section 3.2.4). However, a different indenter was used which had a radius of 5 μm and a half-apex angle of 45° . The load, speed, and distance were 10 mN, 50 $\mu\text{m/s}$, and 4 mm, respectively. On the other hand, the macro-scratch testing was performed using ST-3001 (Teer coatings ltd, UK). Macro-scratches were performed using a Rockwell C-type tip (with a radius and half-apex angle of 100 μm and 60° , respectively). The load, speed, and sliding distance were 15 N, 0.5 mm/s, and 6 mm, respectively.

Software analyses

Analysis of EBSD data was performed using MTEX [170]. Post-processing was performed on EBSD maps to remove zero-solution datapoints. The segmentation angle for HAGBs was set to 15° . Geometrically necessary dislocations (GNDs) were calculated using based

on Pantelon's paper[176] using MTEX. M were calculated using MTEX's built-in, *calcTaylor()* function. For simplicity, the indenter acting upon the tribosurface was assumed to induce a uniaxial plane strain. Correspondingly, the rank-2 deformation tensor (ε) was defined as:

$$\varepsilon = \begin{bmatrix} 0.5 & 0 & 0 \\ 0 & 0.5 & 0 \\ 0 & 0 & -1 \end{bmatrix}. \quad (3.7)$$

To find the effect of crystallographic stiffness on the coefficient of friction, M -values were plotted against the CoF. Where multiple single-scratches were performed, the CoF values were averaged for all scratches performed on tribosurfaces of the same crystallographic texture. To exemplify, consider a multigrade tribosurface comprising a repeating set of $\langle 001 \rangle$, $\langle 011 \rangle$, and $\langle 111 \rangle$ textures. CoF-values for all scratches performed on $\langle 001 \rangle$ surfaces were averaged (and similarly for the $\langle 011 \rangle$ and $\langle 111 \rangle$ surfaces). As for M , the values were obtained by averaging M values for a sample area below just below the scratch (Figure 3.13). To improve accuracy, the average of 100 pixels along the transverse section of the scratch were used to determine the average value of M for that column. This yielded much higher sampling rate of M -values compared with CoF-values. Accordingly, the array containing the M -values was down-sampled using a custom MATLAB script to match the sampling rate of the CoF-vector. Finally, M versus CoF was plotted.

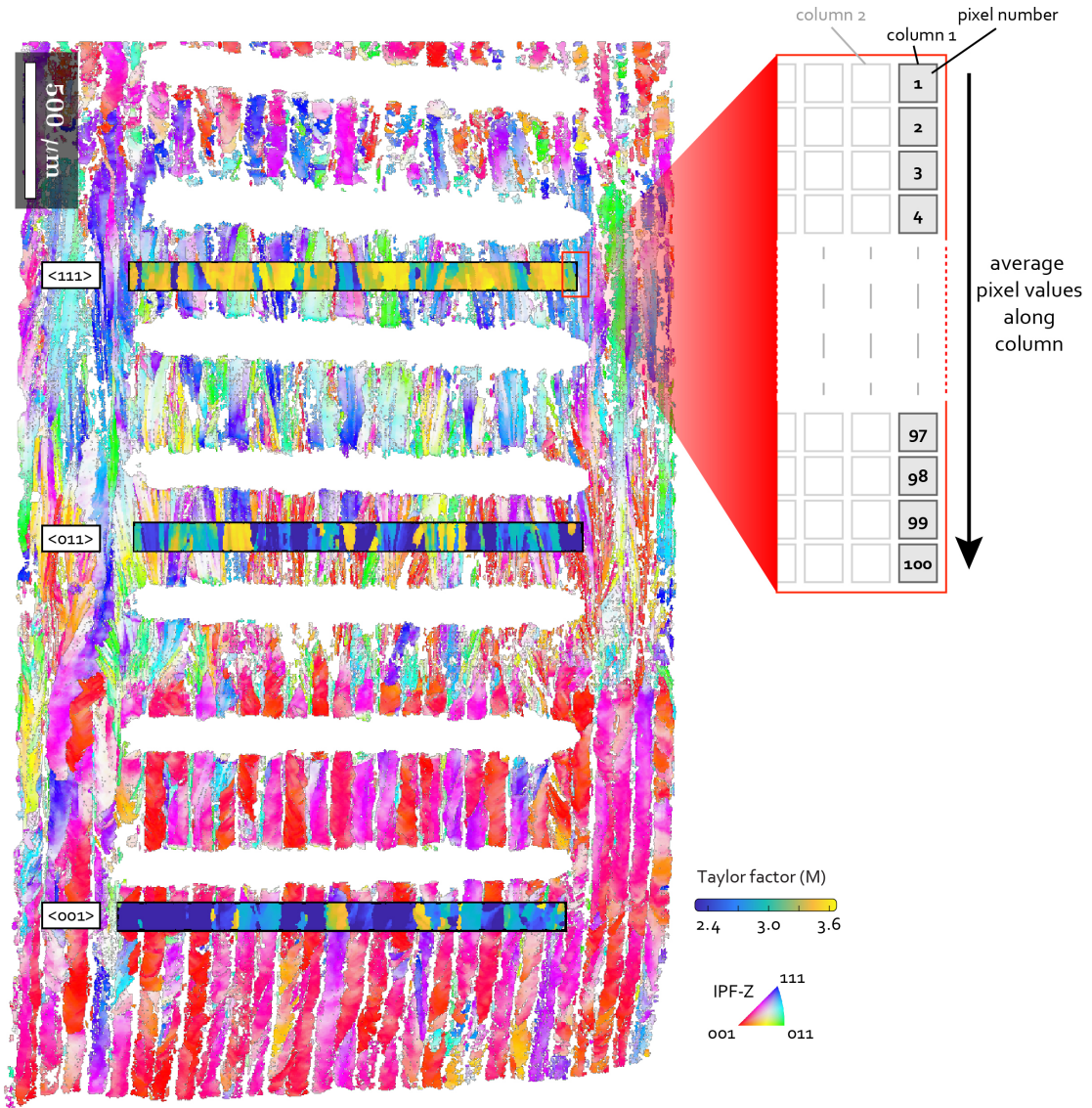


Figure 3.13: Calculation method for M . M values were averaged for each column. Subsequently, the sampling rate for the averaged M values was reduced to match that of the CoF.

The ideal, plane-specific stiffness for FCC 316L single crystal was obtained the following way. The matrix containing the elastic moduli (C_{ij}) for a cubic structure are given by:

$$C_{ij} = \begin{bmatrix} c_{11} & c_{12} & c_{13} & & & \\ c_{12} & c_{11} & c_{13} & & & \\ c_{13} & c_{13} & c_{11} & & & \\ & & & c_{44} & & \\ & & & & c_{44} & \\ & & & & & c_{44} \end{bmatrix}, \quad (3.8)$$

where c_{11} , c_{12} ($= c_{13}$), and c_{44} (obtained from Zinovieva et al.[177]) are 206 GPa, 133 GPa, 119 GPa respectively. The compliance moduli (s_{ij}) can be calculated via equations. Equations (3.9) to (3.11)[178, 179]:

$$s_{11} = \frac{c_{11} + c_{12}}{(c_{11} - c_{12}) + (c_{11} + 2c_{12})}, \quad (3.9)$$

$$s_{12} = \frac{-c_{12}}{(c_{11} - c_{12}) + (c_{11} + 2c_{12})}, \quad (3.10)$$

$$s_{44} = \frac{1}{c_{44}}. \quad (3.11)$$

Finally, the plane specific Young's moduli (E_{hkl}) can be calculated from Equation 3.12 [180]:

$$E_{hkl} = s_{11} - 2\left(s_{11} - s_{12} - \frac{s_{44}}{2}\right)\left(\frac{(hk)^2 + (kl)^2 + (hl)^2}{(h^2 + k^2 + l^2)^2}\right). \quad (3.12)$$

For visualization purposes, the MATLAB software Anisovis [172] was used to generate a three-dimensional map of the FCC 316L's stiffness.

3.5 Chapter conclusion

Aiming to meet the research objectives, multiple equipment, techniques, experimental designs, and collaborations were leveraged. These elements were distributed among two themes: 1) correlating the microstructure and tribology of SLM-manufactured 316L, and 2) developing a multigrade tribosurface via SLM. The first theme comprised two experimental designs, each focusing on a different mechanistic perspective (i.e., wear mechanism anisotropy versus observing the anisotropic contact evolution). The second theme consisted of leveraging the obtained knowledge into the design of multigrade tribosurfaces by microstructure control. The results and discussion related to these three experimental designs are presented in Chapters 4, 5, and 6, respectively.

Chapter 4

Wear mechanisms anisotropy

The content of this chapter was adapted from the following publication:

M. Bahshwan, C. W. Myant, T. Reddyhoff, and M.-S. Pham. “The role of microstructure on wear mechanisms and anisotropy of additively manufactured 316L stainless steel in dry sliding”. In: Materials & Design 196 (2020), p. 109076. issn: 0264-1275. doi: <https://doi.org/10.1016/j.matdes.2020.109076>

4.1 Chapter introduction

Much of the research concerning SLM-manufactured 316L had focused on quantifying the performance of wear without emphasis on understanding the mechanisms which give rise to wear in the first place. While some studies contributed to this understanding by contrasting the microstructural differences between SLM- and conventionally manufactured 316L [82, 181], the emphasis on correlating the microstructure with the exact wear mechanisms remains lacking. This is especially important considering the influence of initial (sub-)surface microstructure on the evolution of the microstructure during sliding (e.g., twinning, lattice rotation, grain refinement) [182]. This issue becomes of great deal of importance given that SLM-based manufacturing offers over 50 process parameters which work in concert to produce a distinct microstructure. Thus, without a mechanistic understanding of how the SLM-induced microstructure impacts the transpiring wear mechanisms, more studies on the topic will certainly lead to further contradictory, yet unexplained findings (see 2.4.2 for an elaborate discussion).

Accordingly, this study focused on discerning the role of microstructure on the wear mechanisms of SLM-manufactured 316L. The impact of the microstructure on the anisotropic behavior of wear mechanisms was also investigated. As a reference material, the study included conventionally manufactured 316L by wire-drawing process, whose microstructure was also examined.

(Note: experimental setup, parameters, and calculations pertaining to this study can be found in 3.3.1).

4.2 Results and discussion

4.2.1 Microstructural characterization

It is known that the material's strength is correlated to its microstructure. Hardness tests are an effective way to gain insight into a material's strength. Therefore, it would be useful to correlate to the hardness (strength) to the underlying microstructure. The Vickers hardness was measured for the three SLM sections (SD-TD, SD-BD, TD-BD) and the conventionally manufactured specimen (WD). The values in Table 4.1 show that the SD-TD and SD-BD exhibited the greatest hardness values of 244.5 ± 6.1 and 245.8 ± 4.0 , respectively. The TD-BD section on the other hand, exhibited a smaller hardness value of 232.4 ± 4.0 . The smallest hardness in all four was that of the conventionally manufactured specimen at 220.7 ± 4.5 .

Table 4.1: The measured hardness of each specimen.

Tribological surface	Hardness (HV $\pm$std. error)
SD-TD (SLM)	244.5 ± 6.1
SD-BD (SLM)	245.8 ± 4.0
TD-BD (SLM)	232.4 ± 4.0
WD (conventional)	220.7 ± 4.5

Boxplots comparing the grain size (in diameter) of each surface are shown in Figure 4.1. The plot shows that the conventionally manufactured specimen (WD) has smaller grains than any of the two-dimensional sections of the SLM-manufactured specimens. The TD-BD section has the widest spread in grain sizes and the largest median. On the other hand, the SD-TD section has the narrowest spread and smallest median.

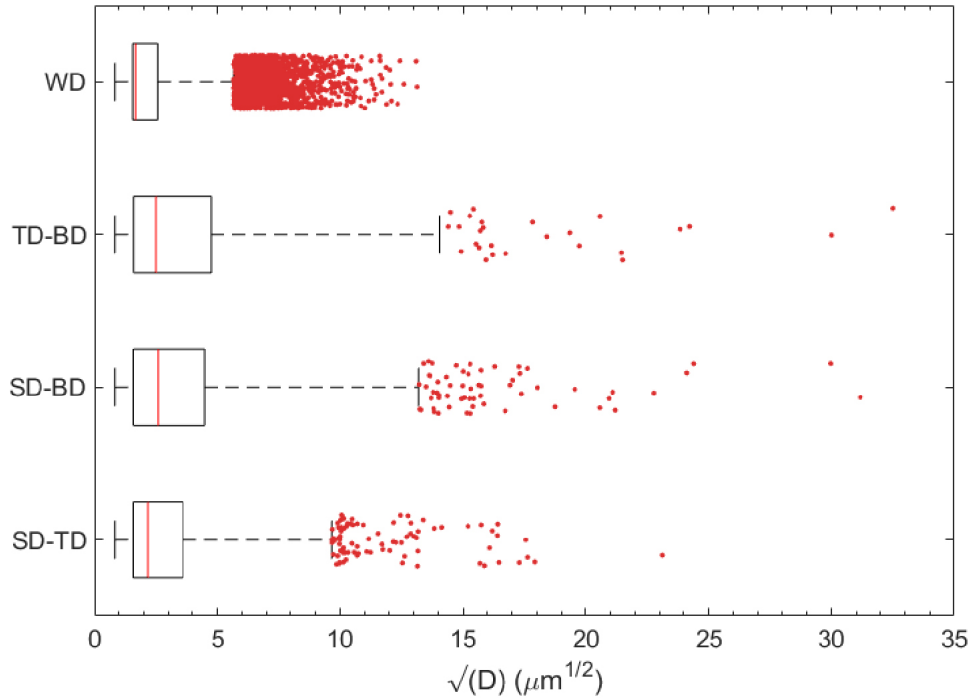


Figure 4.1: Boxplots showing grain-size distribution of each specimen [183].

To compare the melt pool morphologies, a sample of each SLM specimen type was sectioned along the SD-TD, SD-BD, and TD-BD, resulting in three sections per sample. These two-dimensional sections were etched and stitched together to form a three-dimensional view of the melt pool for each sample (Figure 4.2). Comparing the melt pools of the three cubes reveals that the melt pools merely rotated in multiples of 90° . However, as a result of this 90° -rotation, the active tribological surface ‘shifts’ onto a different reference plane. The active tribological surface for each specimen type is overlaid on top with an $\otimes$ mark. Note that the hardness was measured for the tribological surfaces, namely, SD-TD, SD-BD, and TD-BD. The etched surface of WD can be seen in Appx. 5.

A SE micrograph of an etched TD-BD section revealing the solidification cellular dendrites (cells/dendrites) is shown in Figure 4.3 below. The trajectory of the cells/dendrites corresponds to a $[100]$ direction. It can be seen that even within a single melt pool boundary, the cells/dendrites exhibit multiple domains (i.e., different trajectories). It is known that FCC crystals, such as austenite in 316L, have a $[100]$ growth direction inline with the maximum heat flux (i.e., the SD pointing into/out of screen) [90, 184]. The other set of $[100]$ is typically perpendicular to the melt pool, i.e., towards the melt pool centerline.

Nonetheless, because of the perturbations in the local heat flux, side-branching, and high-symmetry of FCC, the solidification cellular dendrites exhibit various $[100]$ directions [16].

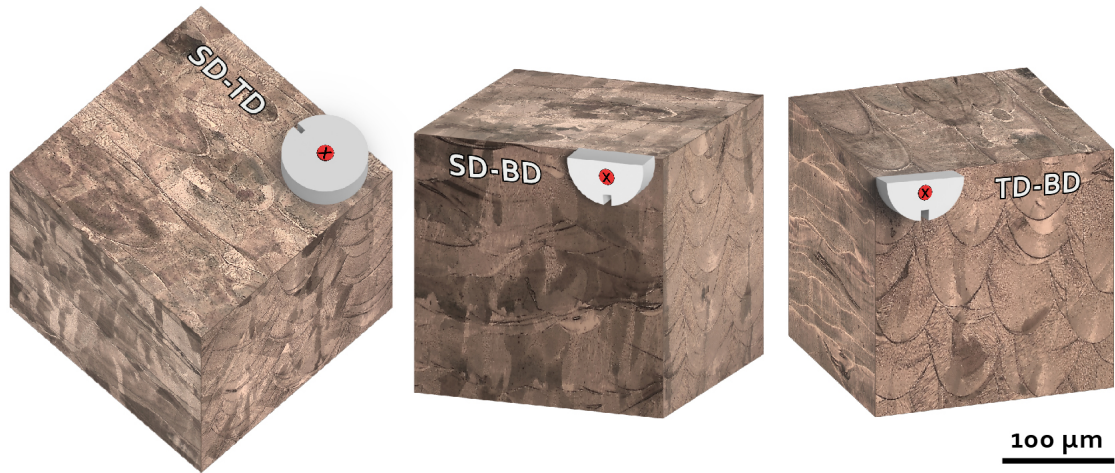


Figure 4.2: Three-dimensional construction of melt pools of each specimen with the tribological surface (marked with $\otimes$) superimposed on top. It can be seen that the melt pools look similar, but the tribological surface shifts amongst these different melt pool planes in each specimen [183].

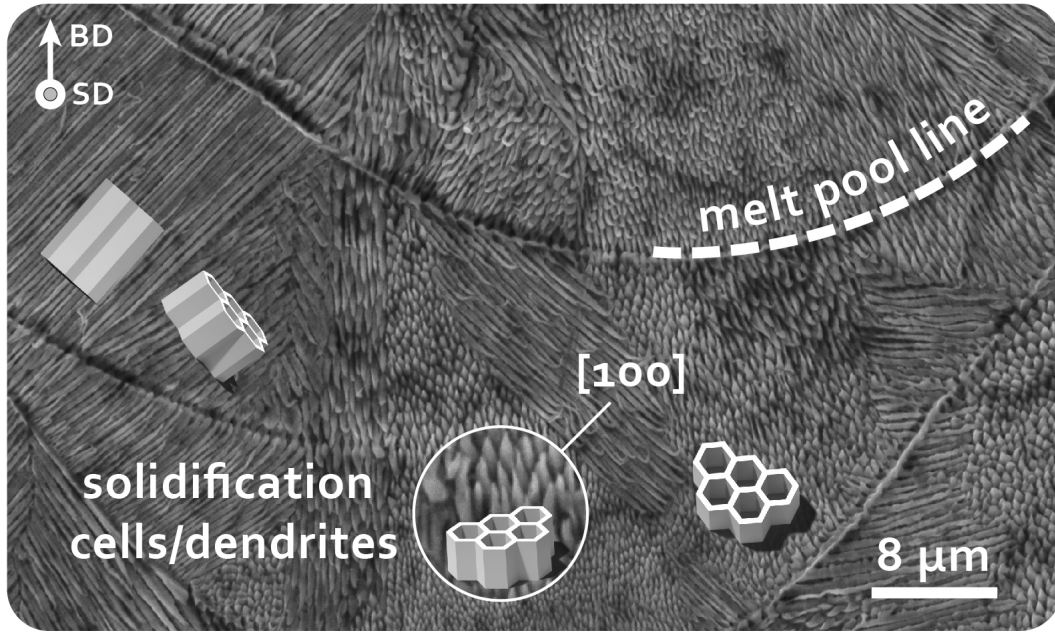


Figure 4.3: Scanning electron micrograph showing the solidification cellular dendrites of the TD-BD plane. The trajectory of the cellular dendrites correspond to a $[100]$ direction [183].

The crystallographic orientation maps are plotted using the IPF-BD map in Figure 4.4 below. It can be seen all the planes, i.e., SD-TD, SD-BD, and TD-BD exhibit a strong green color. This green color corresponds with a $[110]$ parallel to the BD axis. The other set of $[100]$ directions have been verified to lie along the SD where the maximum heat flux occurs. This crystal orientation is commonly referred to as Goss texture. To understand the why the Goss texture emerges, it is useful to inspect the ‘chevron-like’ pattern on the front face of the cube. The close-up diagram in Figure 4.4 shows two melt pools (crescent shapes) colored red and black. Depending on the hatch spacing and the spot size, these melt pools may overlap as shown on the figure. The overlap results in side-branching and broadening of grains which grow perpendicular to the melt pool boundary at about 45° angles from the BD [16]. It can also be seen that along the centerline of the melt pool there is a slender set of red-colored grains. These grains grow epitaxially without changing direction. They have all their $\langle 001 \rangle$ parallel to the BD, SD, and TD – a Cube component. Nonetheless, the ratio of the Goss-to-cube texture is significantly large.

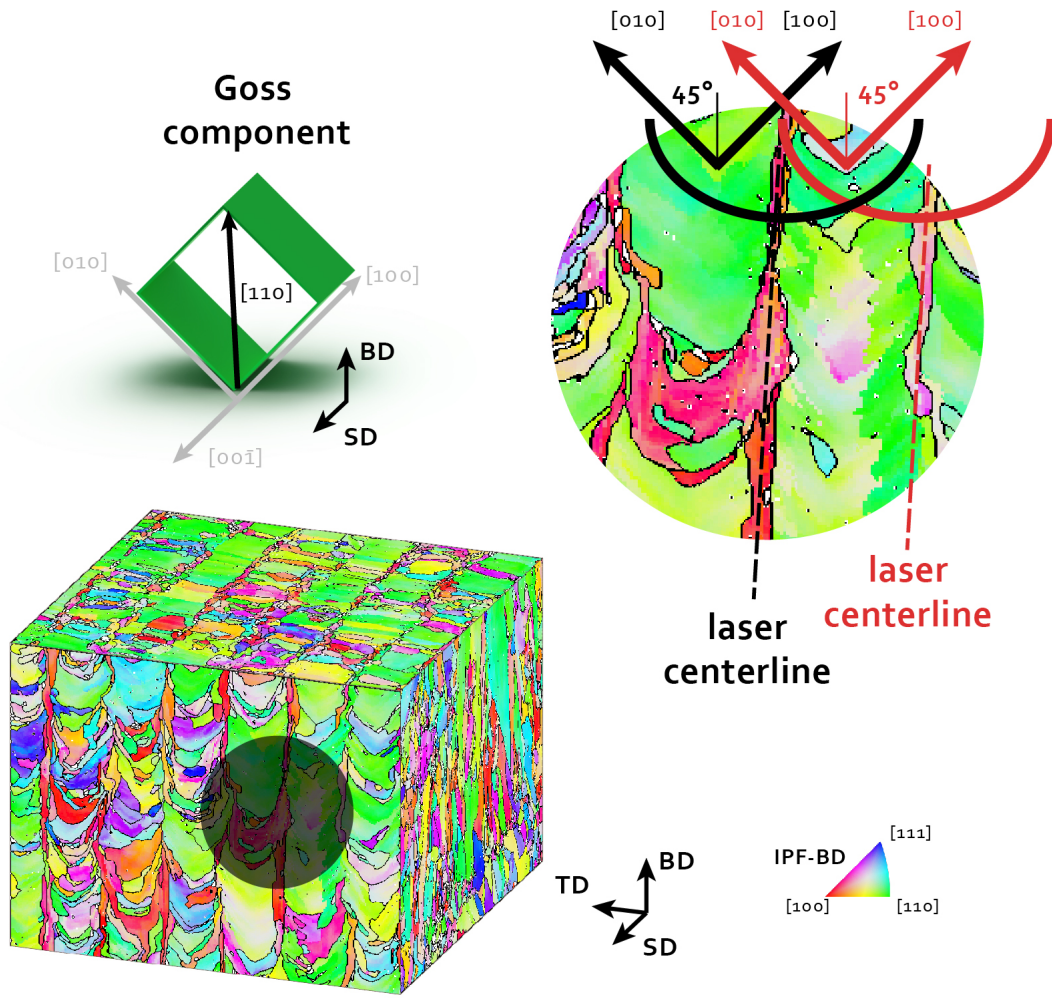


Figure 4.4: A three-dimensional construction of the SD-TD, SD-BD, and TD-BD planes' orientation maps. The orientation maps, generated with the BD axis as a reference, show a striking green color which indicates a Goss texture. On the other hand, slender areas which are mostly confined to the laser's centerline exhibit a Cube texture (red color) [183].

To gain quantitative and qualitative insights into the texture, contour maps obtained from the pole figures diagrams are shown in Figure 4.5a-d. The contour maps are shown for the (100), (110), and (111) planes. To put the Goss texture into perspective, it was represented by a small cube diagram which was superimposed on the contour lines. Doing so helps appreciate the direction of the Goss texture observed from different views (e.g., compare the cube orientation in the three (100) maps in Figure 4.5a-c). For example, it can be seen the Figure 4.5c that tribological surface is parallel to the cube's face (100). The strength of the texture can also be compared between the SLM- and conventionally manufactured specimens via the contrast bars. The strength of texture in the SLM sections is in the range of 8-10, whereas WD exhibited a weak texture with a maximum strength of about 2. This is most likely due to the fact that the SLM specimens were analyzed in their as-built condition, while the WD was analyzed in its annealed condition. Depending on

the temperatures and materials involved, annealing can weaken the texture of a material as it goes through recrystallization.

The Taylor orientation map of the three SLM sections with respect to the normal load (on each respective section) is shown in Figure 4.6. It can be easily noticed that the SD-TD and SD-BD sides exhibit prominent yellow color, while the TD-BD face exhibits a prominent blue color. The former faces have M -values closer to the upper end of 3.6, while the latter has M -values colors to the lower end of 2.3. These two types of grains are so-called ‘hard grains’ and ‘soft grains’, respectively.

As highlighted in the literature review, the initial texture can significantly impact the stiffness of the material relative to the loading conditions. In fact, the Goss texture, compared to other common FCC textures, has the largest contribution to plastic anisotropy [185]. The literature review covered how a [011] direction in FCC (e.g., Fe-12Cr-18Ni) can exhibit a modulus of 283.5 GPa compared with 198 GPa modulus for a [001] direction. Thus, we can vary the elasto-plastic anisotropy by changing the plane of the tribological surface with respect to the texture component. To put matters into perspective, the combined Hall-Petch and Taylor strength equation (2.6) is restated [27, 28, 54, 55]:

$$\sigma_y = \sigma_o + M\alpha\mu b\sqrt{\rho} + \frac{k_y}{\sqrt{D}}$$

Where σ_y is the material’s yield strength, σ_o 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 both materials are nominally similar in composition, it can be assumed that σ_o and Hall-Petch coefficient (k_y) are similar across all specimens, including WD.

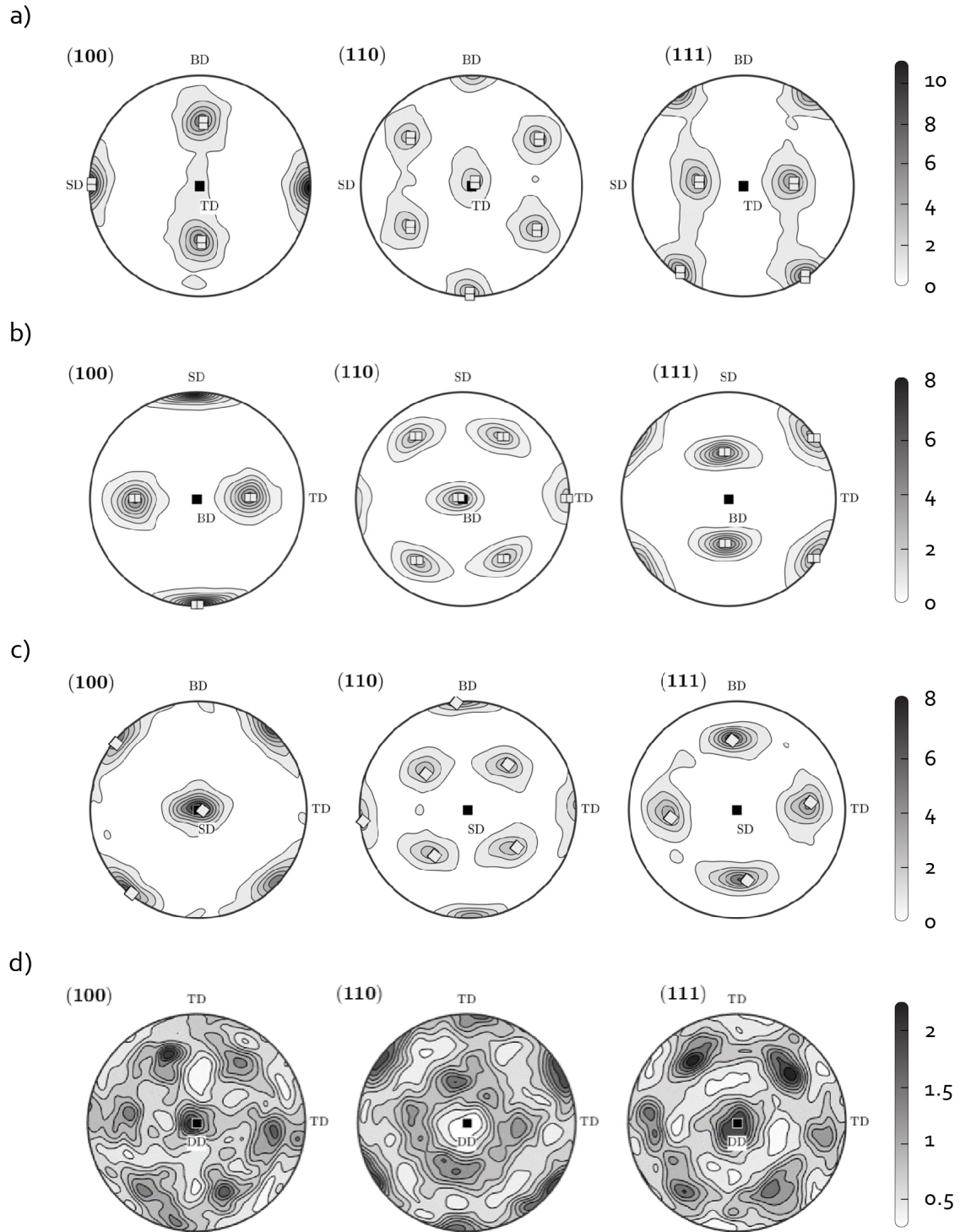


Figure 4.5: Contour pole figure plots of the (a) SD-BD, (b) SD-TD, (c) TD-BD, and (d) TD-TD planes. Note, because of the drawing process, all cross-sectional directions of the wire-drawn specimen are denoted as TD. The scale bars denote the intensity of occurrence of the planes in question [183].

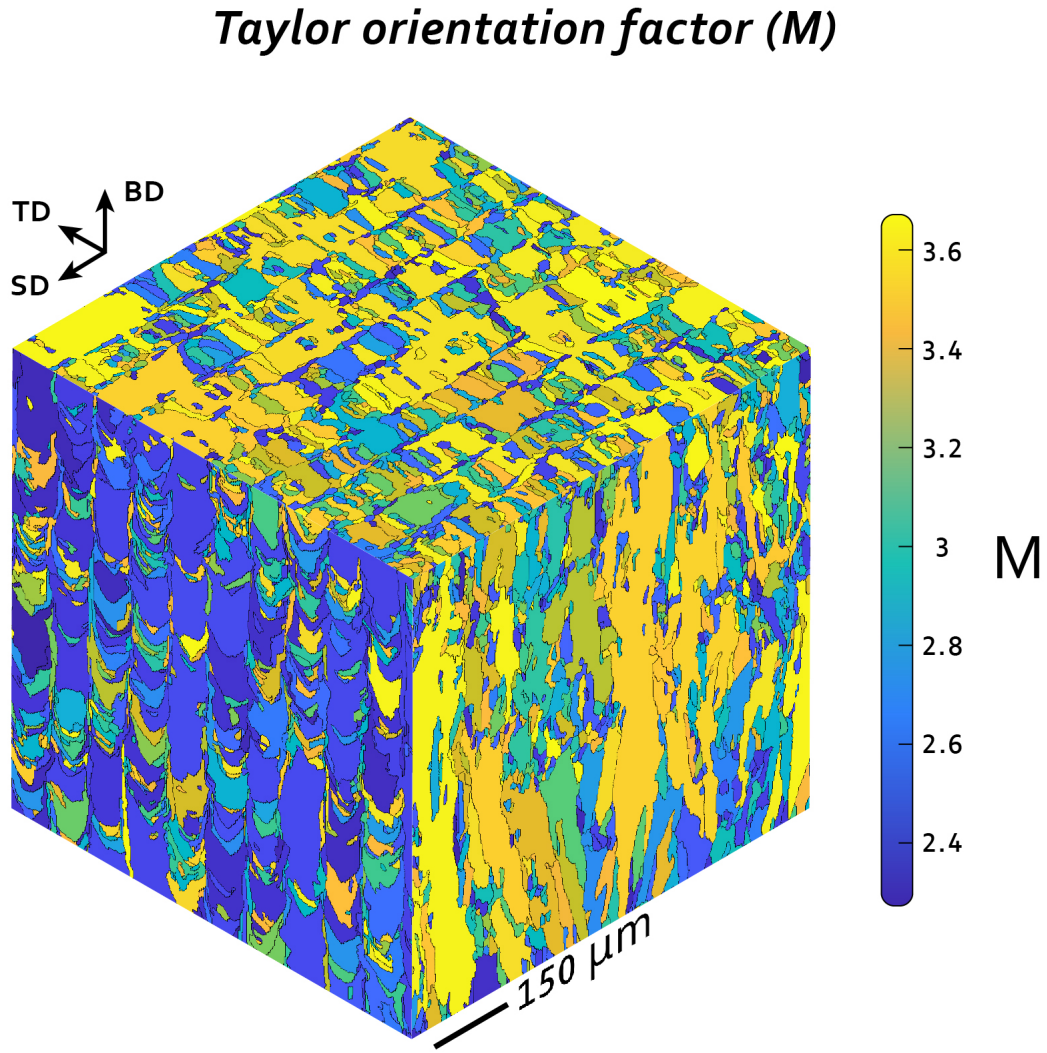


Figure 4.6: The corresponding Taylor orientation maps M of the SLM-manufactured specimens. It can be seen that the TD-BD exhibits low M compared to the SD-BD and SD-TD planes [183].

As for dislocations, it is convenient to discuss their density in the three-dimensional space. Specifically, when discussing the dislocation density for the SLM sections (SD-TD, SD-BD, and TD-BD), we should think of those three sections as two-dimensional resemblances of a three-dimensional object. Thus, the dislocation density in the three-dimensional volume is the same, no matter what two-dimensional section we look at. However, there is a small subtlety involved. Because the specimens were fabricated with different orientation onto the SLM build plate, the thermal gradients and cooling rates per layer are not exactly the same. As a result, the cellular dendrites' orientations/trajectories are not the same; and because these are different, the dislocation density should vary. However, even if the dislocations were assumed to be similar, the dislocation interactions that occur based on loading from a different direction are another story. Because of the

SLM material's texture, the dislocation-dislocation interactions cannot be assumed to be similar for all loading directions. Nonetheless, comparing the dislocation densities of the SLM- with the conventionally manufactured specimen is relatively straightforward. The dislocation density is expected to be far less in the conventionally manufactured specimen compared with the SLM-manufactured one. The reason is due to the annihilation of dislocations as a result of the annealing. The dislocations in the SLM can be seen in the ECCI micrographs of an unetched specimen shown in Figure 4.7a,b. While lack of etching conceals the cellular dendrites, it provides better contrast for observing dislocations using ECCI. The dislocations assume the shape of a honeycomb-like structure surrounding the cellular dendrite. The width of these honeycomb-like structures spanned about 400 nm. Observing the honeycomb-like structure typically means that the solidification direction is more or less perpendicular to the screen. Nonetheless, when the honeycomb-like structures appear stretched, this means that their axis is misaligned from the axis of the screen (as highlighted on Figure 4.7a). In Figure 4.7a, the honeycomb-like structures appear to have unequal major and minor axes. As such, when the sample is cut parallel to the screen, the honeycombs-like structures have a non-uniform aspect ratio. On the other hand, the dislocations may be observed as parallel lines, indicating that solidification direction is parallel to the screen. This alignment can be seen in Figure 4.7b where the dislocations appear as continuous lines with a uniform spacing in-between.

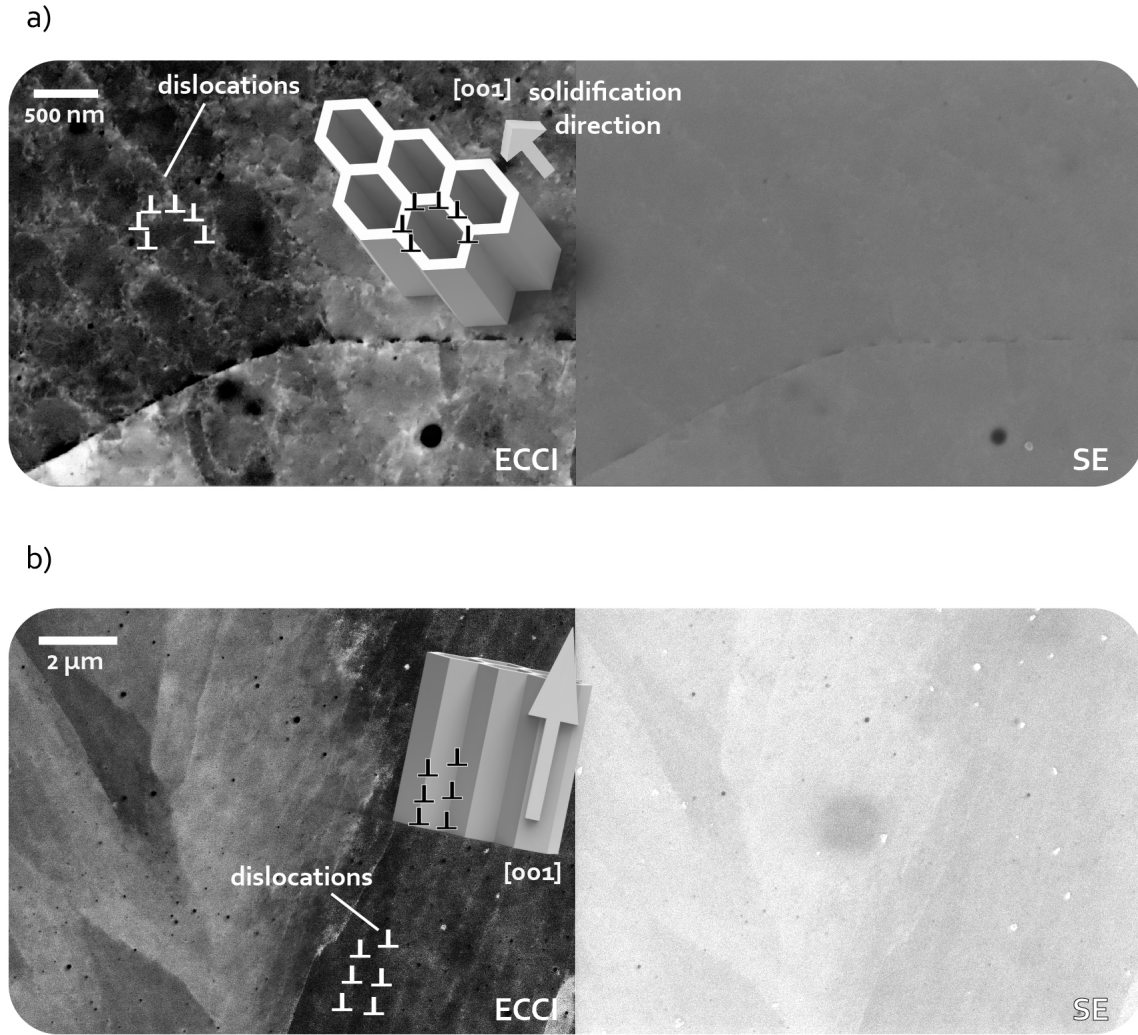


Figure 4.7: Dislocations in the as-built, SLM-manufactured 316L showing (a) cellular dendrites that are almost perpendicular to the screen versus (b) cellular dendrites that are almost parallel to the screen [183].

In the case of the SLM specimens, the texture's impact on the hardness was appreciable. This impact was observed on the hardness of the TD-BD section, which lower than the other two. In addition, the large grains on the two-dimensional section of the TD-BD section may have contributed to its weaker hardness value.

As for the grain size, it is important to consider that the SLM specimen does not exhibit equiaxed grains like the conventionally manufactured specimen. In other words, depending on the sectioning, the grains may appear larger or smaller. As a result, the SLM material is expected to exhibit anisotropic behavior, regardless of the texture. Nonetheless, the combined effect of the texture and non-equiaxed grain morphology makes SLM materials highly anisotropic. For example, the TD-BD section in this case has the lowest Taylor orientation factor compared to the other sections, as well as the largest grain dimensions.

Therefore, it makes sense its measured hardness (i.e., strength) was the lowest of the three. On the other hand, the annealing of the conventionally manufactured specimen (WD) resulted in reducing its hardness below all of the SLM specimens.

4.2.2 Wear performance

The impact of increasing load on the wear rates for different specimens is shown in Figure 4.8. It can be seen that the conventionally manufactured specimen (WD) has flatter curve compared to the SLM-manufactured specimens. In fact, a decreasing trend can be seen in the wear rates of the SLM-manufactured specimens with increased load. Nonetheless, each of the SLM-manufactured specimens exhibited a different trend. For example, SD-TD shows the fastest, non-linear decline with increased load, whereas SD-BD and TD-BD both declined linearly. Nonetheless, even between SD-BD and TD-BD, there was a noticeable difference where SD-BD's declining wear rate flattened from 2.94 N onward.

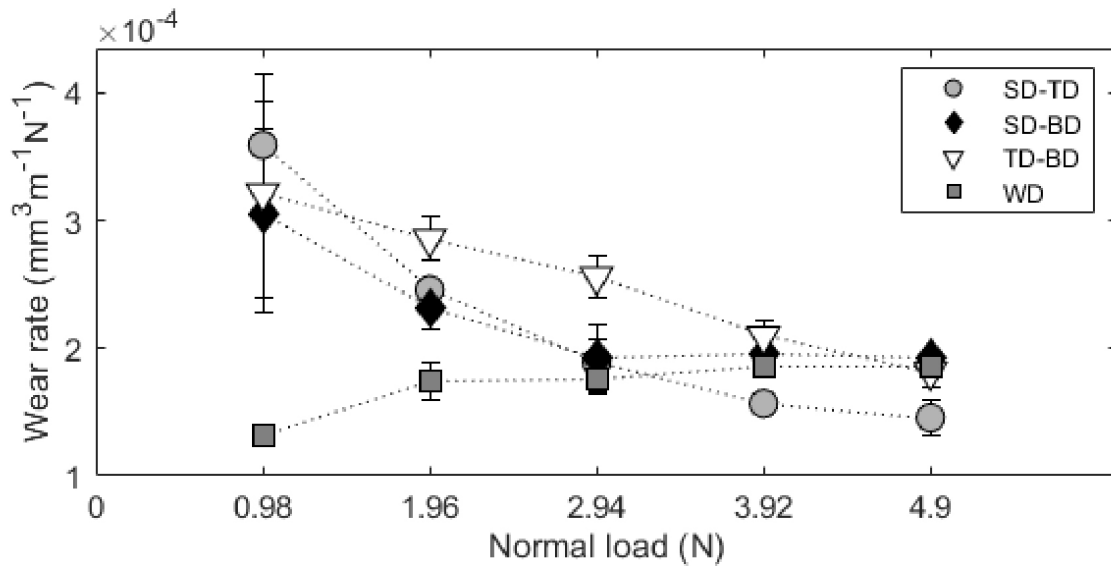


Figure 4.8: The wear rate of each specimen as a function of applied normal load [183].

Figure 4.9 presents a side-by-side visual comparison of the wear tracks from the same load which shows that wear mechanisms vary based on the specimen type. In particular, the SD-TD and SD-BD wear tracks look similar when compared to the TD-BD or the WD wear tracks. In fact, the TD-BD and WD wear tracks contained dark and bright patches, respectively. These different wear behaviors allude to different mechanisms of wear.

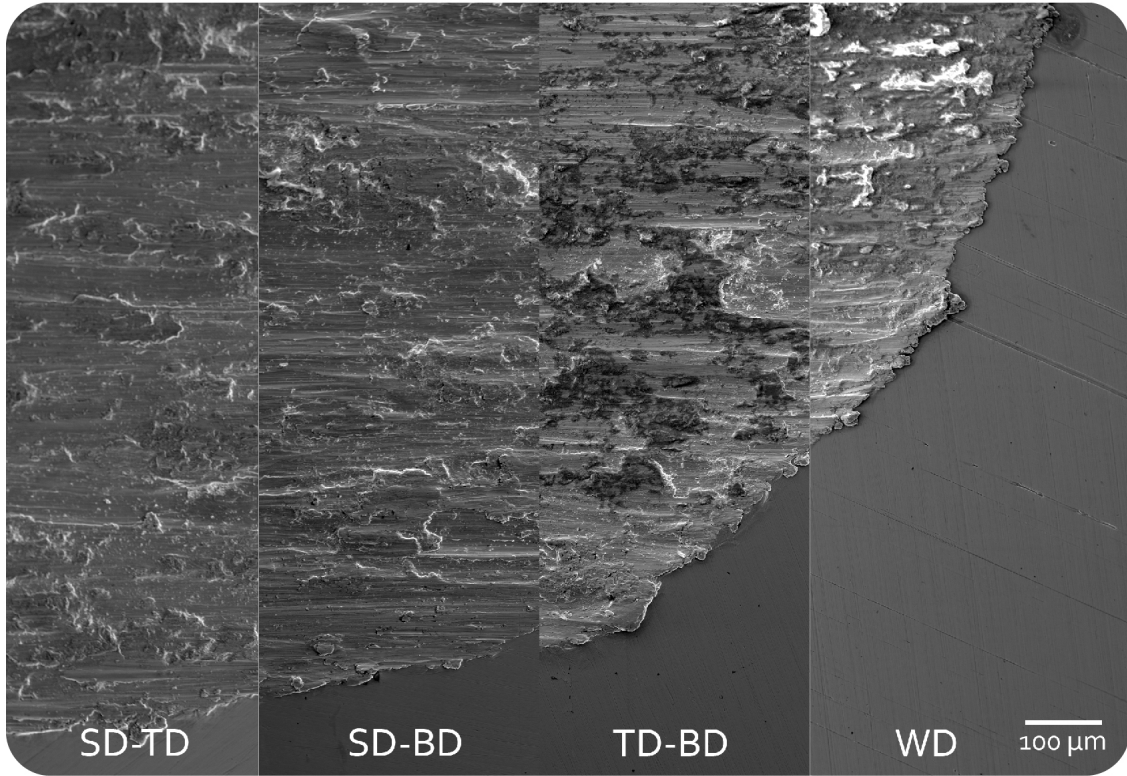


Figure 4.9: Scanning electron micrograph of the wear tracks side-by-side. Bright patches appear on the WD specimen, whereas dark patches appear on the TD-BD specimen. In comparison, neither the SD-BD nor the TD-BD showed traces of these patches. The SEM micrographs were of tracks subjected to 4.9 N normal load.[183].

These SEM micrographs highlight that different specimens exhibit different wear mechanisms, which ultimately impact their wear performance. The following sections will discuss these wear mechanisms in greater detail.

4.2.3 Porosity and fracture

Eliminating porosity is key in preserving the mechanical integrity of SLM-manufactured specimens. In dry sliding contact, these porosities can unfavorably exacerbate the wear rates [126, 186, 187].

SEM inspection of the wear tracks revealed craters as wide as $200\text{ }\mu\text{m}$, which were prominent in the TD-BD specimen (Figure 4.10a). These craters were attributed to the pre-existing porosities on the specimen's surface (Figure 4.10b). The fact that these porosities were present both on the bottom of the wear track and on the unworn surface suggests that these porosities span a depth of at least $100\text{ }\mu\text{m}$ (see wear track depth profile in Appx. 6). Furthermore, Figure 4.10b shows that these porosities are lack-of-fusion pores whose spatial dimension is non-uniform. Thus, when the slider slid over these porosities with non-uniform shapes, stress concentrations on the sharp corners of the porosities propagated rapidly, leading to these observed fractures.

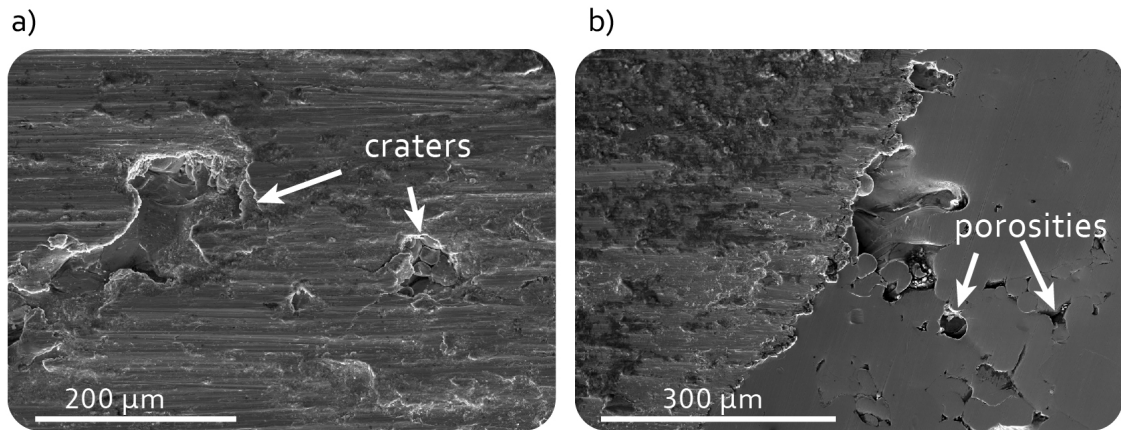


Figure 4.10: Effect of porosity on wear. (a) showing porosity-induced craters. (b) showing the porosities on the unworn surface (outside the wear track) [183].

Yang et al.'s study [188] on porosity formation mechanisms showed that the spatial distribution of porosity is impacted by the scan strategy and build orientation. The specific scan strategy used in PhD project was unfavorable to the tribological surface of TD-BD; specifically because the layer scanning end points coincided with the tribological surface of TD-BD. Figure 4.11 shows the three SLM-manufactured specimens to highlight why the lack-of-fusion porosity was prominent for the tribological surface TD-BD. The end points in this uni-directional scanning strategy correspond to sharp drops in energy densities (arising from switching off the beam), and thus correspond to sites for lack-of-fusion porosities. As such, TD-BD is more susceptible to fractures as a result of sliding contact.

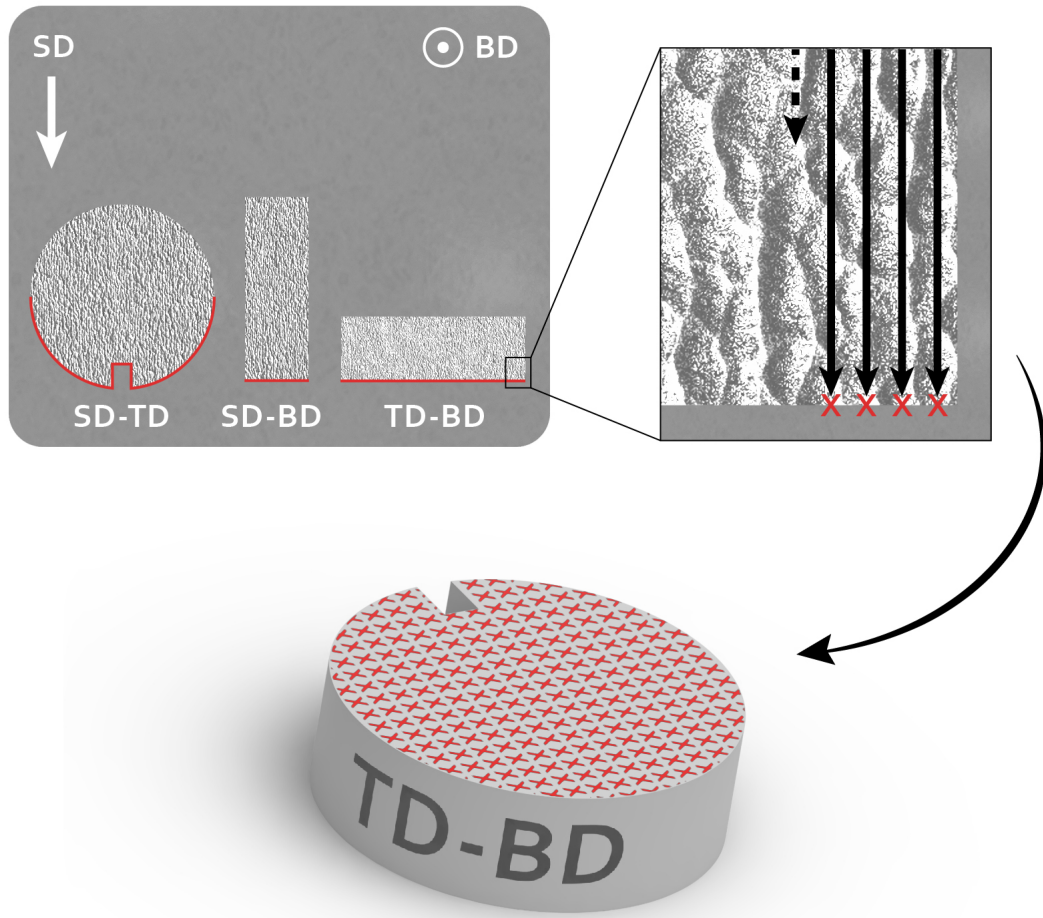


Figure 4.11: A schematic of the laser scan end-points in each specimen type. In the TD-BD, the endpoints coincided with the tribological surface, which ultimately led to higher frequency of porosities compared to the tribological surfaces of the other SLM-manufactured specimens [183].

Reevaluating Figure 4.8 in light of these porosity-induced fractures suggests a correlation between the two. While it can be generally stated that increase in normal load led to a decrease in wear rate for the SLM-manufactured specimens, TD-BD's decline was different than the other two. It can be seen in Figure 4.8 that the trend of decline in SD-BD and SD-TD were non-linear, while in the TD-BD it was linear. In other words, the attribute of declining wear rate with increased load for TD-BD was counteracted by the porosity-induced fractures, hence, the wear rates declined slowly compared to the other SLM-manufactured specimens. Nonetheless, it should be noted that the wear rate of SD-BD flattened out towards the greater normal loads. This observation may be hint that an isotropic behavior has come into play. Alternatively, it may be the case that the increased load led to pore filling and thus reduced the wear rate.

Beyond the porosity-induced fractures, it is crucial to highlight that the rate of wear is a collective result of many controllable and uncontrollable factors, such as, load, speed, distance, material(s), geometries, temperatures, etc. Upon changing, these factors may or

may not lead to a change in wear mechanism. Thus, Sun et al. [126] correctly pointed that porosity does contribute to wear rate. Nonetheless, porosity should be put in context with all the aforementioned factors which can lead to changes in wear mechanisms and thus impact the wear rate as well.

4.2.4 Oxidative wear

Although it is usually approximated that the Hertzian contact of a ball-on-flat would be of a circular shape, the actual contact comprises individual asperities with various heights and radii often undergoing extreme pressures [189]. When two asperities interlock during sliding, the asperity which gives in will first be removed from its parent surface and end up adhering to either of the two sliding surfaces via material transfer or mixed as a third layer. However, in this case, it is difficult to ascertain via EDS/EDX which of the two bodies contributed the most towards material transfer given that both the ball and specimens were made of the same material (316L). Furthermore, it is far more difficult to predict which of the two asperities will prevail considering the number of factors involved (geometry, angle, height, height-to-width ratio, hardness, etc.). Nonetheless, here we will consider one of the readily available factors which is the hardness value of the parent material.

The hardness ratios between the ball and specimens ($HV_{specimen/ball}$) are given in Table 4.2. Experimental studies of sliding metal pairs showed that the closer the ratio to 0.8, the more susceptible a material to severe wear [163]. It can be seen that the ratios corresponding to the SD-TD and SD-BD are equally the greatest. Whereas the ratio corresponding to TD-BD was slightly lower than those two. Finally, the ratio corresponding to the conventionally wire-drawn specimen (WD) was the lowest. Thus, the specimens can be ordered from the softest-to-hardest as follows: WD, TD-BD, SD-TD/SD-BD. This order correlated with the wear mechanisms observed previously in Figure 4.9 as will be discussed later.

Table 4.2: The specimen-to-ball hardness ratios. Ball is made of 316L stainless steel [183].

$HV_{specimen/ball}$	Ratio
$HV_{SD-TD/ball}$	≈ 1.05
$HV_{SD-BD/ball}$	≈ 1.05
$HV_{TD-BD/ball}$	≈ 0.99
$HV_{WD/ball}$	≈ 0.94

Although 316L exhibits superior corrosion resistance, it does suffer from corrosive attacks if the chromium-induced passive film (which protects from corrosion) is removed. Upon removal of this film, the bare surface of 316L can react with the atmosphere and oxidize, which is known as oxidative wear. However, depending on the chemical composition of the material and the surrounding environment, this passive film can self-heal and protect the material from corrosion/oxidation.

Figure 4.12 compares the oxygen present in the wear tracks under different loading conditions. It can be seen that there is no particular trend with which all specimens comply; rather, each specimen exhibited a unique behavior. However, it is clear that the conventional specimen (WD) suffered the most from progressive oxidative wear as loads increased. In fact, the oxide started agglomerating even at lower loads, but they grew larger as the load increased (note: track the size of the white masses on the WD panels). The oxide agglomerates in WD's wear track were found at the end of grooves (prows) (Figure 4.13). This finding is almost identical to Rainforth et al.'s sliding wear test on 316 stainless steel against a ceramic counter body [190]. Rainforth et al. suggested that these prows formed due to the transfer of metal to ceramic followed by a progressive re-transfer from the ceramic back to the metal. However, once the material has been detached from its parent surface, it oxidizes and thus is re-transferred as an oxide which adheres back to the parent surface. A similar trend, yet significantly milder in magnitude, was observed for the TD-BD specimen. Nonetheless, the oxide agglomerates only took form towards the maximum load. As for SD-BD and TD-BD, both showed onset of oxide agglomeration at 2.94 N, which then subsided when the load was increased to 4.9 N.

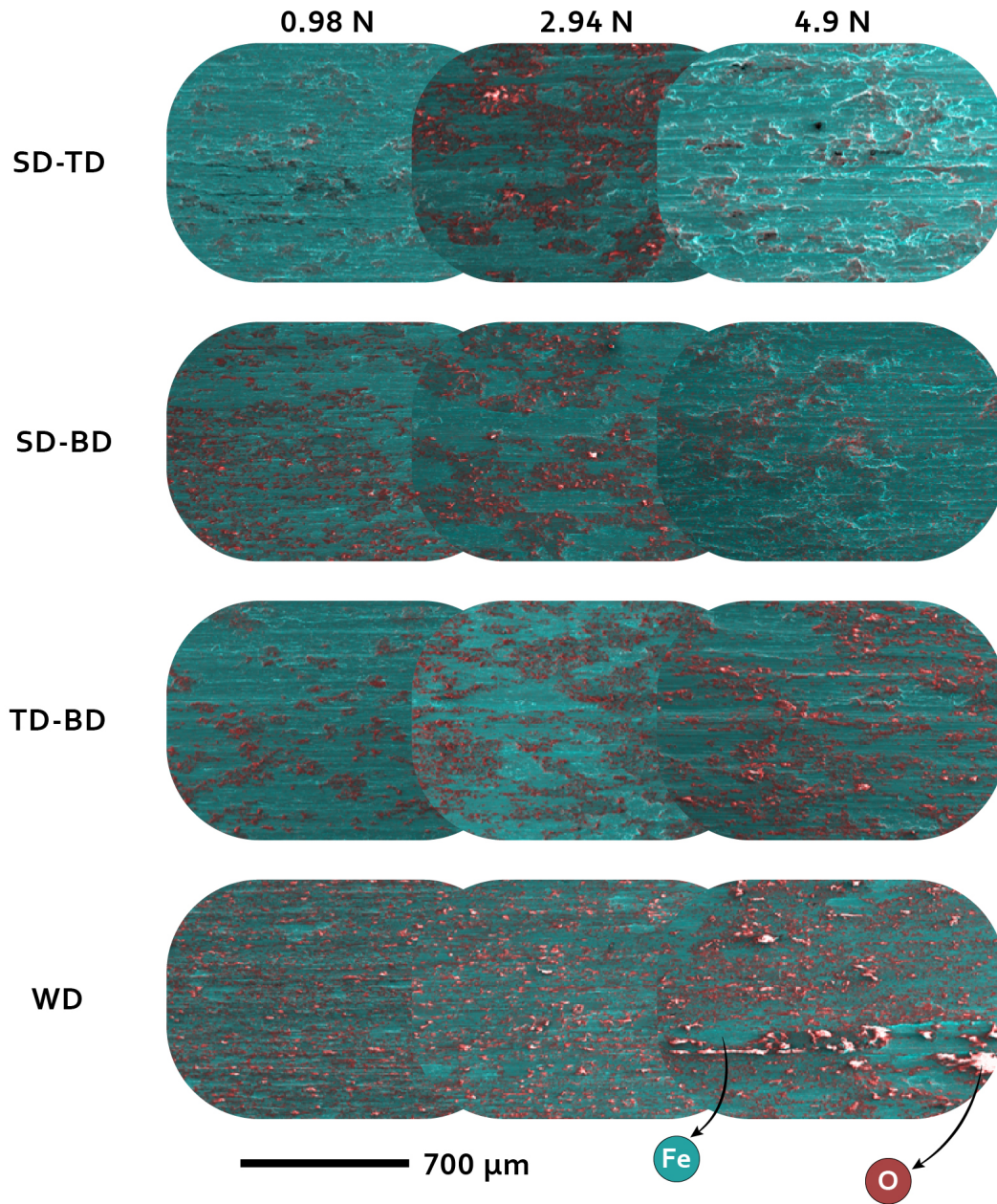


Figure 4.12: EDS mapping of each specimen's wear track in 0.98, 2.94, and 4.9 N loading conditions. The maps compare the level of oxidation in each specimen under these loads [183].

Thus, we see that the susceptibility to adhesive and oxidative wear varies even with materials of nominally similar composition. In fact, even when the material possessed identical composition (i.e., the SLM-manufactured specimens), the adhesive and oxidative wear response varied due to anisotropy.

Several studies compared the corrosion potential of SLM- vs. conventionally manufactured 316L [191–194]. The findings indicated that the passive film is thicker in SLM-manufactured 316L; but the passive film becomes thinner upon heat-treating the SLM-manufactured specimens. The reason this film is thicker in SLM-manufactured 316L is owed to the Cr-Mo-enriched solidification cellular dendrites, which serve as nucleation sites that accelerate the film’s growth. Of course, this passive film would also enhance the SLM-manufactured 316L specimens’ resistance to oxidative wear. As such, their susceptibility to oxidative wear is lower than the conventionally manufactured specimen. This remark is most likely the reason why the conventionally manufactured specimen suffered the most from oxidative wear.

Although the three-dimensional microstructures of SLM-manufactured specimens are statistically the same, the spatial variations within the microstructures stipulate that the material behaves anisotropically, at least under certain circumstances. On top of this, the high-density defects on the tribological surface of TD-BD also stipulate that the tribological behavior be different. The spatial variation of the microstructure entails that the cross-sectional density of Cr-Mo-enriched cellular-dendrites vary based on the sectioning (i.e., SD-TD, SD-BD, and TD-BD are not the same). On that basis, the different SLM sections may possess different tribological resistance to oxidative wear due to varying speeds of passive film growth. Of course, the susceptibility to material removal/transfer due to varying hardness must also be taken into account. Furthermore, studies have shown that porosities can serve as nucleation sites for corrosive attacks [191, 193]. Therefore, it is expected that the specimen whose tribological surface was heavily impacted by porosities (i.e., TD-BD) would exhibit a higher susceptibility to oxidative wear than the other two SLM-manufactured specimens.

In conjunction with these interpretations, it is crucial to note a number of complexity-based limitations. First, given that oxygen build-up changes from layer to layer in the printing process, the susceptibility to oxidative wear may vary within the same specimen when assessed across different layers. In addition, variation in the humidity and/or temperature between experiments may improve or impair the potential to oxidative wear.

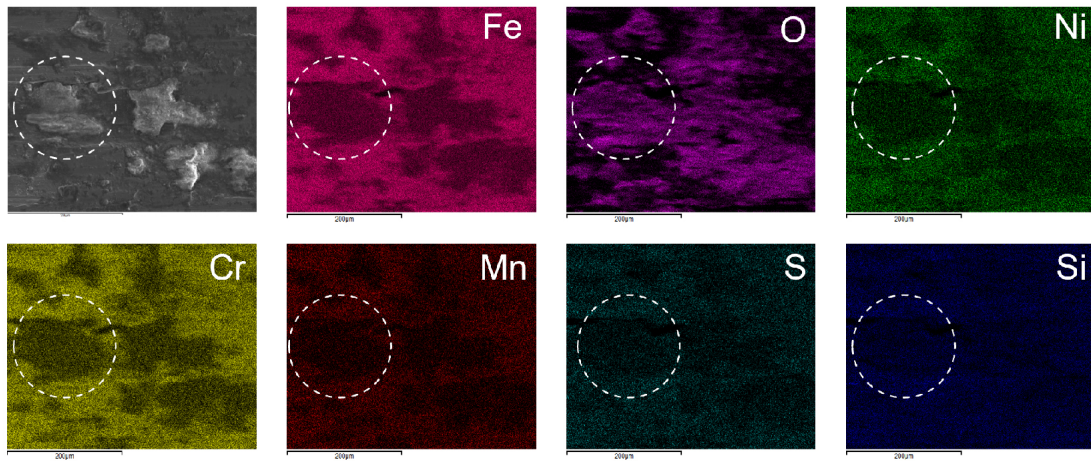
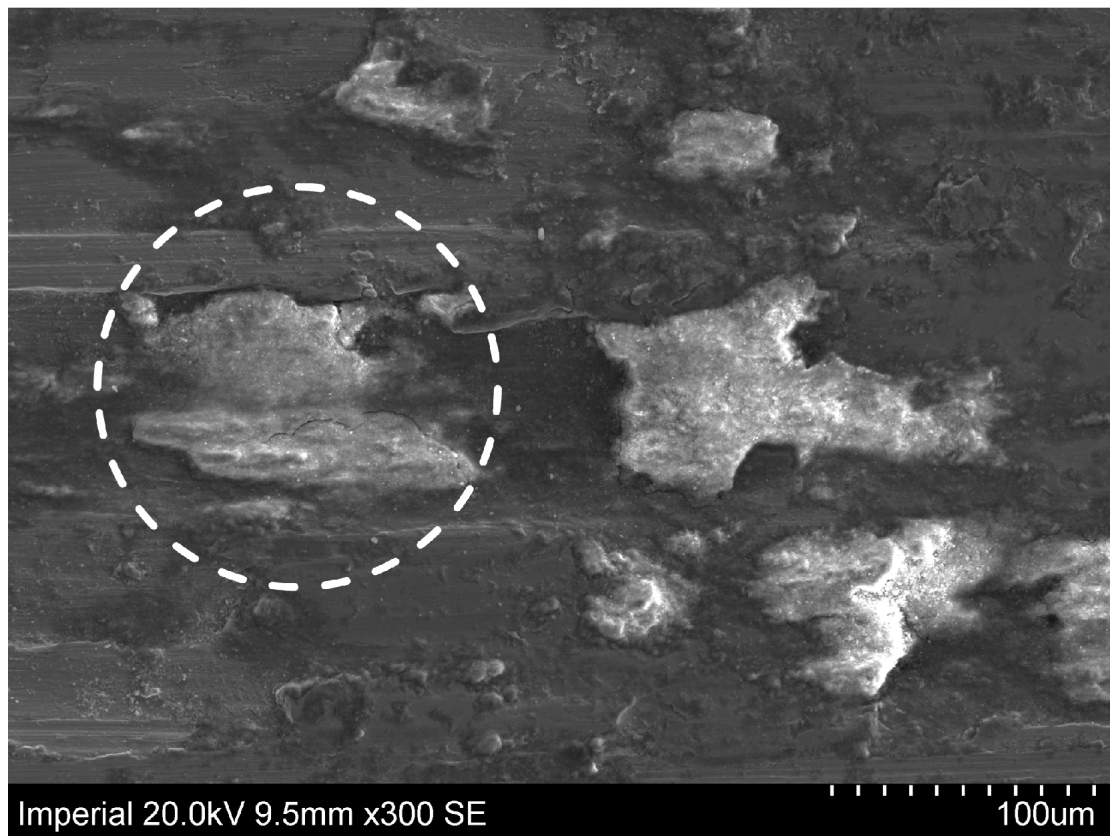


Figure 4.13: SEM and EDS analyses reveals that agglomerates are oxides which were found as prows at the end of grooves. [183].

4.2.5 Delamination wear and plastic deformation

Although Archard's adhesive wear theory [159] has been immensely useful and thus widely accepted, it was criticized by Suh [162] for ignoring the physics of metal deformation. On the other hand, Suh's theory is based on ductile-fracture mechanisms arising from sub-

surface plastic deformations. The theory postulates that continuous sliding on the surface leads to pile-up of dislocations in the subsurface. Subsequently, these pile-ups lead to a formation of a void, from which a crack propagates towards the surface. When the crack finally reaches a certain length, the surface detaches (i.e., delaminates) in the form of a sheet-like debris. Therefore, the delamination theory is can help explain the sub-surface plasticity. For example, we can explain the plasticity occurring in the subsurface via Eq. 2.6 which takes into account the microstructure constituents, such as the dislocation densities (ρ), Taylor orientation factors (M), and grains sizes (D). For example, the equation accounts for the high-density of dislocations at the cellular dendrites' boundaries. Furthermore, it takes into the account the crystallographic texture via the M -values shown in Figure 4.6. However, the Suh's delamination theory has also been criticized for neglecting the fact that the maximum critically resolved shear stress occurs at the surface [190].

Nonetheless, the contribution of the constituents above must be put into the context of the tribological set-up. In this case, the wear track spanned a width and depth of 2400 μm and 100 μm , respectively, whereas the cellular dendrites' cross-sectional width was about 400 nm. Furthermore, the cellular-dendrites were not well-arranged (Figure 4.3). With these aspects considered, the contribution of cells to the wear behavior is probably minute, compared with the crystallographic texture and grain boundaries. Nonetheless, the wear anisotropy in SLM-manufactured 316L has been reported to be more prominent at lower loads [82], which likely means that the contribution of the cellular-dendritic arrangement on wear increases with decreased normal loads.

Superimposing the idealized Hertzian contact onto the sliding surfaces is a good way of visualizing the cross-sectional interaction between the ball and grain boundaries of different specimens. Especially considering that sliding induces dislocations which largely pile-up at the HAGBs. Figure 4.14 depicts the Hertzian contact ($r = 45.29 \mu\text{m}$) for an arbitrary sliding distance ($d = 1000 \mu\text{m}$) and the maximum normal load used in this study ($N = 4.9 \text{ N}$). It can be visually appreciated that sliding on the SD-TD surface engages more HAGBs than any other specimen. As such, this specimen's work hardening is expected to be faster than others. And indeed, its wear rate shows this behavior (Figure 4.8). In comparison, the HAGB intersections in SD-BD is less than SD-TD, and TD-BD is less than SD-BD (and accordingly, their work hardening rates are proportionally affected). Although WD visually shows a large number of HAGB intersections, the impact of such on its work-hardening attribute cannot be compared with the SLM-manufactured specimens in the same proportion. Because the annealing in WD eliminated many of the dislocations which lead to work hardening. Therefore, it is reasonable to assume that WD would be the slowest material to work-harden.

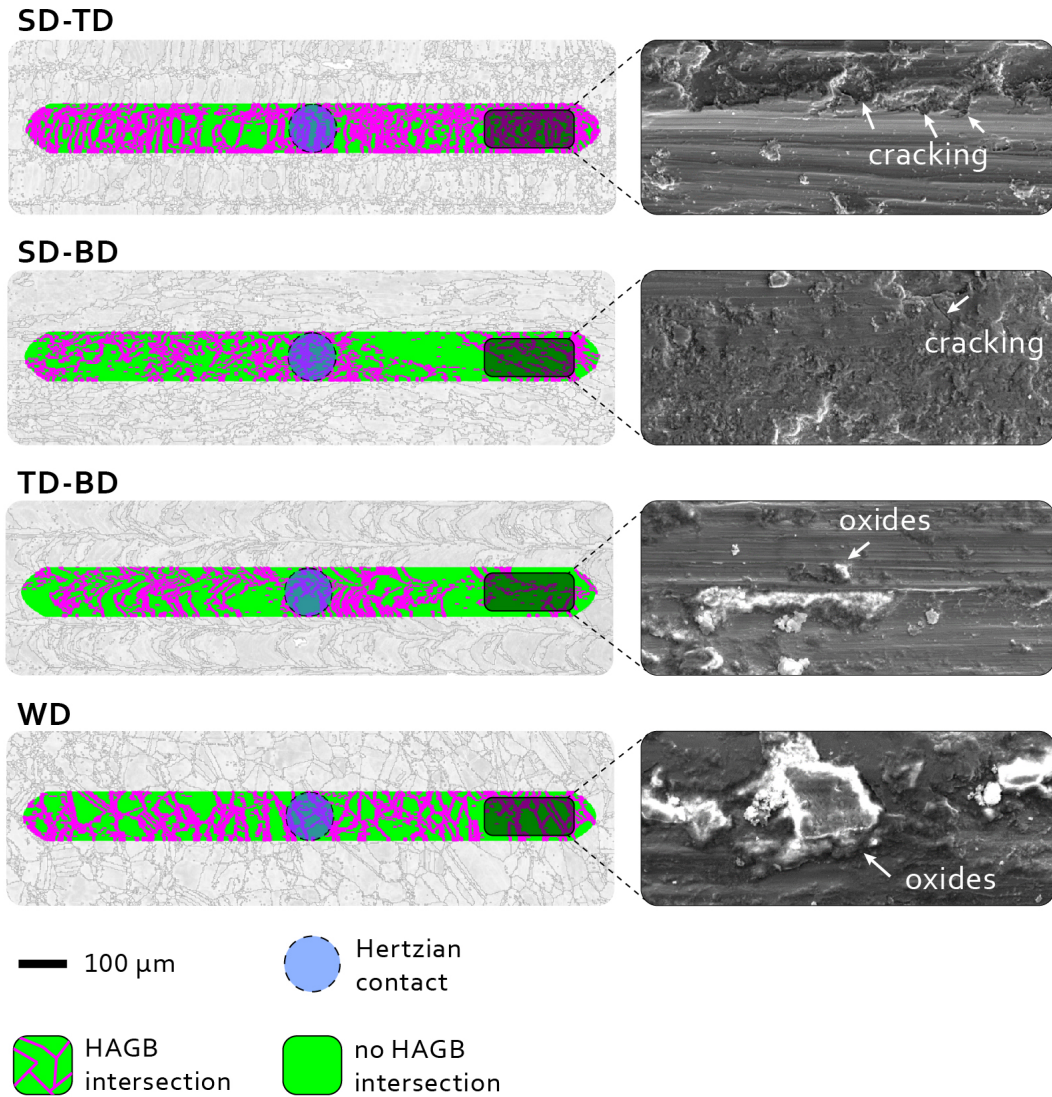


Figure 4.14: A comparison of the high-angle grain boundary (HAGB) intersections with the Hertzian contact in each specimen. On the side of each track, a resembling SE micrograph of the wear track [183]. These SEM micrographs were taken from a wear track subjected to a normal load of 4.9 N.

The wear tracks in Figure 4.14 reveal different wear mechanisms that can be correlated to the microstructure. The SD-TD showed traces of cracking and delamination, whereas SD-BD showed smaller magnitudes of cracking. On the other hand, the TD-BD showed conspicuous mechanism of adhesion and mild oxidation. Lastly, the WD specimen showed the strongest corrosion mechanism in the form of large oxide agglomerates. As established earlier, because the work-hardening rate is of the following order: SD-TD; SD-BD; TD-BD; and then WD, the susceptibility to crack formation also follows the same order. This would explain why the cracked chips were prominent on the SD-TD, and less visible on the SD-BD. And also why no indication of cracks or delaminated grains found on TD-BD nor WD.

It is known that mechanically mixed wear debris, also known as the tribo-layer, has the potential to contribute to the wear and friction. Depending on the composition of this tribo-layer it can be harder than its parent material [195]. As a consequence, it can contribute to abrading the material and thus increase the wear rate. This effect can play an important role when the phase shifts to a harder phase (e.g., from austenitic to martensitic). However, this effect is unlikely in the low carbon content in the present material. Here, the tribo-layer was mostly soft enough to plastically deform between the two bodies and into fine debris. However, its impact on wear rate was not readily apparent nor was in the scope of the project.

4.3 Chapter conclusion

This study focused on underscoring the underexplored role of microstructure on the anisotropic wear mechanisms in additively manufactured 316L by selective laser melting (SLM). To this end, the study put into context multiple aspects involving the materials under investigation as well as the tribological set-up. It was found that wear mechanisms do transpire anisotropically in SLM-manufactured 316L, owing to a number of key factors: 1) The Taylor orientation factor, 2) the work-hardening-induced dislocation densities, and 3) the grain morphology at the contact surface, which can be visualized by means of HAGBs. The hardness of the material and the porosity do offer valuable insights, however, none of which is the sole determiner of whether SLM-manufactured 316L is superior or inferior to conventionally manufactured 316L. Rather, all factors, especially the microstructure must be considered in parallel in order to achieve a realistic interpretation.

Chapter 5

In-situ observation of tribological anisotropy

The content of this chapter was adapted from the following publication:

M. Bahshwan, M. Gee, J. Nunn, C. W. Myant, and T. Reddyhoff. “In situ observation of anisotropic tribological contact evolution in 316L steel formed by selective laser melting”. In: Wear 490 (2022), p. 204193. issn: 0043-1648. doi: <https://doi.org/10.1016/j.wear.2021.204193>

5.1 Chapter introduction

A recent review of literature which compared the tribological performance of SLM- versus conventionally manufactured 316L reveals interesting findings. Some studies [183, 196] reported lower CoF values in the SLM-manufactured specimens, while other studies [5, 181] reported the opposite. Similarly, some researchers [5, 82, 181, 196] reported superior wear resistance in SLM-manufactured specimens, while others [6] reported inferior wear resistance. Such discrepancies naturally lead one to question the reason behind them. Are some of the results correct and others are not? Or are they all correct in their own contexts?

As conspicuously highlighted in 2.4.1, studies covering Tribo-SLM316L have implemented a wide range of different tribological configurations that make it almost impossible to compare the findings by separating them from their methodological contexts. For example, some studies used dry [6, 82, 126, 147, 148, 150], whereas others used lubricated tests [5, 127, 151]; some used room temperature environment [6, 82, 126, 147, 148, 150], whereas others used high-temperature environments [146, 149]; some used linear sliding configurations [82, 147, 149, 150], whereas others used rotary sliding [6, 126, 146, 148] configurations; and so on. Distilling findings from these studies into one overarching conclusion which states that SLM- or conventionally manufactured 316L is ‘superior’ to the other would assume that their tribological response to these different configurations is identical. However, the results based on Chapter 4 have shown that the underlying microstructure in 316L by SLM does not tribologically behave similarly to conventionally manufactured 316L [183]. Furthermore, the magnitude of anisotropic response in SLM-manufactured 316L is dependent on experimental parameters such as the normal load [82, 183]. In fact, Yang et al. [82] reported that anisotropic in SLM-manufactured 316L behavior is more prominent under lower loads. Thus, it would be sensible to plan the experimental parameters in accordance with the specific qualities which need to be investigated.

Accordingly, this study aims to better understand the anisotropic behavior of SLM-manufactured 316L as well as compare its behavior to conventionally manufactured 316L. To do so, it is convenient to perform the investigation in micro-scale, single-asperity contact as it provides better signal-to-noise ratio compared to experiments on the macro-scale. This is especially true at lower contact loads, as the effect of single asperities can become more pronounced than that of the theoretical Hertzian contact [197]. Furthermore, it would be convenient to visualize the contact evolution as compared to the tribological response of the material. Doing so would integrate the visual evidence with the measured tribological response. Accordingly, an in-situ SEM micro-tribometer, designed and manufactured by the NPL [168], was used to perform the tribological tests. The results herein will underscore the anisotropic tribological response of SLM-manufactured 316L. Furthermore, the results will demonstrate the importance of distinguishing between the tribological response of two nominally similar material (SLM- versus conventionally manufactured 316L) based on their underlying microstructures.

(Note: experimental setup, parameters, and calculations pertaining to this study can be found in 3.3.2).

5.2 Results

5.2.1 Microstructure

The full microstructural characterization was presented and discussed in 4.2.1. As the discussions in this chapter build upon those previously discussed in 4.2.1, the author recommends that it is read before proceeding into this chapter.

5.2.2 Friction

The average CoF values per cycle for each specimen are plotted in Figure 5.1a. In all specimens, the final average CoF values reached steady-state after a continuous decline. Noteworthy, all specimens – except TD-BD – reached steady-state CoF well before the 20th cycle. On the other hand, TD-BD's steady-state was reached only shortly after the 30th cycle. As highlighted in the close-up view on the same figure (Figure 5.1b), there is a small distinction between the steady-state CoF of the SLM-manufactured specimens and the conventionally manufactured one; the steady-state CoF of the SLM-manufactured specimens were ≈ 0.08 while the conventionally manufactured specimen was ≈ 0.07 .

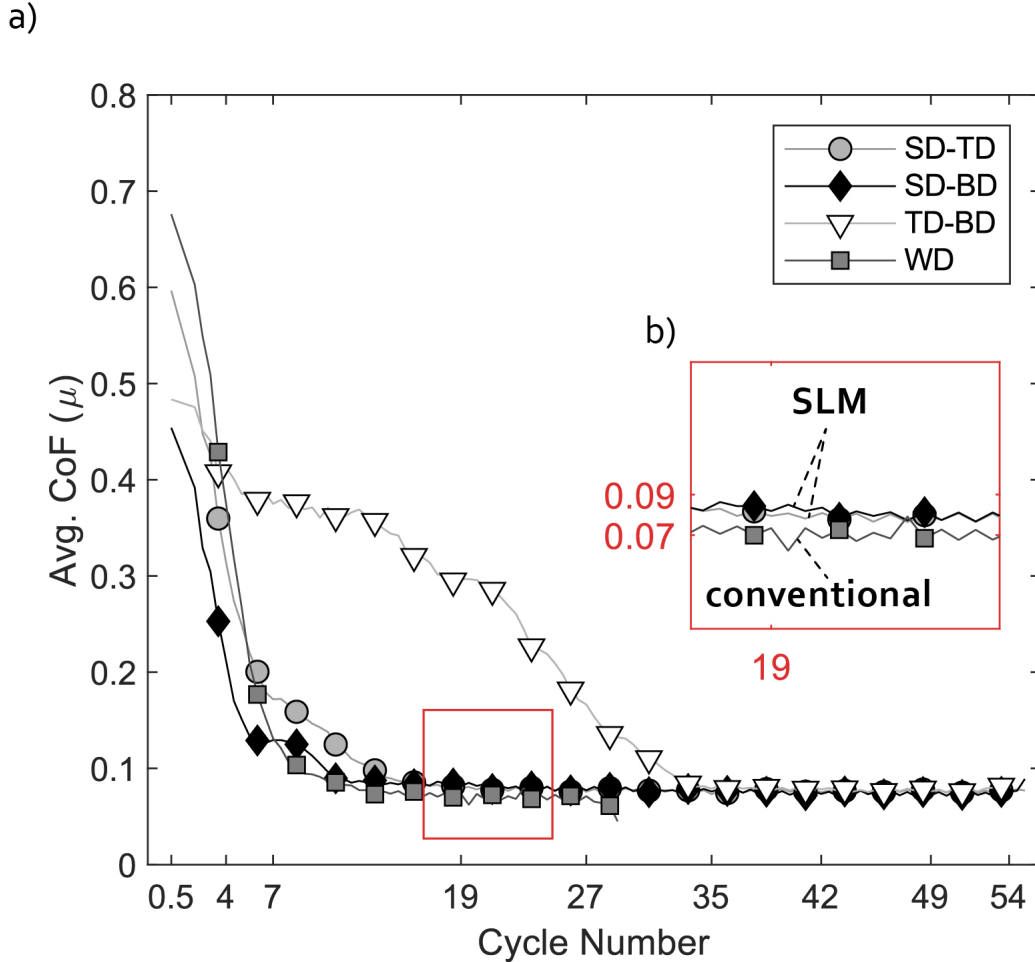


Figure 5.1: Averaged coefficient of friction values per cycle (a) for all specimens. (b) A detailed view showing the small difference in average coefficient of friction between the SLM- and conventionally manufactured specimens [198].

Examining the CoF values without averaging them is also useful in revealing intricacies which may occur along each stroke. Hence, the non-averaged CoF values measured along the stroke were plotted for each consecutive cycle in separate panels in Figure 5.2a-d. The x- and z- axes on the plot correspond to the scratcher's location (between -500 and 500 μm) and the cycle number (between 0.5 and 40), respectively. The mountain-like shapes correspond to spikes in CoF. It can be seen that the spikes in CoF (moving from one cycle to the next) were at times consistent in their position along the x-axis, but at other times completely unpredictable. Despite either case, the spikes tend to generally dampen as cycles progress, which logically led to the decrease in the average CoF presented earlier.

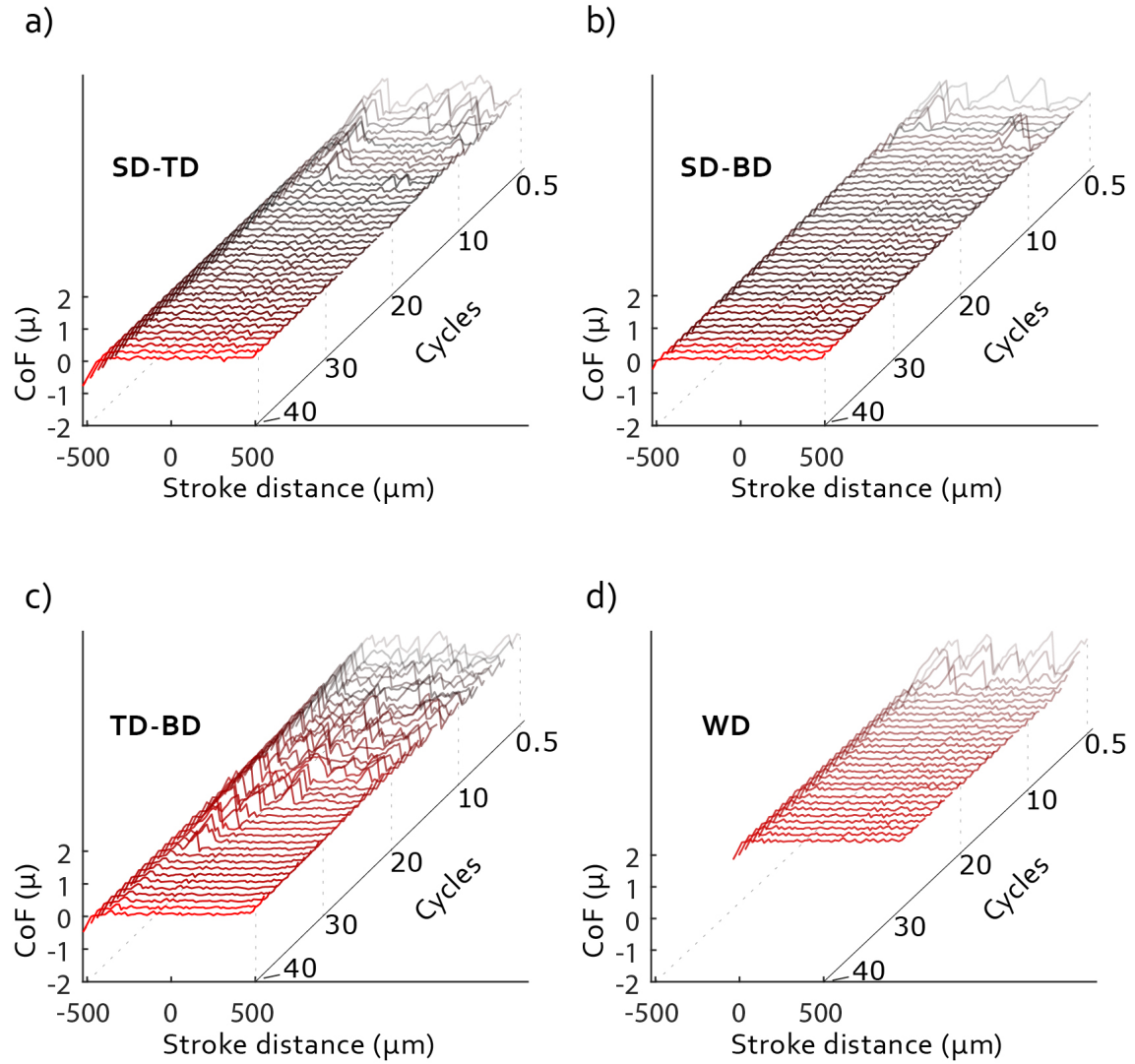


Figure 5.2: Non-averaged coefficient of friction plots per cycle, per stroke distance for (a) SD-TD, (b) SD-BD, (c) TD-BD, and (d) WD [198]. Note: If the reader wishes to examine the CoF values, they may find it helpful to refer to an alternative format of this figure, which can be found in Appx. 9.

5.2.3 Wear onset and evolution

As highlighted in the corresponding methods to this chapter, this experiment consisted of a single-pass and a multi-pass wear tests. The former allows for analyzing the onset of wear, whereas the latter allows for analyzing the evolution of wear.

The wear onset from the first scratch is shown in Figure 5.3a. It can be seen that the wear scar was heterogenous for all samples, where in some locations the damage was heavy but in other areas the damage was light (full tracks are provided in Appx. 7). In the regions with the heaviest damage, the distance between the ridges and the deepest point was about 4 μm (Figure 5.3b). Furthermore, the contact diameter was roughly about 7 μm (Figure 5.3b). It can be seen on Figure 5.3b that the depth of the wear track in those regions transcended the spherical-cap transitional depth. In other words, the contact mode in those areas is predominantly sphero-conical, rather than a spherical-cap one.

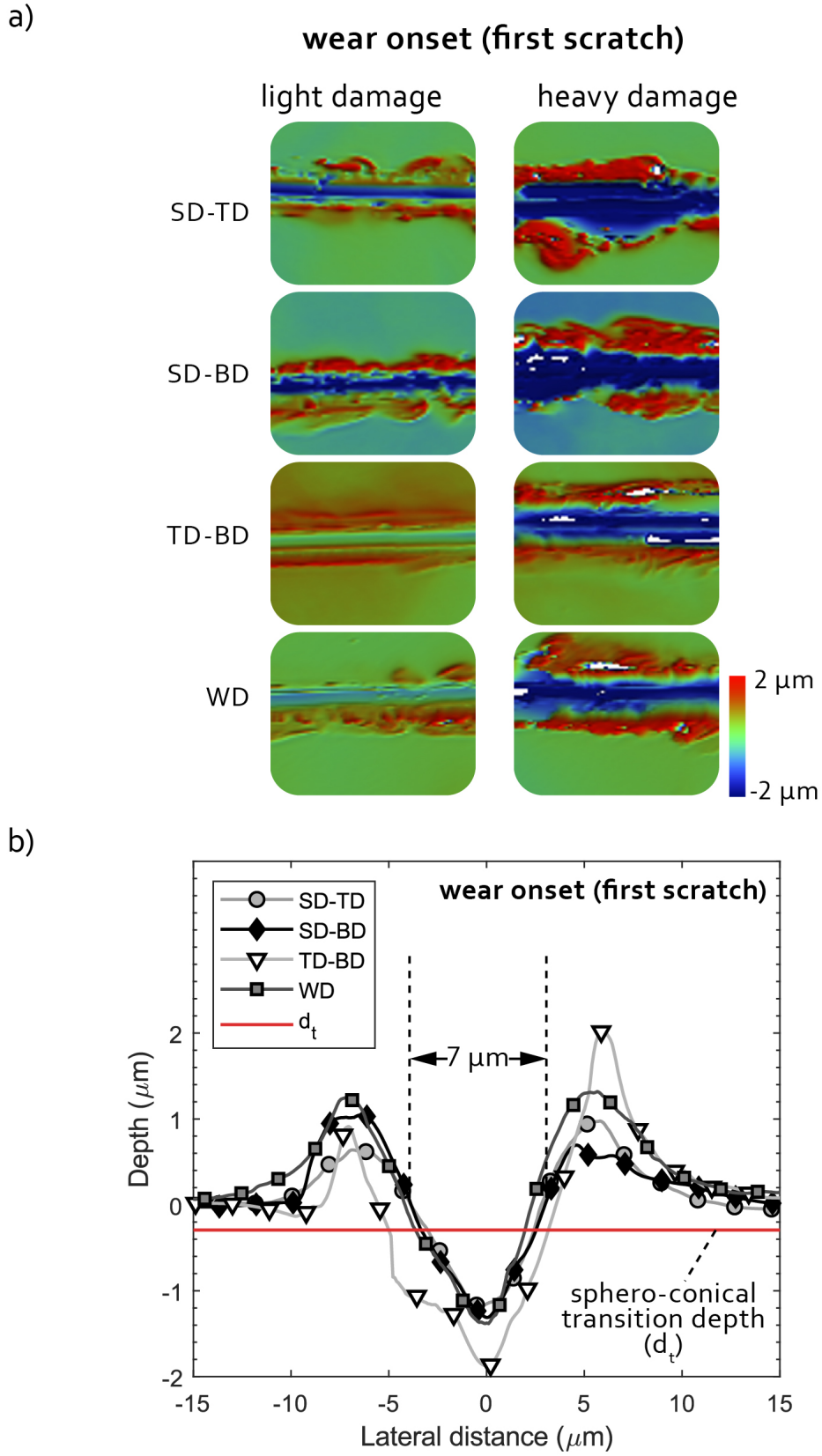


Figure 5.3: The onset wear for all specimens. (a) contrasting the magnitude of damage along the same stroke, and (b) showing the depth profile of the heaviest damage regions. In (b), the contact transitioned to sphero-conical and (i.e., $d_t \gg 0.293\ \mu\text{m}$ defined in Eq. 3.3) its average width was approximately $7\ \mu\text{m}$ [198].

Crystallographic analysis of the interaction between the wear onset and the grains is shown in Figure 5.4a. This figure shows that grains are split in half as a result of the scratch. In both the SLM- and conventionally manufactured specimens, the grains were completely ruptured by the scratches (Figure 5.4a,b). Nonetheless, a subtle reorientation (discoloration) in some areas of the grains were found along the scar's periphery. This reorientation occurred in both the conventionally (Figure 5.4a) and SLM-manufactured (Figure 5.4b) specimens. [198]

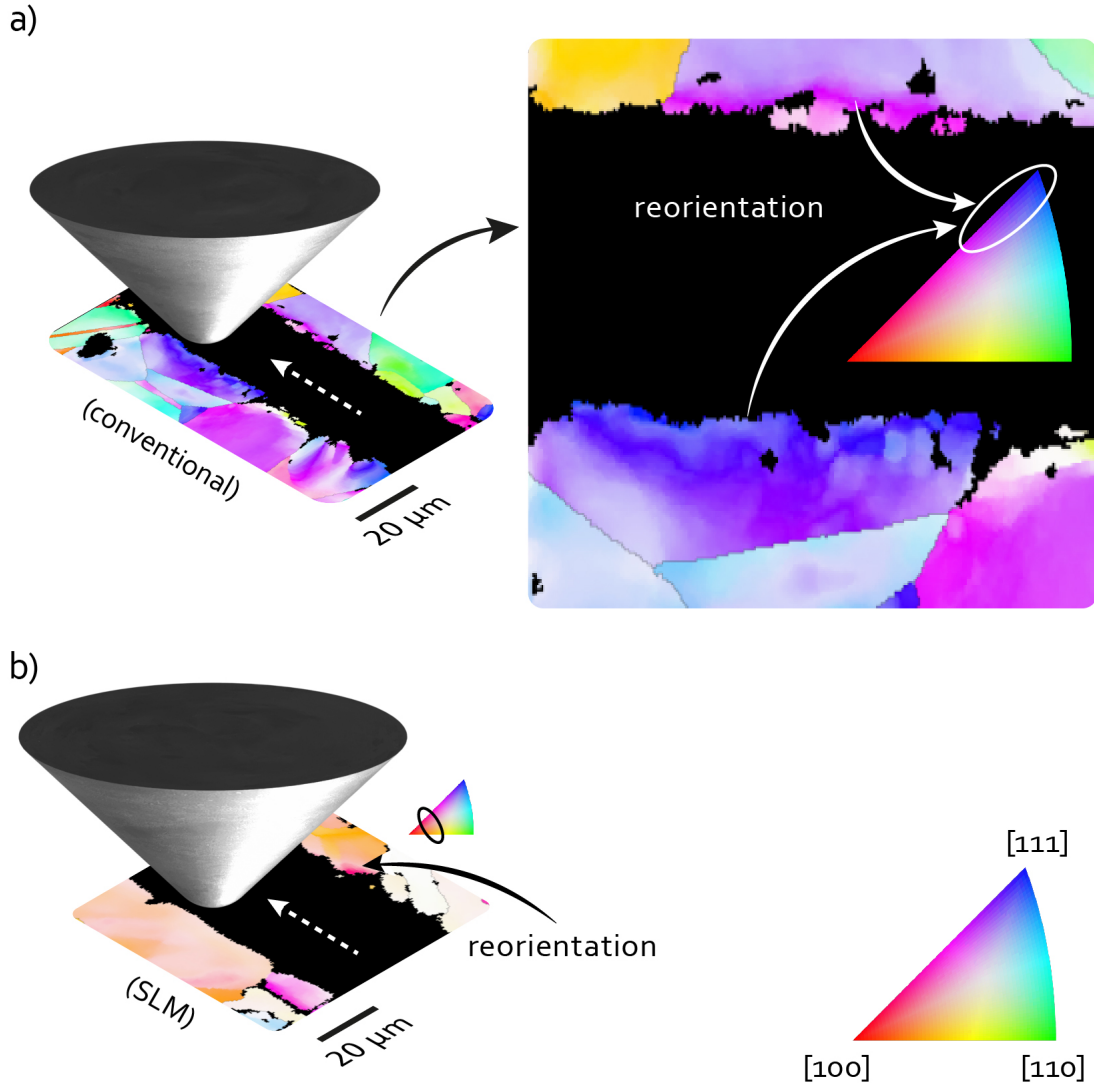


Figure 5.4: Qualitative assessment of grain/crystal interaction with the scratcher in (a) conventionally manufactured specimen and (b) SLM-manufactured specimens. Although the grain was ruptured into two parts, discolorations along the scratch's periphery indicate reorientation [198].

The evolution of wear was assessed both quantitatively and qualitatively. In the former, the minimum conformal scratch width (W^*) was calculated (refer to 3.3.2 for the definition of W^*) and plotted in Figure 5.5. The plot shows that W^* generally increased as cycles increased, apart from specific instances with TD-BD (i.e., cycles 19 and 27). The corresponding applied pressures (P_o) values were calculated and presented on the same figure. It can be seen that despite P_o starting off in the range of 1-3 GPa, it had remained below 1 GPa in the later cycles.

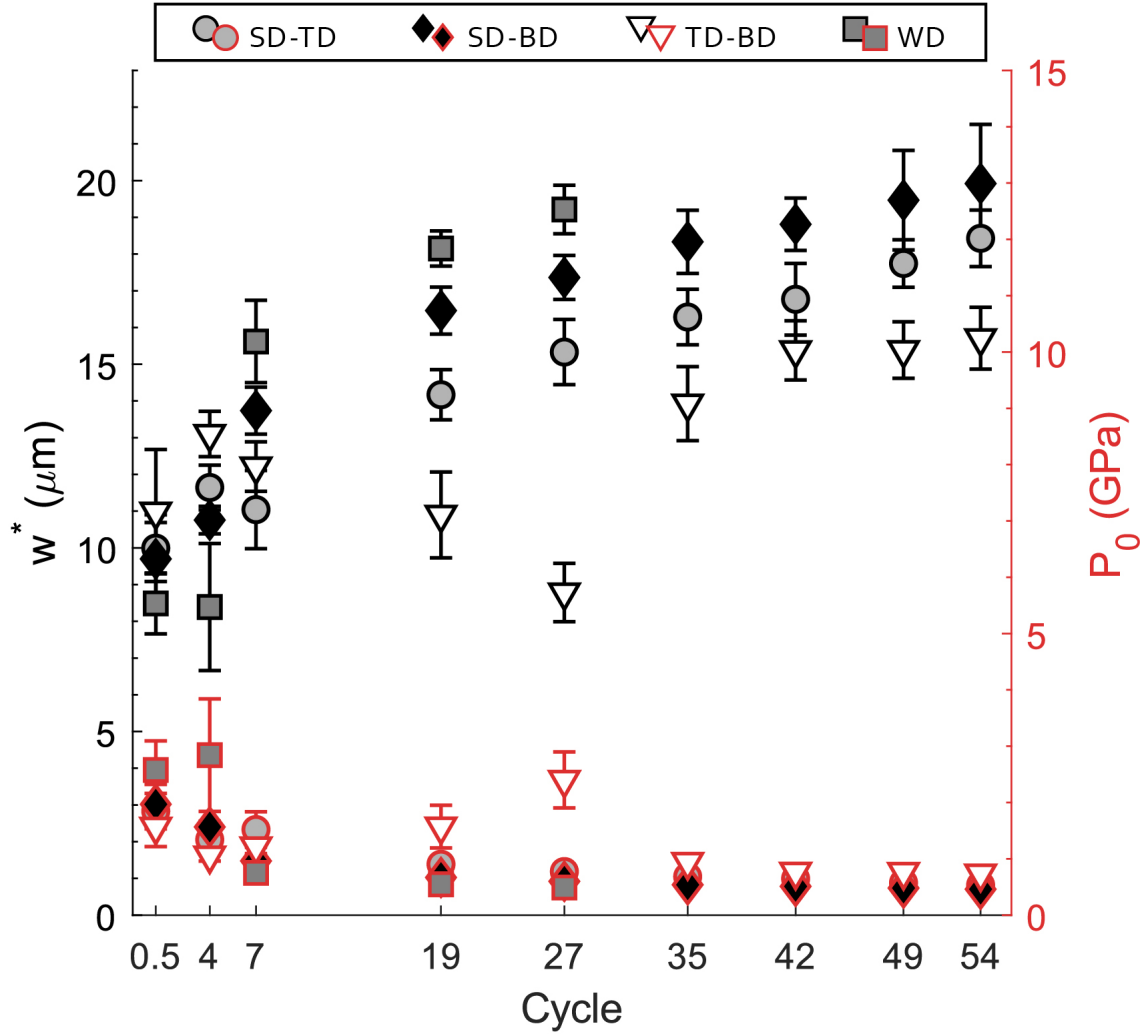


Figure 5.5: The minimum conformal scratch width (W^*) and the corresponding applied contact pressure (P_o) per cycle for each specimen [198].

The qualitative assessment on the other hand revealed a mixture of common and distinct features.

One common feature was the presence of crescent-like ridges along the trace of the scar Figure 5.6a. These ridges were mostly found during the initial cycles. Furthermore, their sides were surrounded by traces of slip lines. Another commonly observed feature was the development of a second scar ensuing from the scratcher skidding off track (Figure 5.6b). Although this event was unpredictable, it was mostly prominent in TD-BD and less so in SD-BD. In all instances of skidding however, the scars eventually coalesced into a single one as cycles progressed.

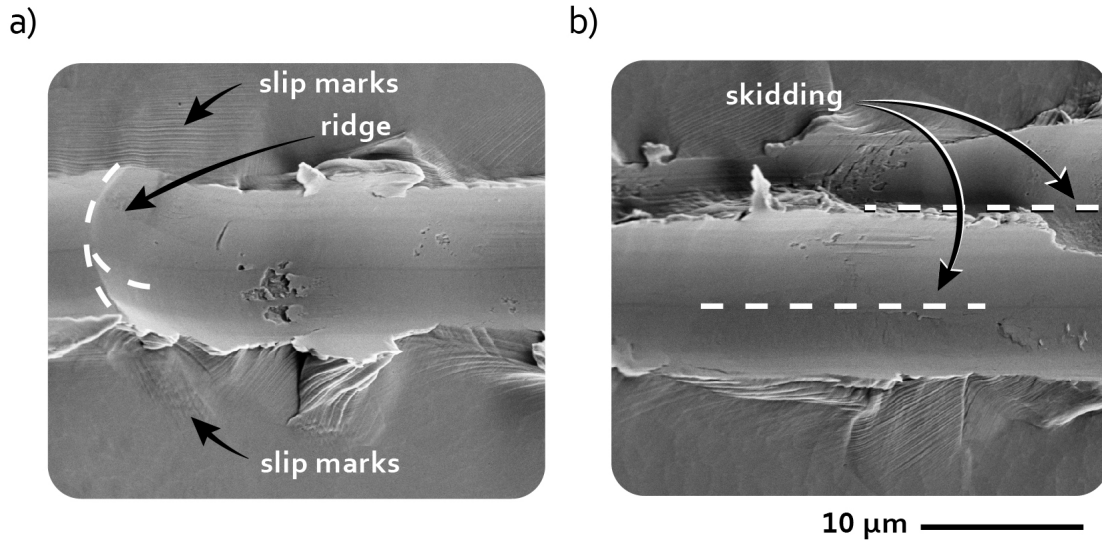


Figure 5.6: Scanning electron micrographs showing common features observed in specimens such as (a) formation of crescent-like ridges and (b) skidding of the scratcher, which leads to more than one track [198].

On the other hand, the microstructural damage build-up of the specimens was idiosyncratic. A comparison of the slip bands adjacent to the scar is depicted in Figure 5.7. It can be seen that the bands span various lengths and align at various angles. The magnitude of these slip bands increased with increased number of cycles. In other cases the grains can be seen being lifted-up as they are being squeezed between the scar and adjacent grains (i.e., SD-TD on the same figure).

The development of slip bands and the grain detachment can be seen in Vid. 5.1 and Vid. 5.2, respectively. Vid. 5.1 shows a side-by-side comparison of all specimens, in which the slipping and debris generation varies from one to the other. On the other hand, Vid. 5.2 shows that the lateral stresses imposed by the scratcher cause the grain to partially detach off the surface. In addition, it shows conjugate slipping occurring along the periphery of the scar, whereas single slipping occurring away from the scar.

Another distinctive feature was the debris of the TD-BD specimen. Unlike other specimens, the debris from this specimen often took shape of continuous, thin sheets with serrated edges (Figure 5.8).

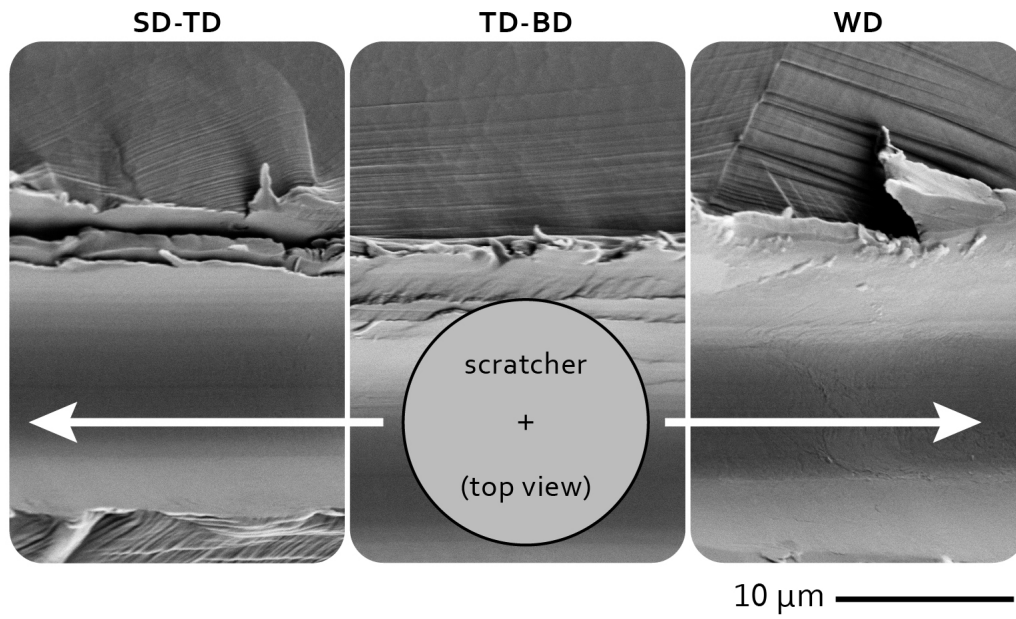
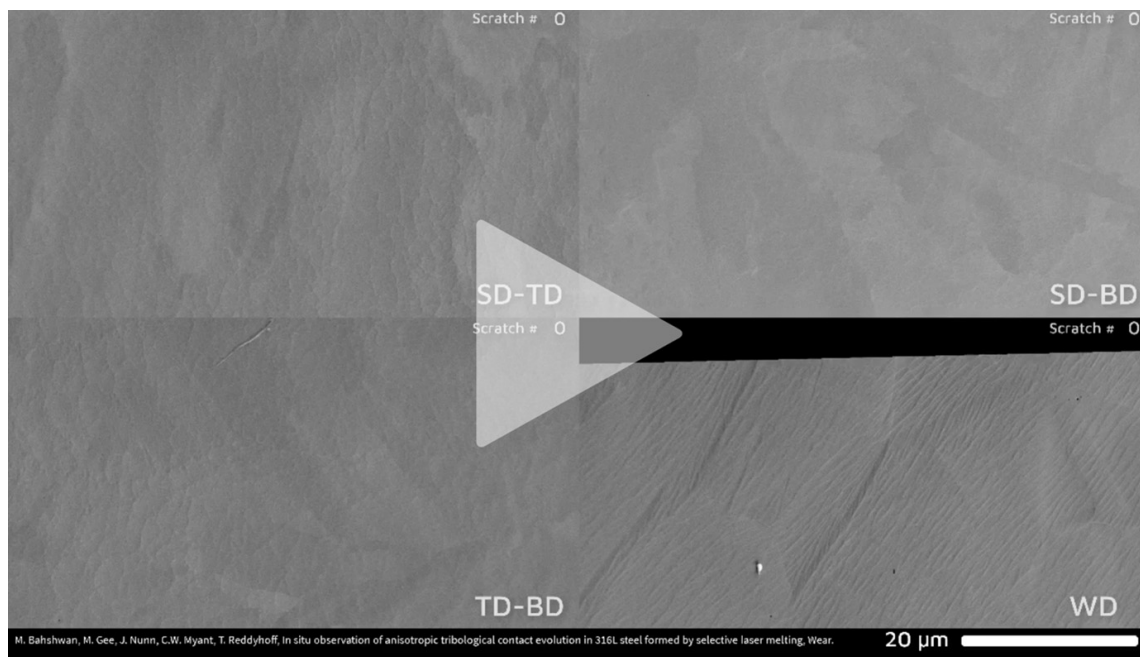
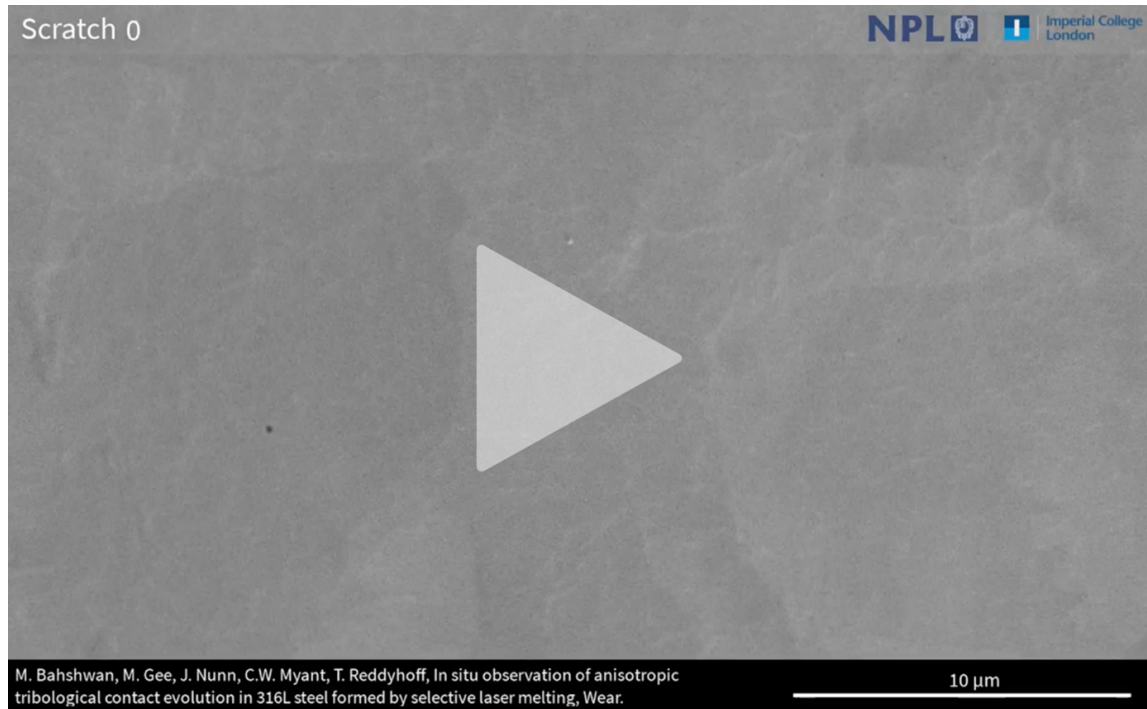


Figure 5.7: Scanning electron micrographs comparing the slip band formation in different specimens near the scratch area [198].



Video 5.1: A video which shows side-by-side comparison of wear evolution in all specimens [198]. (Note: the reader may refer to the following URL: <https://ars.els-cdn.com/content/image/1-s2.0-S0043164821005767-mmcl.mp4> or an adapted print version in Appx. 8).



Video 5.2: A video which shows the evolution of wear [198]. (Note: the reader may refer to the following URL: <https://ars.els-cdn.com/content/image/1-s2.0-S0043164821005767-mmc2.mp4>).

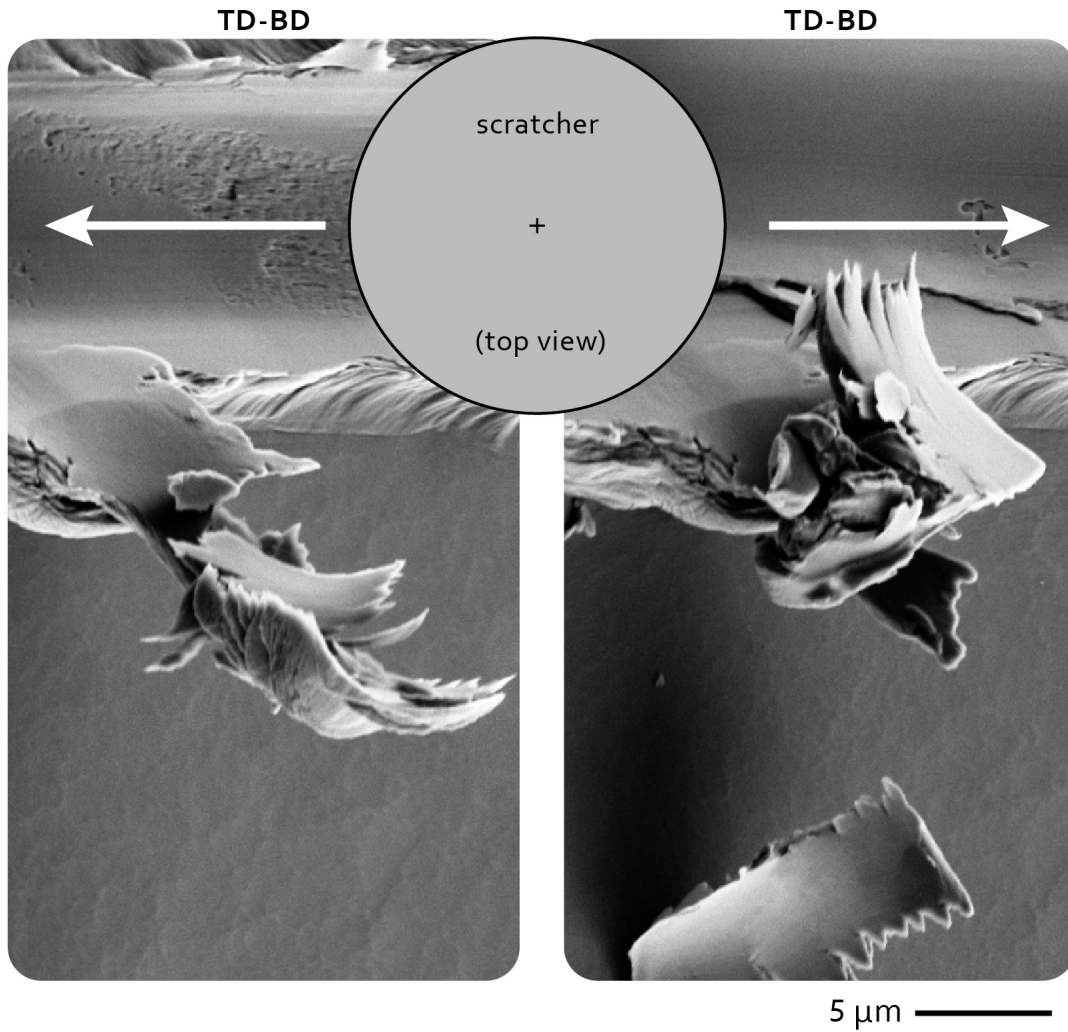


Figure 5.8: SE micrograph showing the continuous, sheet-like debris ejected by the TD-BD specimen [198].

5.3 Discussion

The resulting tribological behavior from single-asperity, micro-scratch tests varied between common and distinct features. There were instances when the SLM-manufactured specimens exhibited similar features to the conventionally manufactured specimens, and vice versa. Even among the three SLM-manufactured specimens themselves, the tribological properties exhibited distinct features which alludes to an anisotropic tribological behavior. The remainder of this section will correlate all the findings and discuss their implications in greater depth.

5.3.1 Microgeometric conformity and its role on the tribological behavior

As highlighted in Figure 5.1, the CoF values exhibited a conspicuous distinction between break-in and steady-state. Although the time spent in break-in varied across specimens' CoF, they all eventually reached steady-state. Break-in curves are known to be influenced by several factors such as contact geometry, material properties, interaction effects, environmental factors, and system parameters. For example, consider the following studies [199, 200] by Ruff and Blau, in which they used different set-up, speed, load, and materials compared with those in this project. They observed that CoF values increase as the cycle numbers increase, which is quite the opposite to what is shown in Figure 5.1. Having established that outcomes can vary drastically, it is crucial to develop insight into what factors influence such outcomes.

To understand why CoF increases or decreases with number of cycles, it is helpful not only to delineate the break-in and steady-state stages, but also to inspect the transition between them. The in-situ SEM set-up provides us the opportunity to connect visual evidence of the tribological contact with the measured CoF values. For example, we may use the captured SEM images per cycle (i.e., in Vid. 5.1) to attempt to explain the spikes in CoF along the stroke (i.e., in Figure 5.2a-d). Doing so for cycles which span both break-in and steady-state can be helpful in revealing any noteworthy differences which may explain the results.

Consider two specimens which exhibited completely different frictional behavior, namely SD-BD and TD-BD. For these, we will examine three cycles while noting their state of break-in or steady-state shown in Table 5.1. Combining the corresponding CoF measurements and the SEM micrographs shows that when the track is smooth, the CoF values are less jagged (compare the scratch tracks and CoF curves for SD-BD and TD-BD in Figure 5.9). This observation corresponded with the steady-state cycles in SD-BD (12, 18, and 34) and TD-BD (34 only). On the other hand, when the track was rough, the CoF values were jagged. Such case corresponded with the break-in cycles in TD-BD (12 and 18).

Table 5.1: The coefficient of friction stage (break-in or steady-state) for three closely examined cycles (12, 18, and 34) in TD-BD and SD-BD [198].

	Stage	
	TD-BD	SD-BD
Cycle 12	Break-in	Steady-state
Cycle 18	Break-in	Steady-state
Cycle 34	Steady-state	Steady-state

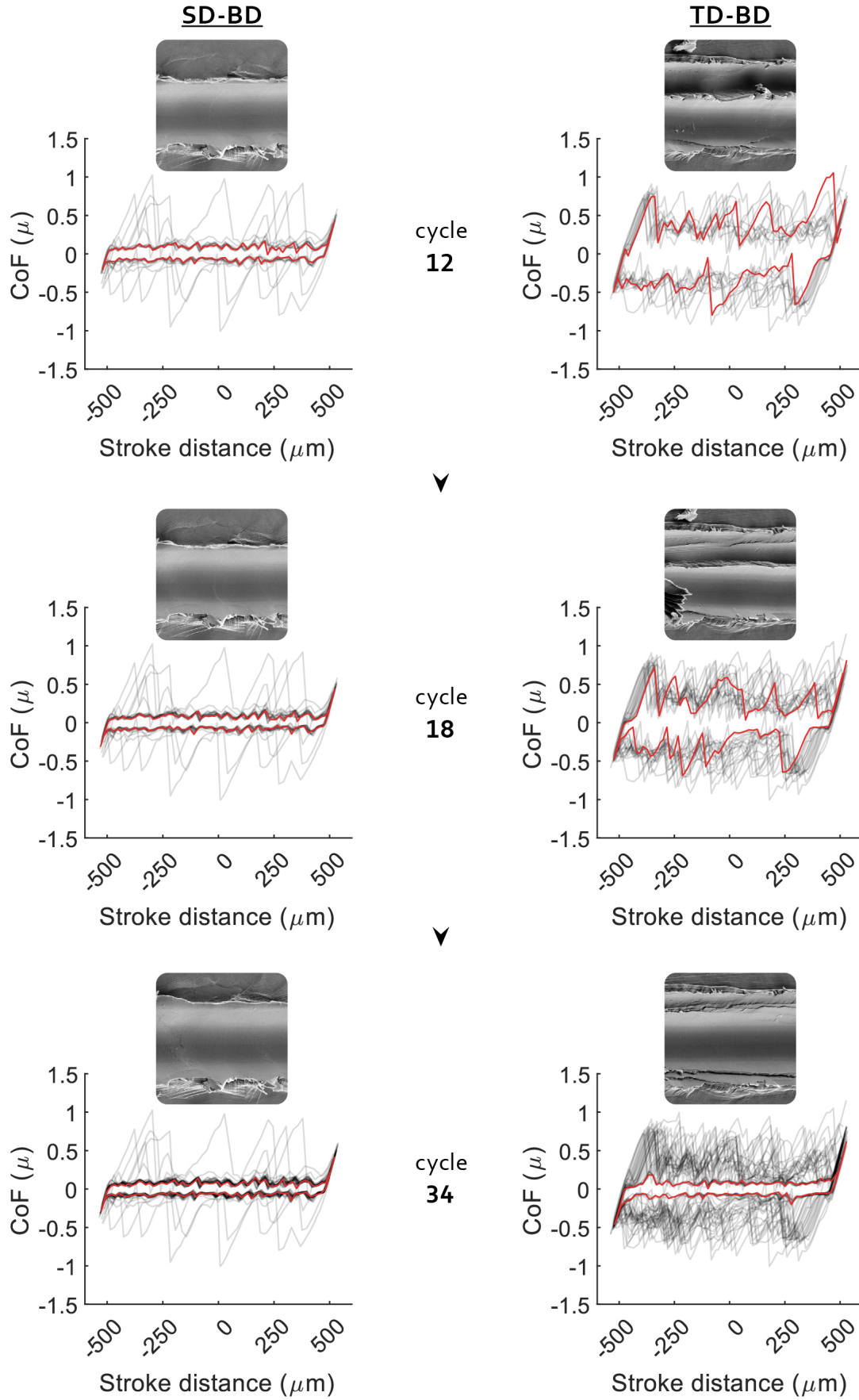


Figure 5.9: Comparing the non-averaged coefficient of friction profiles per stroke for three cycles (12, 18, and 34) while simultaneously showing the smoothness of the corresponding wear track for the SD-BD and TD-BD specimens [198].

These results suggest that a non-uniform physical interaction between the tip of the scratcher and the sliding surface is responsible for the spikes in frictional forces. More specifically, as the tip encounters ridges or valleys, rough sides that brush against the tip, or (semi-)loose debris along the stroke, frictional forces increase. Given that friction involves the dissipation of energy [201], one means for reducing friction is to reduce the energy being dissipated. A physical way of doing so is to increase the conformity of the wear track. This notion is partially captured in the contact pressures plot (Figure 5.5) in which the pressures are seen to decrease as conformity increases with cycles. On the other hand, friction can be increased by increasing the energy expended by the scratcher. An example of such is in early cycles where the onset wear is heterogenous (Appx. 7). In fact, the greatest energy expended is likely in the initial scratch, where the scratcher attempts to establish microgeometric conformity by subjecting the initially flat surface to significant strains. During this initial stroke, the orientations of the crystals are not aligned with the easy shear associated with the scratcher's motion and load. Thus, the crystals initially resist the plastic deformation imposed by the scratcher, which leaves a trail of a heterogenous scar. However, as conformity becomes increasingly established, the same crystals reorient themselves to achieve an easy shear that is nominally parallel to the scratcher's shear stresses (i.e., they undergo a localized texturing) [199, 202, 203].

In a hypothetical scenario where the crystals are randomly oriented, it may be assumed that the bulk crystallographic orientation is not parallel with the scratcher's easy shear. Rather, each randomly oriented single crystal has, likewise, a random misalignment angle from that easy shear. As such, individual crystals tend to resist the plastic deformation in proportion to their misalignment angle from that easy shear. Now let us consider the situation at hand, in which the SLM-manufactured specimens exhibited a Goss texture (see 4.2.1 for more details on texture). In this case, most of the break-in tribological response should stem from these like-oriented crystals, whereas less of the tribological response will stem from the other randomly oriented crystals. Therefore, when the scratcher's load and stroke are positioned differently with respect to this texture of the sample as seen Figure 5.10, an anisotropic break-in tribological response is expected. Nonetheless, from a practical perspective, the situation is quite complex. The actual number of crystals, crystallographic orientation, and boundaries will vary significantly even if a scratch was administered just adjacent to the original one. Furthermore, there may exist subsurface pores which are not readily visible via the SEM inspection. This unpredictability will in turn yield unpredictable tribological behavior. As such, we may observe ridges/valleys (Figure 5.6), deeper/shallower tracks, or wide/narrow ridges outside the track, etc. Sometimes the crystal/grain may rupture with – seemingly – no reorientation as depicted in Figure 5.4. However, this cannot be taken at face value, given that the ridges alongside the stroke are not smooth, which cannot be indexed via EBSD. Furthermore, any loose debris covering the peripherals of the track will also not be indexed via EBSD. Nonetheless, it can be stated with high confidence that the increase in number of cycles led to an increase in a less-energy-requiring microgeometric conformity, which ultimately reduced friction.

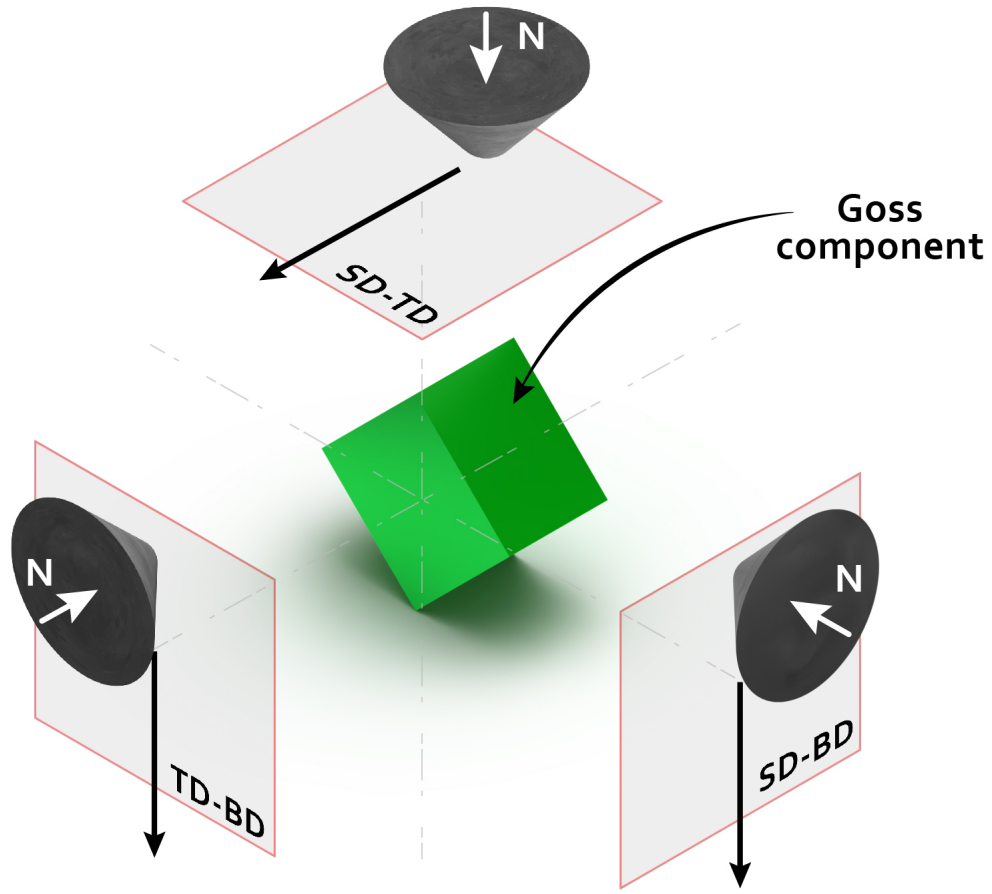


Figure 5.10: The scratcher's loading and sliding direction with respect to the Goss component in the SLM-manufactured specimens [198].

5.3.2 Dissecting the steady-state friction in conventional and SLM-manufactured specimens

When averaged per cycle, the steady-state CoF values for the SLM-manufactured specimens approximately converged at 0.08, whereas the conventionally manufactured specimen's CoF was approximately 0.07 (Figure 5.1b). Given that microgeometric conformity has been established at steady-state, the plastic deformation-induced strains are localized within the re-textured region. And because all the strains are bound to the re-textured region, the initial Goss texture no longer impacts the CoF [202] (note: this argument is only valid to single-phase materials). This notion is further supported by the lack of anisotropic CoF behavior among the SLM-manufactured specimens.

However, to explicate the subtle difference in the averaged steady-state CoF between the conventionally and the SLM-manufactured specimens (0.07 versus 0.08, respectively), a different approach needs to be considered. It is known that plastic deformation-induced slip involves the movement atoms as to accommodate for the large strains. However, before atoms can slip, they must break their existing cohesive bonds with the neighboring, motionless atoms and subsequently form new cohesive bonds with the next motionless atom. This process has been known to induce

microscale friction in sliding contacts [204]. Thus, if the cohesive bonds require more energy in order to be broken, then one should expect an uptick in frictional forces as a result. One microstructural constituent responsible for strengthening the cohesive bonds is a dislocation [204]. And the bonds' strength tend to grow proportionally to the number (i.e., density) of dislocations present.

Because the conventionally manufactured specimen has been well annealed compared to the as-built SLM-manufactured specimens, many of its strain-induced dislocations have been annihilated. On the other hand, the SLM process itself is known for inducing high-density of dislocations around the solidification cellular dendrites (see ECCI micrograph in 4.2.1). These dislocations in turn increase the cohesive bonds and thereby the frictional forces compared to the annealed, conventionally manufactured specimen. As a result, an uptick in the averaged steady-state CoF was noticed in the SLM-manufactured specimens.

5.3.3 The anisotropic microcutting behavior

As shown in Figure 5.8, the TD-BD specimen exhibited distinctive wear debris compared to the other specimens. Its wear behavior can be better appreciated in Vid. 5.1. The difference between this specimen and the other two SLM-manufactured ones, is that it possesses larger grains with low Taylor orientation factors [183] (see section 4.2.1). As such, this specimens' stiffness is lower than the other two SLM-manufactured specimens. Yet, its stiffness was greater than the conventionally manufactured specimen as measured by hardness tests (see section 4.2.1). If we assume that low hardness is responsible for this type of wear behavior, then the conventionally manufactured specimen would have exhibited a similar, or in fact even worse behavior. But this was not the case. Therefore, it unreasonable to assume that hardness is the sole contributor to such wear behavior.

Given the initial contact width of approximately 7 μm (Figure 5.3b) and the corresponding grain dimensions in each specimen (see 4.2.1), the scratcher's tip crosses fewer HAGBs in the case of TD-BD compared to other specimens (see Figure 5.11a,b below). This remark can be visually appreciated through Vid. 5.1, which shows long/continuous slip lines in TD-BD, indicating that scratches are occurring within a single grain. Therefore, it is logical that TD-BD's wear debris be quite distinctive.

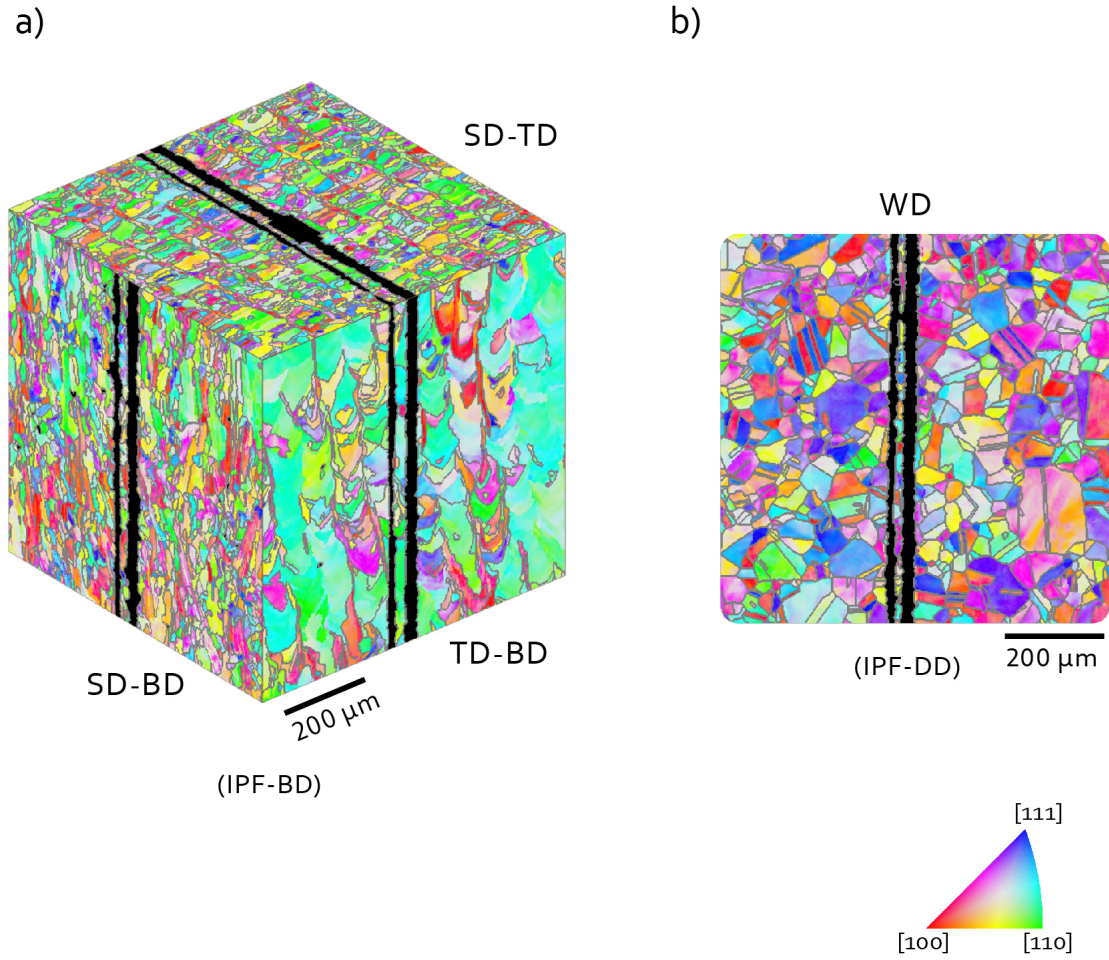


Figure 5.11: Visual comparison of the high-angle grain boundaries (HAGBs) in (a) SLM-manufactured specimens, and (b) conventionally-manufactured specimen [198].

Sheet-like debris similar to TD-BD's was documented in previous work and was linked with microcutting [205]. The following mechanism is proposed for the formation of such long, thin sheets with serrated edges [198]:

- (a) Initially, the moving (hard) scratcher initiates microcutting of the ductile specimen in the form of a smooth thin sheet (Figure 5.12a);
- (b) Continued movement increases the sheet's length to a critical point; once reached, two symmetrical cracks on the sides of the sheet appear near the scratcher (Figure 5.12b);
- (c) Continued movement increases the sheet's length further, and simultaneously the size of the cracks. Furthermore, two symmetrical tracks appear near the scratcher (Figure 5.12c). This step continues in the absence of abrupt change(s);
- (d) Finally, in the case of an abrupt change (in motion, speed, load, etc.), the debris detaches from the parent material in the form of long, thin sheets with serrated edges (Figure 5.12d).

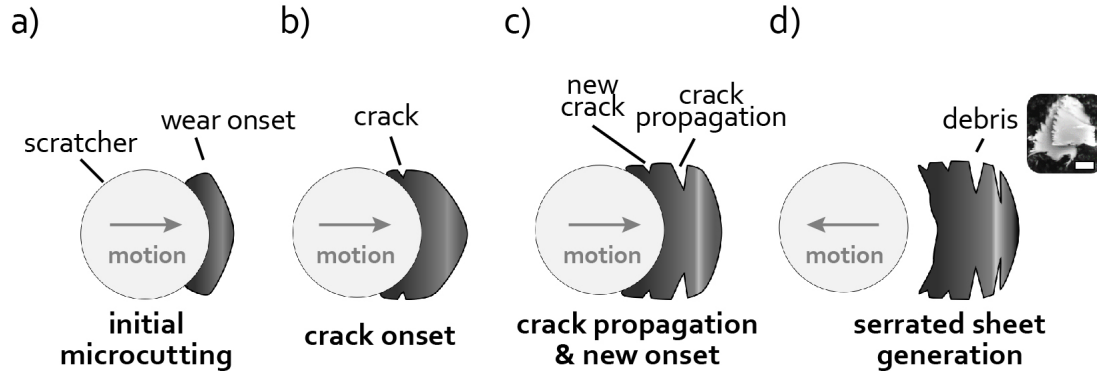


Figure 5.12: The proposed mechanism by which TD-BD wear debris develop. (scale bar on SEM micrograph is $4\text{ }\mu\text{m}$) [198]. a) Initially, the moving (hard) scratcher initiates microcutting of the ductile specimen in the form of a smooth thin sheet. b) Continued movement increases the sheet's length to a critical point; once reached, two symmetrical cracks on the sides of the sheet appear near the scratcher. c) Continued movement increases the sheet's length further, and simultaneously the size of the cracks. Furthermore, two symmetrical tracks appear near the scratcher. This step continues in the absence of abrupt change(s). d) Finally, in the case of an abrupt change (in motion, speed, load, etc.), the debris detaches from the parent material in the form of long, thin sheets with serrated edges .

5.4 Chapter conclusion

This study focused on revealing the anisotropic behavior of SLM-manufactured 316L as compared to conventionally manufactured 316L in sliding contact. To heighten the anisotropic response, single-asperity (diamond tip), micro-tribological experiment was conducted at a low load of 100 mN. Further, the experiment was conducted SEM in-situ as to allow for observing the contact evolution of each specimen. The main findings are summarized as follows:

- (i) No matter which specimen was tested, it was found that once microgeometric conformity has been established, friction would reach steady-state.
- (ii) Friction in SLM-manufactured 316L straddles between two states of anisotropy and isotropy, each of which is confined to steady-state and break-in, respectively.
- (iii) The steady-state CoF in SLM-manufactured 316L was slightly higher than the conventionally manufactured counterpart. This uptick was owed to the presence of high-density of dislocations (surrounding the cellular dendrites) which increase the cohesive bonds, and as a result, the energy required to perform plastic deformation.
- (iv) Wear debris was anisotropic, especially for grains with low Taylor factor. In these grains, the debris was characterized as thin, long sheets with serrated edges.

These findings show that the tribological response of SLM-manufactured 316L is far from equal to that which is conventionally manufactured because of the differences in the underlying microstructures. Furthermore, the findings show that even for SLM-manufactured 316L, the microstructure needs to be carefully analyzed as it plays a significant role in the anisotropic tribological behavior of the material.

Chapter 6

Multigrade tribosurface via microstructure control

The content of this chapter was adapted as a publication manuscript at the time of thesis submission.

6.1 Chapter introduction

In the previous chapter, we examined how the different microstructures of additively and conventionally manufactured steel impacted the tribological properties. Furthermore, we visually appreciated the effect of anisotropy on the wear and frictional response in additively manufactured steel. In relation to the latter, the results revealed that the Taylor orientation factor (i.e., the crystal orientation-induced stiffness) was correlated with the wear debris and the measured value of friction. Yet, it is still not understood what precisely the relationship between the Taylor factor and the tribological properties is. Considering SLM's ability for inducing deliberately designed microstructures[101], understanding the impact of Taylor factor on the tribological properties would be indispensable in guiding the design of microstructures towards the desired tribological response. Likewise, it is crucial to understand whether the strength of crystallographic texture plays any role in those tribological properties.

We highlighted previously that it is almost impossible to compare two independent pieces of literature due to the nuanced differences in experimental parameters and microstructures induced by the SLM process. In particular, we discussed (in section 2.4.1 of the thesis) contradictory conclusions about the tribological 'superiority' of additively manufactured 316L compared to its conventionally manufactured counterpart. To resolve this uncertainty, the experimental process needs to be much simplified. Rather than conducting the tribological test on two separate SLM specimens manufactured with different parameters (or on different planes of the same specimen), here we directly compare the tribological response on a single specimen comprising multigrade microstructures achieved via microstructure control. This method serves to show the actual tribological transition across the multiple grades of the microstructure. We focus much heavily on the impact of crystallographic orientation stiffness and texture strength on the wear and friction. The results in this chapter form the basis for designing multigrade tribosurfaces via SLM.

(Note: experimental setup, parameters, and calculations pertaining to this study can be found in 3.4).

6.2 Results and discussion

6.2.1 Characterization of the tribosurfaces

Three different types of multigrade tribosurfaces were produced, each comprising a different configuration of crystallographic texture. Figure 6.1a-c shows IPF maps of those three tribosurfaces. The IPF reference axis was parallel to the axis normal to the tribosurface (i.e., IPF-z). The first tribosurface (Figure 6.1a) comprised a repeating set of three textures along the TD. Each block of the three textures was produced by rotating the SD about the BD. Each block was approximately 1-mm thick. This technique was based upon the findings of Sofinowski et al.'s layer-wise engineering of grain orientation (LEGO) [101]. The second tribosurface (Figure 6.1b) comprised a dual-texture configuration along the BD. The two textures evenly divided the entire tribosurface. The third tribosurface (Figure 6.1c) comprised a repeating set of three textures along the BD. Each texture block in this tribosurface was approximately 1-mm thick. As seen in Figure 6.1a-c, all specimens exhibited high-aspect-ratio grains whose major axis was parallel to the BD.

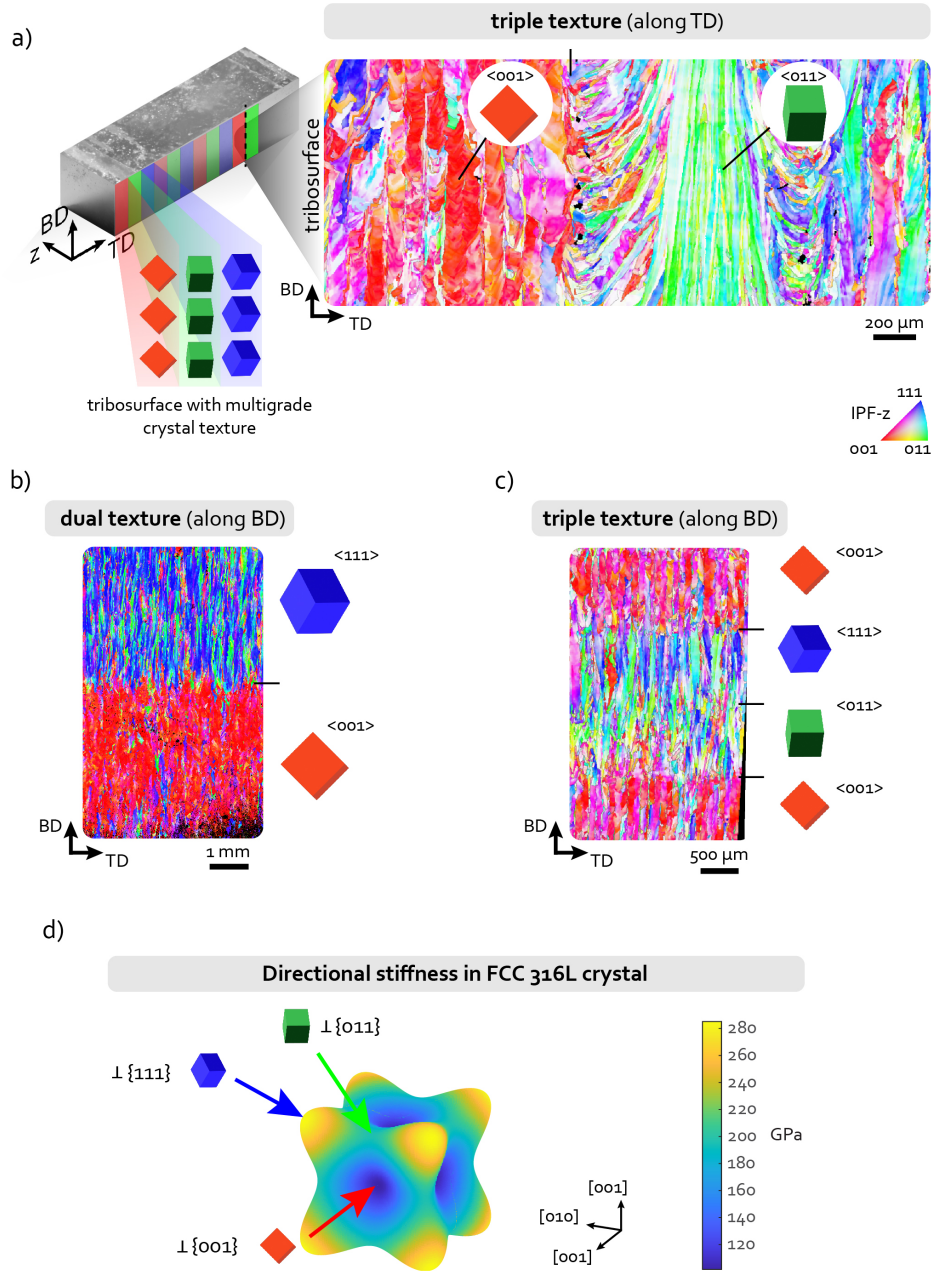


Figure 6.1: 316L stainless steel multigrade tribosurfaces comprising various textures. a-c. Inverse-pole-figure maps along the z-axis (into screen). a. Tri-grade tribosurface comprising $\langle 001 \rangle$ (red), $\langle 011 \rangle$ (green), and $\langle 111 \rangle$ (blue) textures along the transverse direction (TD). Clusters of strong textures as well as randomly oriented crystals can be seen at different sites of the tribosurface. b. Bi-grade tribosurface comprising $\langle 001 \rangle$ and $\langle 111 \rangle$ textures along the build direction (BD). c. Tri-grade tribosurface comprising $\langle 001 \rangle$, $\langle 011 \rangle$, and $\langle 111 \rangle$ along the BD. d. The directional stiffness for a single FCC 316L crystal along the $\{001\}$ (101.64 GPa), $\{011\}$ (196.45 GPa), and $\{111\}$ (285.11 GPa) planes.

The role of texture strength as a factor influencing the tribological properties would be extremely useful for metal additive manufacturing processes. The texture strength varied between the three multigrade tribosurfaces. The texture strength was clustered in certain regions in the case of the first tribosurface (Figure 6.1a). Contour pole figure (6.2) shows the strength of texture in the highlighted areas on Figure 6.1a marked as $\langle 001 \rangle$, $\langle 011 \rangle$, and ‘?’ . The color bar shows that the texture strength ranged between 20-25 for the $\langle 001 \rangle$ and $\langle 011 \rangle$ areas. On the other hand, the texture of the second tribosurface (6.1b) was consistent throughout the entire sample. The strengths of texture for the $\langle 001 \rangle$ and $\langle 111 \rangle$ textures are shown in the contour pole figure (6.3). Noteworthy, the transition of texture from $\langle 001 \rangle$ to $\langle 111 \rangle$ occurred approximately after 0.6 mm of when the SD was rotated (Figure 6.5). Such lag occurs because it is much efficient for crystals to continue growing along their original direction (i.e., epitaxially) than for crystals to nucleate along a new direction (i.e., the new $\langle 111 \rangle$ in this case) [101]. Finally, the third tribosurface (Figure 6.1c) exhibited the weakest textures of all three (see Figure 6.4). With exception of the $\langle 001 \rangle$ blocks, the other blocks only showed hints of the intended $\langle 011 \rangle$ and $\langle 111 \rangle$ textures.

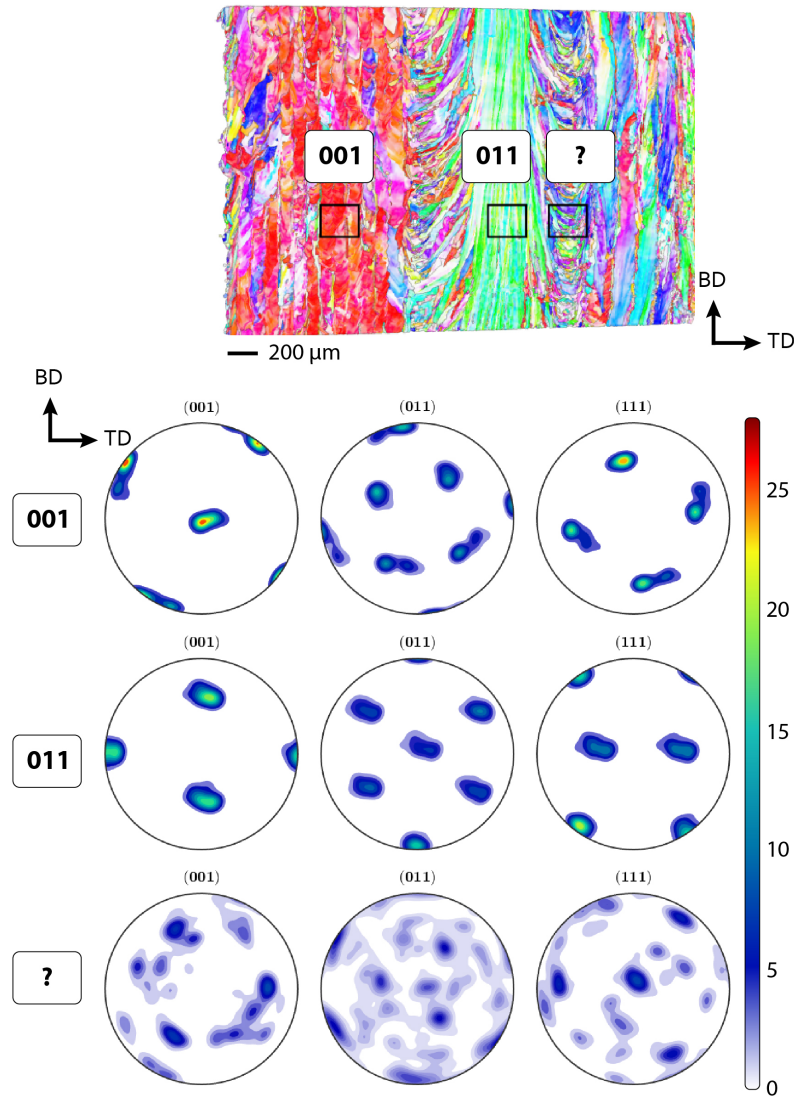


Figure 6.2: Texture strength of a tri-grade tribosurface with texture blocks spanning the transverse direction (TD) axis. A comparison of three sites, $\langle 001 \rangle$, $\langle 011 \rangle$, and $\langle ? \rangle$ is shown via the contour pole figures below the orientation map. It can be seen that the $\langle 001 \rangle$ and $\langle 011 \rangle$ texture strengths reaches approximately 25 and >20, respectively. On the other hand, the $\langle ? \rangle$ region barely reach below 10.

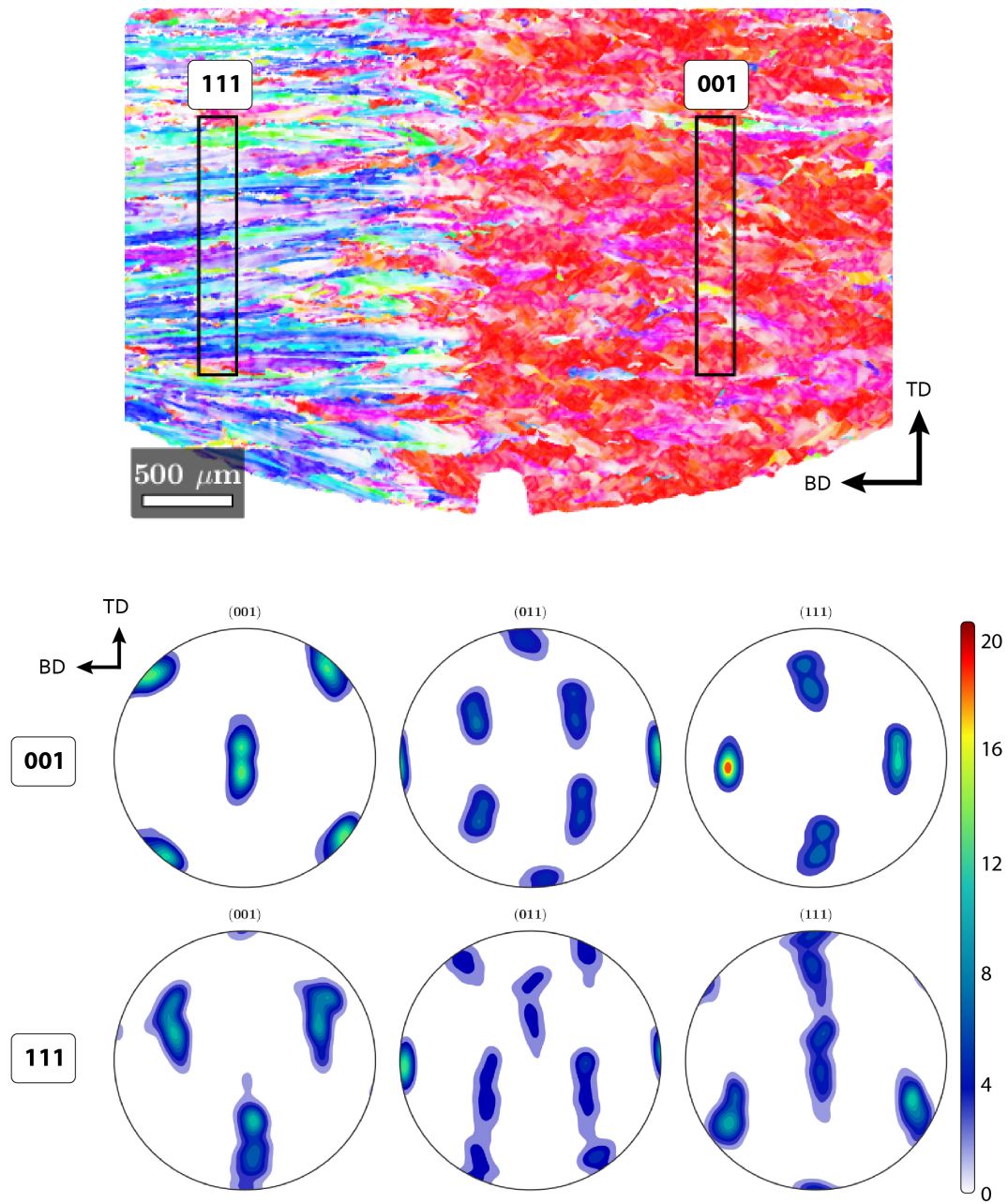


Figure 6.3: Texture strength of bi-grade tribosurface with equally split $\langle 001 \rangle$ and $\langle 111 \rangle$ -textured regions. The texture strength of the $\langle 001 \rangle$ region reaches approximately 20. The texture strength of the $\langle 111 \rangle$ region reaches approximately 14.

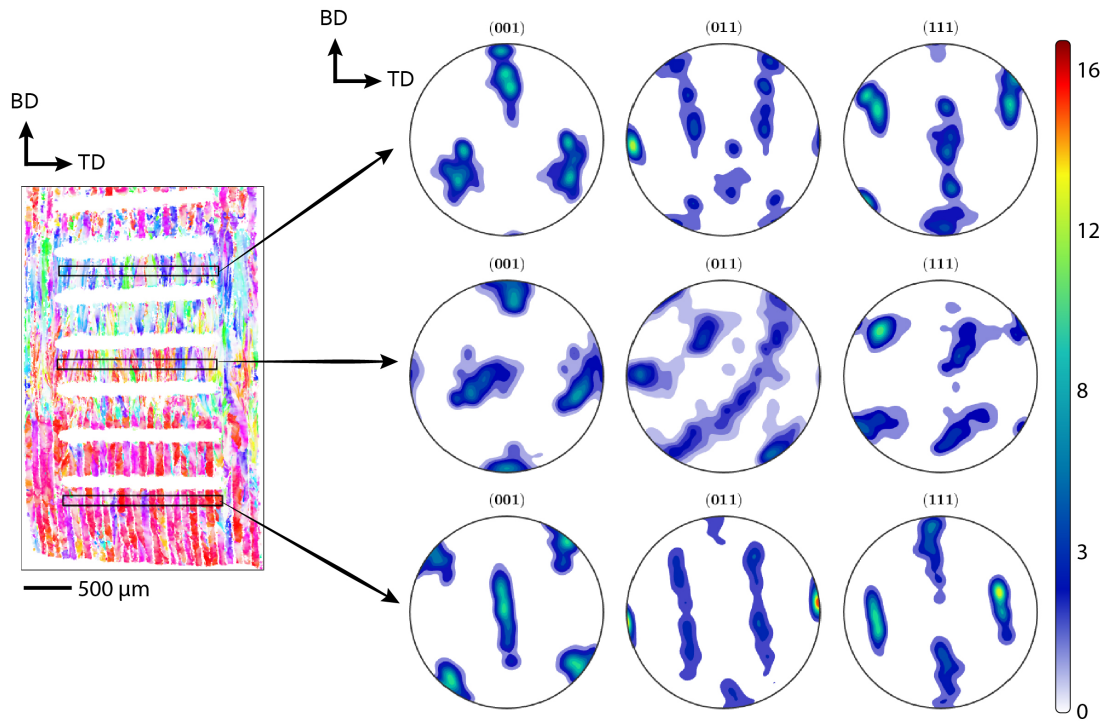


Figure 6.4: Texture strength of tri-grade tribosurface with repeating texture blocks spanning the build direction (BD) axis. The three boxes from bottom-to-top correspond to the $\langle 001 \rangle$, $\langle 011 \rangle$, and $\langle 111 \rangle$ texture blocks respectively. It can be seen that the strongest texture (approximately 16) was reserved for the $\langle 001 \rangle$ block. By comparison, both the $\langle 011 \rangle$ and $\langle 111 \rangle$ exhibited relatively weaker textures of approximately 11 and 13, respectively.

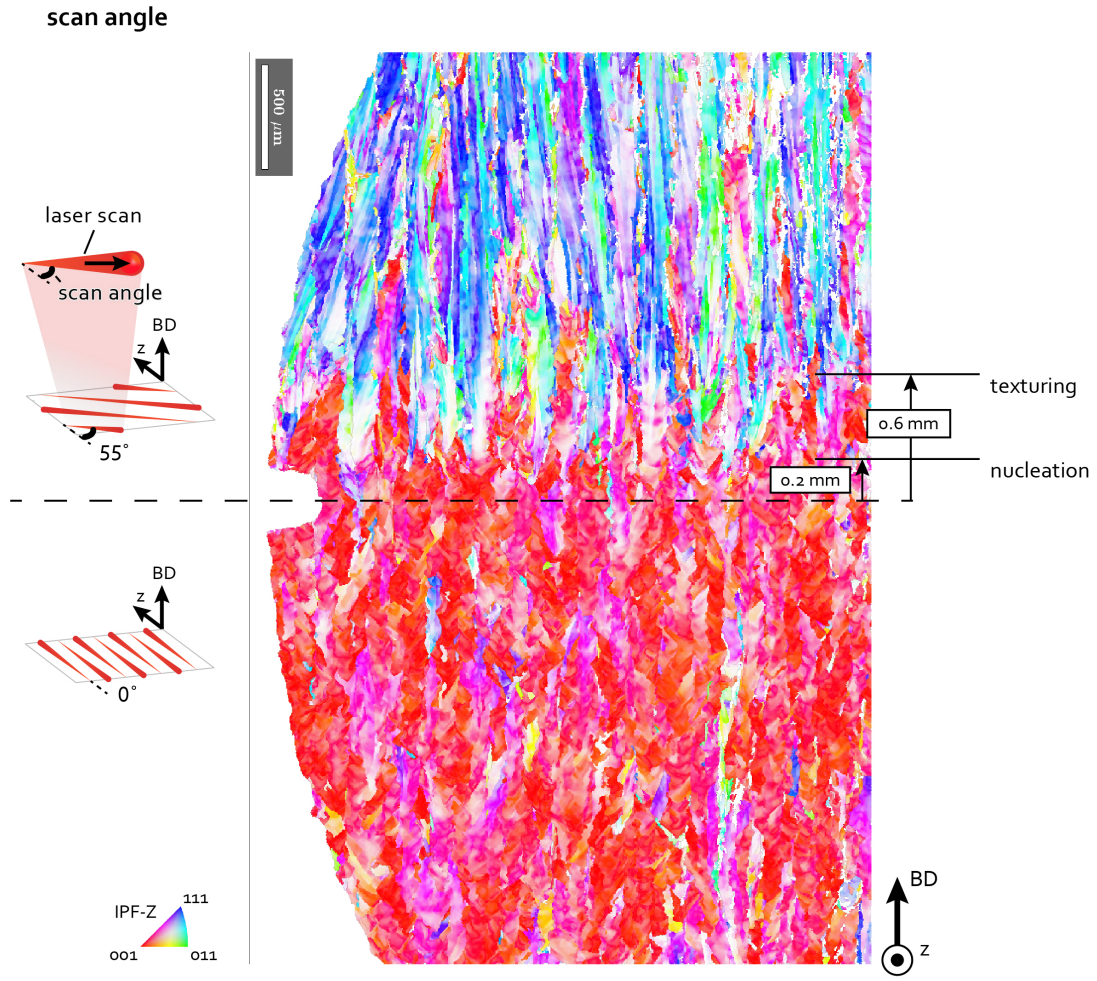


Figure 6.5: A delay in the $\langle 001 \rangle$ to $\langle 111 \rangle$ transition occurs when the scan direction (SD) angle is changed (denoted by the horizontal dashed line). The nucleation of new $\langle 111 \rangle$ grains occurred approximately after 0.2 mm of when the SD angle was changed. The texturing of $\langle 111 \rangle$ started approximately 0.6 mm of when the SD angle was changed.

The component relating the orientation of crystals to the strength of the material is incorporated in k'' discussed previously in Chapter 2 (Eq. 2.5):

$$k'' = M\alpha\mu b,$$

where M is the Taylor orientation factor, α is the constant accounting the interactions of dislocations in different slip systems, b is the Burgers' vector magnitude, μ is the shear modulus. More fully, the materials strength due to dislocations (Eq. 2.2c) is written as:

$$\sigma_y = \sigma_o + M\alpha\mu b\sqrt{\rho},$$

where σ_o denotes the strength in absence of dislocations, and ρ is the dislocation density. M is also viewed as the factor relating the yield strength to the critically resolved shear stress required for initiating slip [23]. M typically varies between 2.499 and 3.674 for FCC crystals [206]. These

two extrema correspond with the $\langle 001 \rangle$ and $\langle 111 \rangle$, respectively. Practically speaking, these values alter the elastic modulus of austenitic 316L between 101.64 GPa and 285.11 GPa, respectively (Figure 6.1d). However, because the produced tribosurfaces are not ideal single crystals, the change in actual elastic modulus due to changes in M lie somewhere in between these two extrema. The stronger the texture, the more the material behaves as a single crystal, in turn, the more pronounced the effect M has on the actual elastic modulus.

ρ in as-built SLM 316L typically is in the range of $10^{14}/\text{mm}^2$, which is close to that of heavily cold-rolled metals ($10^{14} - 10^{15}/\text{mm}^2$) [23, 207]. A comparison between as-built SLM 316L and annealed SLM 316L is shown in the ECCI micrograph in Figure 6.6. The dislocations in SLM components typically encircle the solidification cellular dendrites [3, 208]. When the cellular dendrites are observed cross-sectionally, the dislocations appear as an array of connected honeycomb-like structures. Whereas when the cellular dendrites are observed longitudinally from the side, the dislocations appear as an array of straight lines. The solidification dendrites grow along a $\langle 001 \rangle$ direction which aligns closely with the thermal gradient. As such, the dislocation arrangement is a function of the underlying crystallographic orientation. The diameter of the cellular dendrites in this study was approximately 750 nm. We have seen in Chapter 5 that these dislocations contribute to a rise in the coefficient of friction as they impede the plastic flow [198]. In this study, all SLM parameters remained constant except for the angle of rotation of the SD. In effect, the maximum heat flux (with which the other $\langle 001 \rangle$ directions align) rotates accordingly. Therefore, only the arrangement of dislocations is modified but not its magnitude. By contrast, if we were to change the scanning strategy across samples (e.g., using a chess-board scanning strategy), the cooling rate would change and subsequently the quantity of dislocations present.

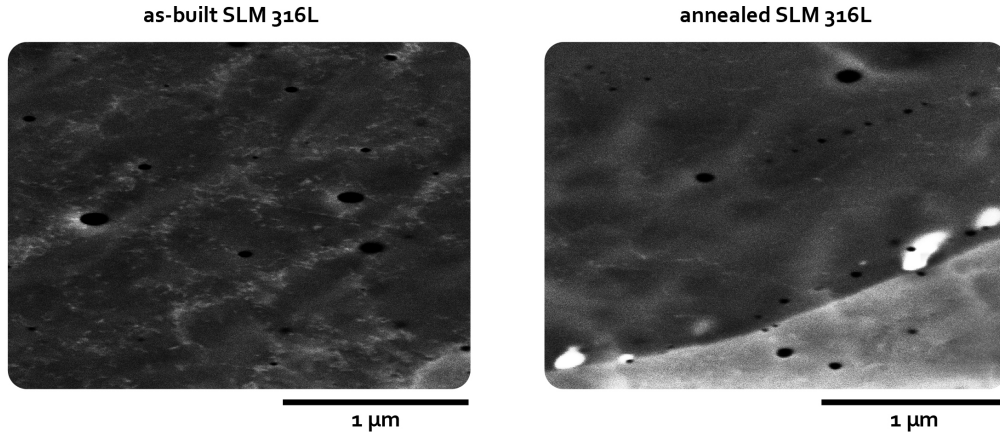


Figure 6.6: Electron channelling contrast imaging (ECCI) of as-built SLM 316L (left) versus annealed SLM 316L (right)

6.2.2 Impact of crystal orientation on friction

The effect of M on the coefficient of friction was investigated through multiple tribological tests. The first test was a micro-scratch test performed on the dual-texture tribosurface comprising $\langle 001 \rangle$

and $\langle 111 \rangle$ textures. The CoF was recorded as a 5- μm -radius diamond indenter scratched the surface under a load and speed of 10 mN and 50 $\mu\text{m/s}$, respectively. The scratch started in the $\langle 001 \rangle$ region and ended in the $\langle 111 \rangle$ region (Figure 6.7a). The CoF started at 0.2 and continued to decline with the movement of the indenter. However, a distinct transition in the CoF can be observed that separates the $\langle 001 \rangle$ from the $\langle 111 \rangle$ region. Specifically, it can be seen that the slope was steeper in the $\langle 001 \rangle$ region than in the $\langle 111 \rangle$ region. Generally, the average CoF was greater in the $\langle 001 \rangle$ region than in the $\langle 111 \rangle$ region. Figure 6.7b,c show ECCI micrographs highlighting the different arrangement of dislocations in the $\langle 001 \rangle$ and $\langle 111 \rangle$ regions, respectively. The dislocations surrounding the cellular dendrites (bright lines) in the $\langle 001 \rangle$ region were arranged longitudinally at an angle of approximately 45° with the scratch direction. These lines are parallel with a set of $\langle 001 \rangle$ which align with the thermal gradient. On the other hand, the dislocation arrangement in the $\langle 111 \rangle$ region appeared as a slightly tilted, honeycomb-like structures. Similarly, these honeycomb-like dislocation structures indicate that the $\langle 001 \rangle$ growth direction of the cellular dendrites is roughly parallel to the screen. More comprehensively, the geometrically necessary dislocations (GND) map in Figure 6.8 shows the effect of the $\langle 001 \rangle$ and $\langle 111 \rangle$ textures on the dislocation arrangement on a global level. The GND map shows that the rotation of the dislocation volume arises as a by-product of rotating the crystal orientation. However, because the cooling rate is not drastically changed between the $\langle 001 \rangle$ and $\langle 111 \rangle$ textures, it is reasonable to assume that the three-dimensional quantity of dislocations is similar between the two. As EBSD only provides two-dimensional information about GND, a precise three-dimensional quantification of dislocations is not possible. Respectively, the slip lines inside and outside the scratch exhibit a different behavior in each of the regions (Figure 6.7d,e). Specifically, the $\langle 001 \rangle$ slip traces appear curved and compact, whereas the $\langle 111 \rangle$ slip traces appear straight and spread apart.

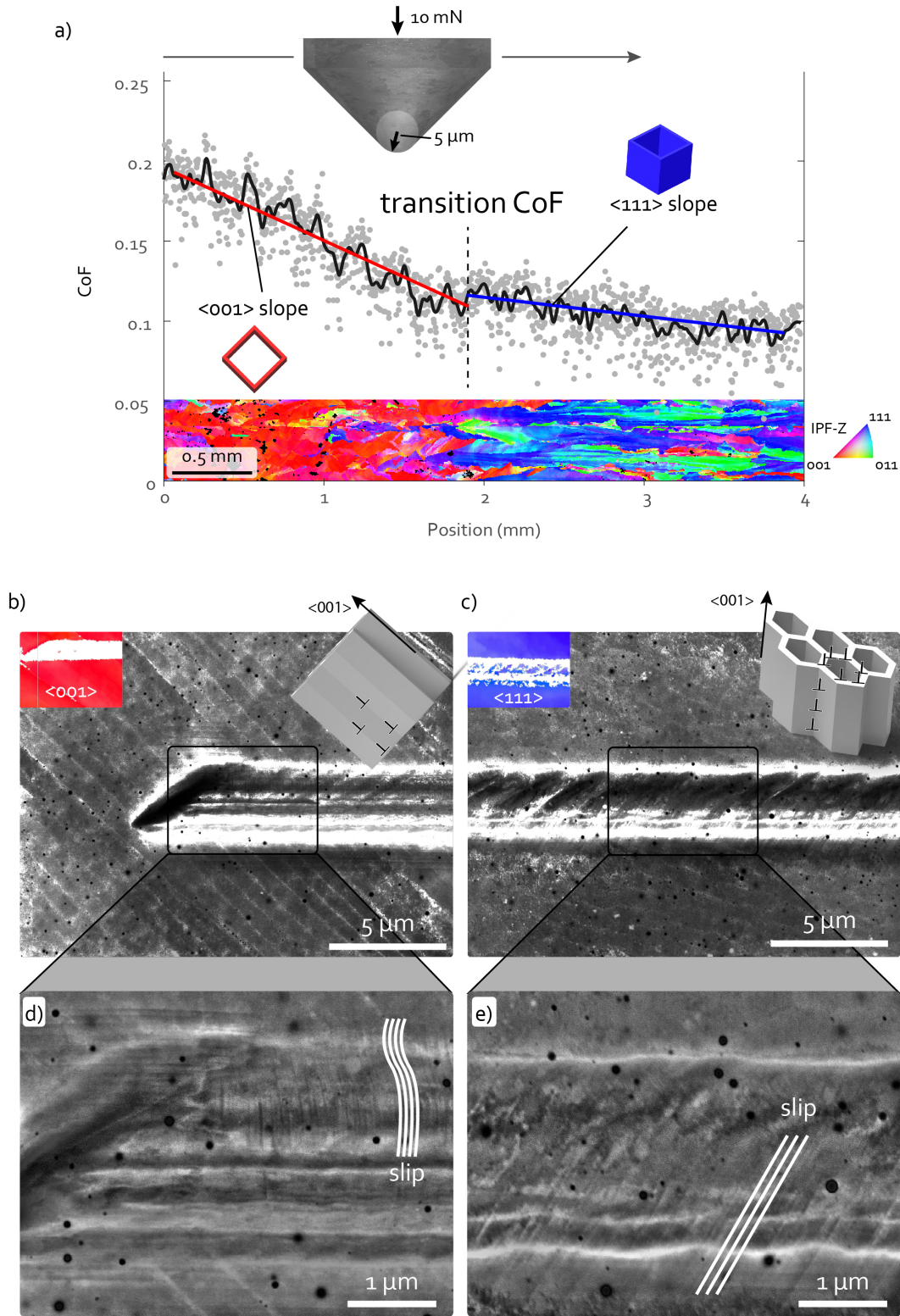


Figure 6.7: Friction behavior of bi-grade tribosurface comprising $\langle 001 \rangle$ and $\langle 111 \rangle$ textures. a. The coefficient of friction (CoF) exhibits two distinct behaviors in the $\langle 001 \rangle$ and $\langle 111 \rangle$ textures. In particular, a steeper slope is seen in the $\langle 001 \rangle$ than in the $\langle 111 \rangle$. The average CoF was greater in the $\langle 001 \rangle$ region than in the $\langle 111 \rangle$. b,c. The $\langle 001 \rangle$ cellular dendrites' growth along the thermal gradient yields a different dislocation arrangement (bright features) for the $\langle 001 \rangle$ and $\langle 111 \rangle$ surfaces. b. Longitudinal arrangement of the cellular dendrites in the $\langle 001 \rangle$ region. e. Cross-sectional arrangement of the cellular dendrites in the $\langle 111 \rangle$ region, showing the honeycomb-like dislocation structure. d,e. Slipping behavior in the $\langle 001 \rangle$ and $\langle 111 \rangle$ regions. d. $\langle 001 \rangle$ slip is compact and curved, whereas e. $\langle 111 \rangle$ slip is loose and straight.

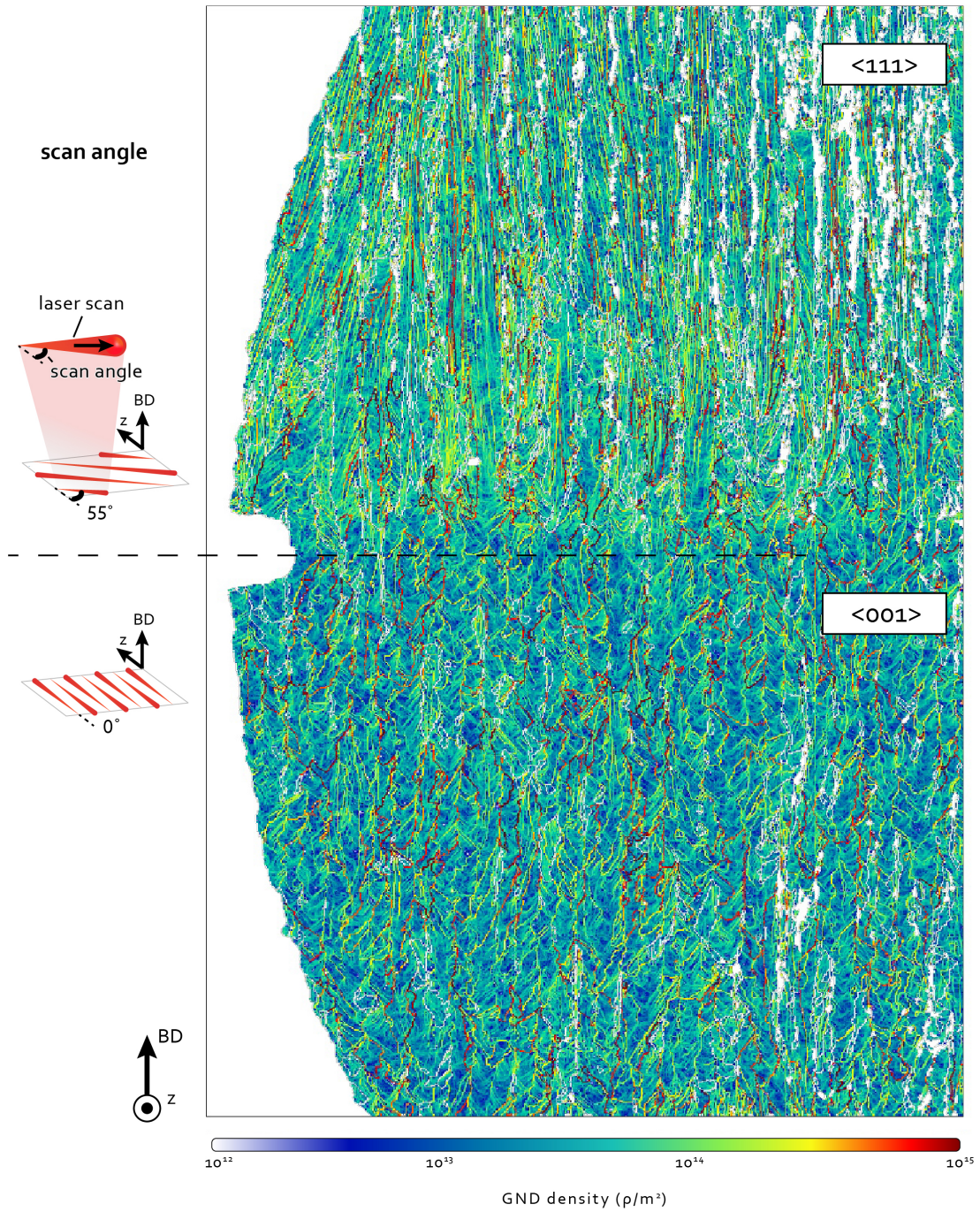


Figure 6.8: Effect of SD rotation on dislocation (GND) arrangement

A macro-tribology test was performed on the same tribosurface under a normal load three-and-a-half orders of magnitude (i.e., 15 N). This experiment was of particular interest as it provides insight as to whether anisotropy due to crystallographic texture continues to play a role in the CoF at higher normal loads. Figure 6.9 shows the CoF along the scratch's path starting from the $\langle 001 \rangle$ region and ending at the $\langle 111 \rangle$ region. It can be seen that the CoF here also exhibited two distinct behaviors in each of the regions. The average CoF was higher in the $\langle 001 \rangle$ region (0.31) than in

the $\langle 111 \rangle$ region (0.25). Interestingly, the CoF can be seen declining sharply where the indenter position (marked by $\otimes$) engages with the onset of the $\langle 111 \rangle$ texture on the tribosurface (see close-up EBSD map in bottom-right corner of Figure 6.9). Once the dominant orientation became $\langle 111 \rangle$, the CoF stabilized at lower values than that observed in the $\langle 001 \rangle$ region.

Collectively, the micro- and macro-tribology results suggest that anisotropy due to crystallographic texture remains in effect over a load ranging from 0.01 up to 15 N. This anisotropic CoF effect arises due to a change in directional stiffness (M) which is induced by varying the SD angle.

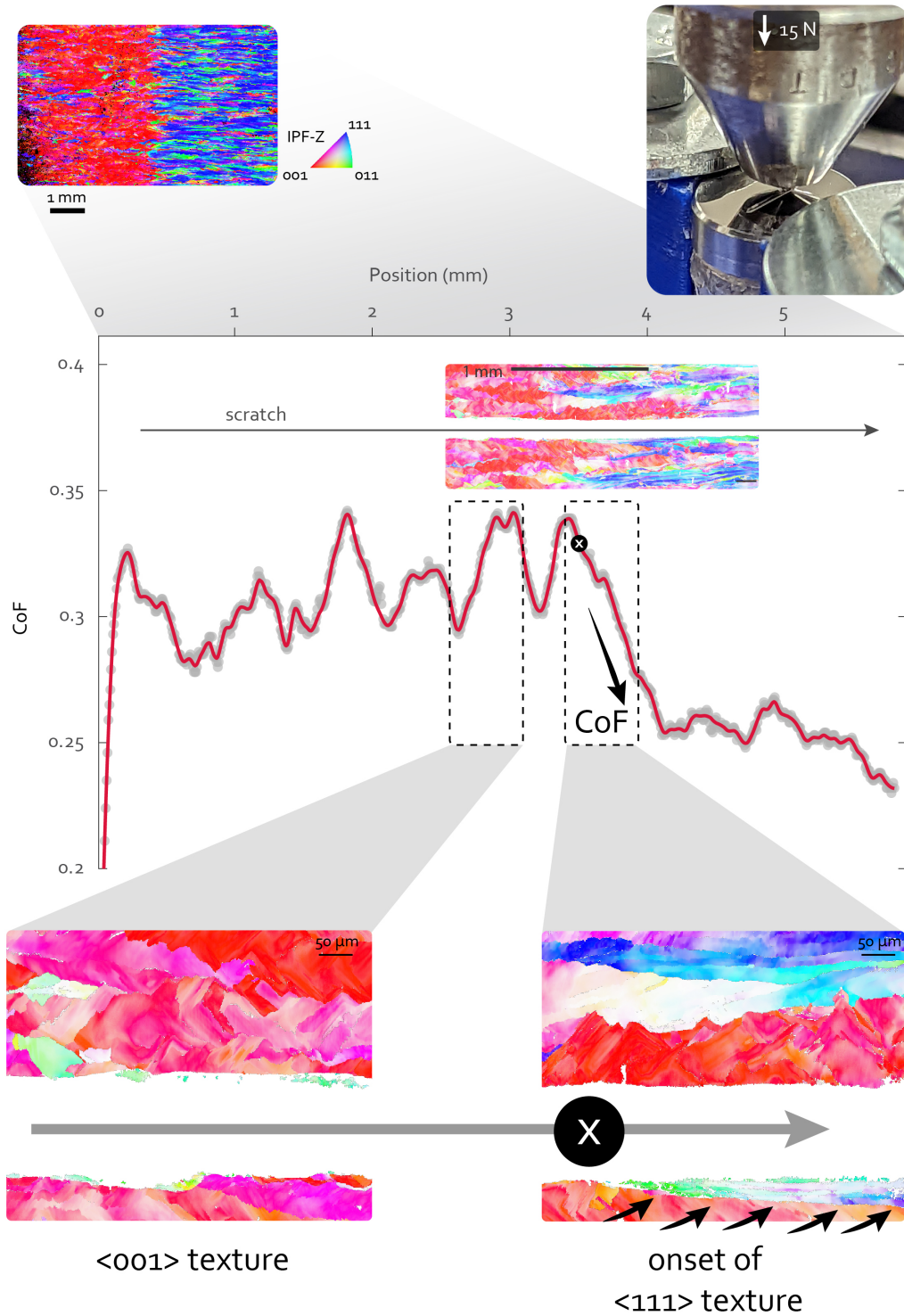


Figure 6.9: Onset of CoF transition in the bi-grade $\langle 001 \rangle / \langle 111 \rangle$ tribosurface. A sharp drop in the CoF occurs immediately with the onset of the $\langle 111 \rangle$ texture (see bottom-right panel). The average CoF in the $\langle 001 \rangle$ and $\langle 111 \rangle$ regions are 0.31 and 0.25, respectively.

The effect of M on the CoF was validated via an additional macro-tribology experiment involving a tribosurface with three textures (Figure 6.1c). Twenty-two 15-N single scratches were administered inside the different $\langle 001 \rangle$, $\langle 011 \rangle$, and $\langle 111 \rangle$ texture blocks as seen in Figure 6.10a. The resultant M -map is shown in the right section of Figure 6.10a; The M -map shows variation in color from blue, to greenish, to yellow as we move from the $\langle 001 \rangle$ towards the $\langle 011 \rangle$, and finally towards the $\langle 111 \rangle$, respectively. The CoF values were averaged for each texture block as well as the corresponding M -values along the scratch (see 3.4 for more details on the calculation of M). Subsequently, unsupervised machine learning k-means clustering algorithm was used to arrive at the three clusters shown in Figure 6.10b. Each of the three clusters' centroids (red dots) represent the two-dimensional mean of the dataset. To explicate, the vertical position of the dot represents the cluster's average CoF for that particular cluster, whereas the horizontal position of the dot represents the cluster's average M -value. The translation of the centroid downward with the increase in M denotes a decrease in the average CoF of friction as stiffness increases (i.e., an inverse relationship between the crystal orientation stiffness and the CoF). This finding supports the previous findings showing higher CoF values in the $\langle 001 \rangle$ region (least stiff) and lower CoF values in the $\langle 111 \rangle$ region (most stiff).

It is worth noting that the spread (or accuracy) in the datapoints in Figure 6.10b is related to the strength of texture as well as the morphological anisotropy of grains. As for the former, the texture strength of this specimen is relatively weak compared to the other specimens (compare Figure 6.3 with Figures 6.2 and 6.4). In turn, the indenter frequently encounters crystals whose orientations do not belong to either of the dominant textures ($\langle 001 \rangle$, $\langle 011 \rangle$, or $\langle 111 \rangle$). It is known that the CoF fluctuates as the indenter encounters a grain whose easy shear is misaligned from that required by the indenter to induce plastic flow [198, 199, 202]. When this occurs, a spike in the CoF occurs in turn. In relation to the morphological anisotropy of grains, the indenter here moves parallel with the minor axis of the high-aspect-ratio grains. As such, the motion of the indenter along this axis makes crossing of HAGBs more frequent than if the indenter were to move parallel with the major axis of the high-aspect-ratio grains. As a result, the encountering of randomly oriented crystals (i.e., diverging from the dominant texture) is exacerbated. Combined, the weak texture and anisotropic grain morphology contribute to the fluctuation in the CoF.

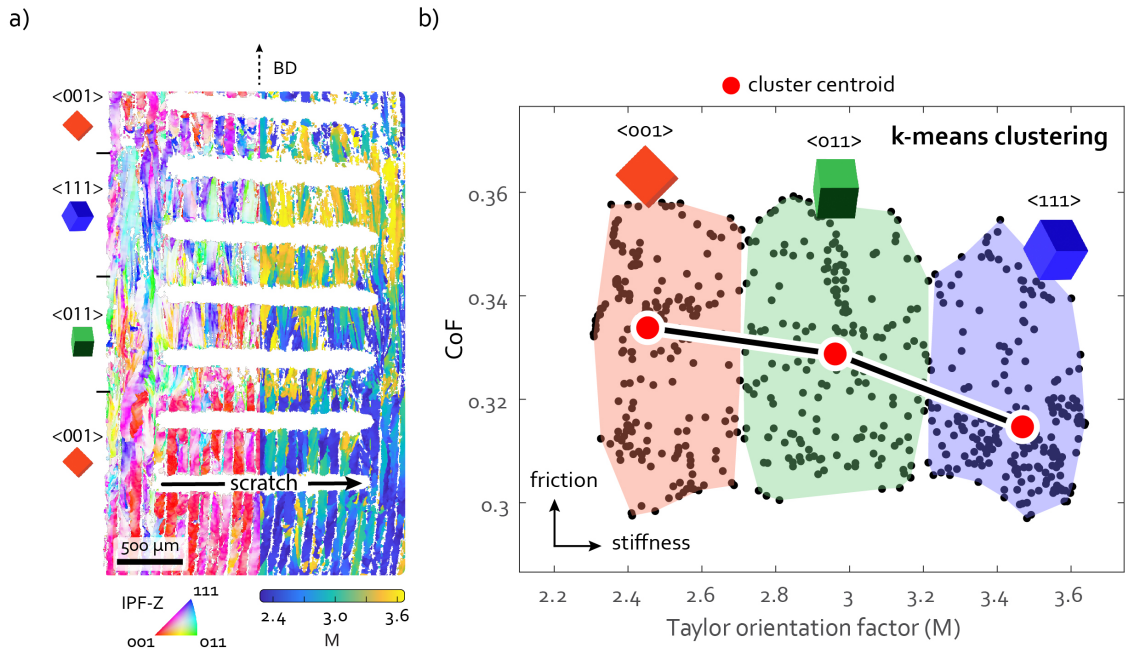


Figure 6.10: Correlation of CoF and Taylor orientation factor (M). a. IPF map and M -map side-by-side showing the effect of $\langle 001 \rangle / \langle 011 \rangle / \langle 111 \rangle$ textures on M . b. Relationship between M and the CoF is revealed by k-means clustering algorithm. The downward shifting of the centroid with increase in M indicates that CoF decreases as the crystal projects its ‘stiffer’ planes perpendicular to the normal load. Although the CoF differences between $\langle 001 \rangle / \langle 011 \rangle / \langle 111 \rangle$ are marginal, it should be noted that the data is not generated of a strongly textured specimens. Thus, it is expected that the local texture variation would impact the averaged value of CoF.

6.2.3 Impact of texture strength on wear

A previous study by Yang et al. [82] concluded that wear in SLM 316L behaves isotropically when the normal load increases, specifically at 5 N. Crucially, this finding means that the anisotropic benefit obtained from SLM steel becomes marginal as load increases. Here, we apply much scrutiny towards this (an)isotropic behavior, especially considering the impact texture strength on wear. To this end, we perform a macro-scratch testing using a normal load three times higher than that used by Yang et al. (i.e., 15 N) on a specimen exhibiting spatially variant texture strengths (Figure 6.1a). Figure 6.11a-d shows the scratch and corresponding orientation, GND- and M -maps in a region exhibiting strong $\langle 011 \rangle$ texture. The maps and SE micrograph clearly show that the scratch ran perpendicular to the major axis of the high-aspect-ratio grains. The orientation gradient between these grains was overlayed on the SE micrograph (Figure 6.11a,b) to highlight the HAGBs ($\theta \geq 15^\circ$) and LAGBs ($\theta < 15^\circ$). HAGBs are denoted by purple-colored dots, whereas LAGBs are denoted by green-colored dots. It can be seen that ridges formed near HAGB-separated grains were more jagged than those formed near LAGB-separated grains (Figure 6.11a). At the same time, the GND map shows restricted motion of dislocations in HAGB-separated grains

compared to facilitated motion of dislocations in LAGB-separated grains (see arrows in Figure 6.11c). ρ in these concentration zones was in the range of $10^{15}/\text{m}^2$. Noteworthy, it is also evident that the restriction of dislocation motion in HAGB-separated grains induces a significant stress that causes grains to alter their orientation; the Taylor orientation map (Figure 6.11d) shows near-scratch HAGB-separated grains undergoing ‘stiffness swap’ (i.e., changing their orientation), which was not witnessed in LAGB-separated grains. Collectively, these findings allude to a different wear behavior in the presence of HAGBs than in LAGBs.

The lateral forces induced by the scratch induce plastic deformation by dislocation motion. The motion of dislocation across grains is facilitated/impeded by the ability to transfer from the slip system of one grain to the other. Regardless of HAGBs or LAGBs, slip transfer is possible if the two slip systems are coincidentally aligned as to allow for such transfer. However, if the two slip systems are not aligned such that transfer is easily allowed, HAGBs induce more reaction forces than LAGBs (due to higher angle) [209]. In this scenario, dislocation motion is opposed as dislocations become sandwiched between the lateral forces induced by the indenter and the reaction force of transfer between two non-complementary slip systems. In turn, a portion of the lattice undergoes rotation as to accommodate these opposing forces (i.e., the stiffness swap in the case of HAGBs). By a similar explanation, a dislocation is freer to travel inside a lattice than across two lattices (when not accounting for dislocation-dislocation interactions). As a result, the deformation of the lattice (i.e., the grain) is relatively easily facilitated than the deformation of an interface between two lattices (i.e., the grain boundary). The end result of this inconsistent traveling of dislocations is likewise an inconsistent plastic deformation, namely, a triangular-shaped ridge. When this happens over multiple grains, the combination of these triangular-shaped ridges yield the jagged form seen in Figure 6.11a.

The role of texture strength can be appreciated by comparing more closely the scratch of non-textured areas (grains with randomly oriented crystals) with textured areas. Figure 6.12a,b depict the orientation map and corresponding wear of two sites along the same scratch. It can be seen that when crystals are randomly oriented (Figure 6.12a), ridges are more jagged than if crystals were of nominally the same orientation (Figure 6.12b). The onset of wear debris generation is seen in when the crystals are randomly oriented. On the other hand, the ridges appear more intact when the surface is textured. This finding suggests that wear debris is more likely to be generated for a tribosurface with randomly oriented crystals than a textured one. Considering the various slip systems existing due to the numerous amount of neighboring random crystals, when a moving dislocation is impeded, the effect is more pronounced (due to higher angle between two coincidentally-non-complementary slip systems). In turn, the impeded dislocations pile-up resulting in ductile cutting in accommodation of the shear stresses.

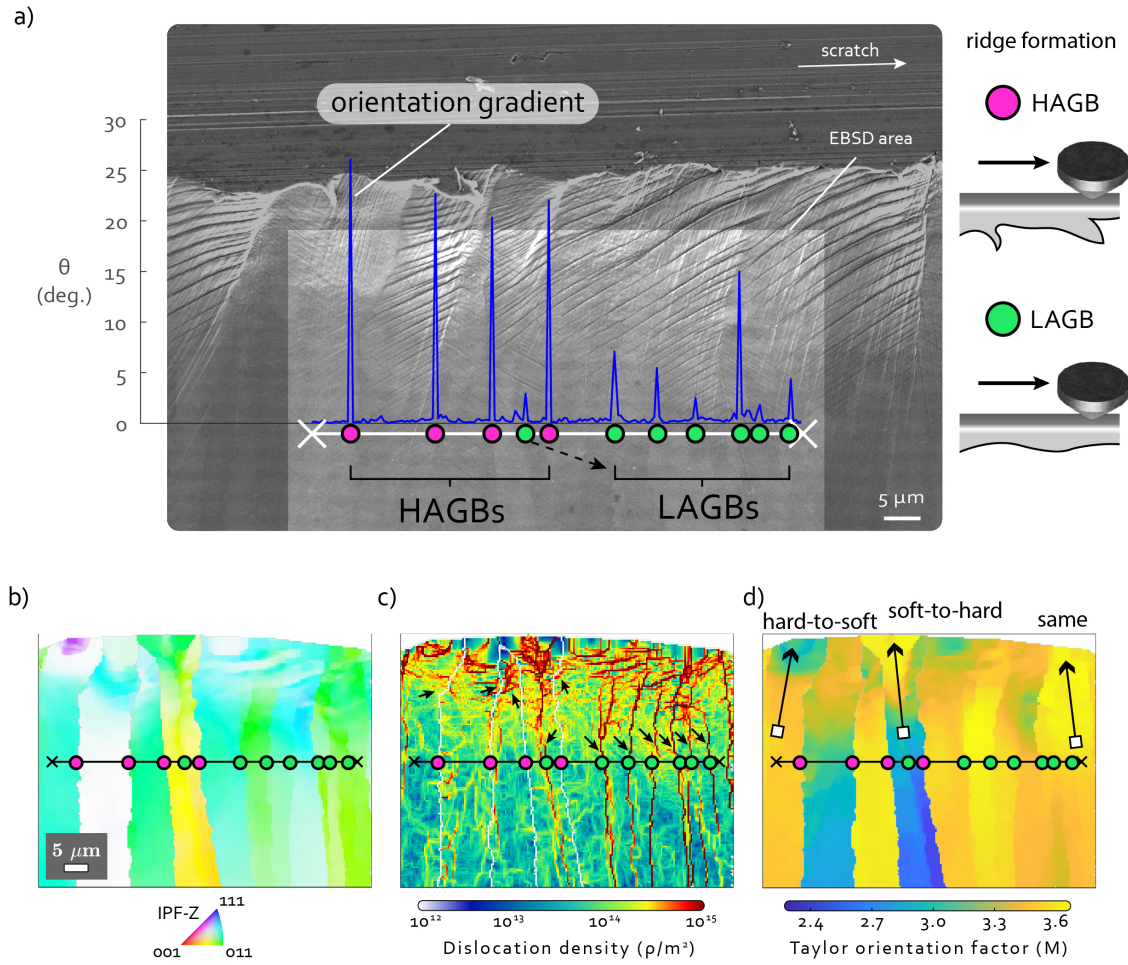


Figure 6.11: The impact of crystallographic texture strength on ridge formation. a-d. Deformation in $\langle 011 \rangle$ -textured area shows that presence of HAGBs correlate with a heterogeneous ridge formation, whereas the presence of LAGBs correspond with a homogenous formation of ridges. a. Orientation gradient along a line graph overlaid on SE micrograph. The left side was mostly dominated by HAGBs, whereas the right side was mostly dominated by LAGBs. b. IPF map highlighting the $\langle 011 \rangle$ -textured high-aspect-ratio grains. c. Geometrically necessary dislocation (GND) map showing the dislocation densities. Dislocations were able to travel far away from the scratch in LAGBs, whereas dislocations were mostly concentrated near the scratch area in the HAGBs case (see arrows). d. M -map shows stiffness swap in HAGB-separated regions, unlike LAGB-separated regions.

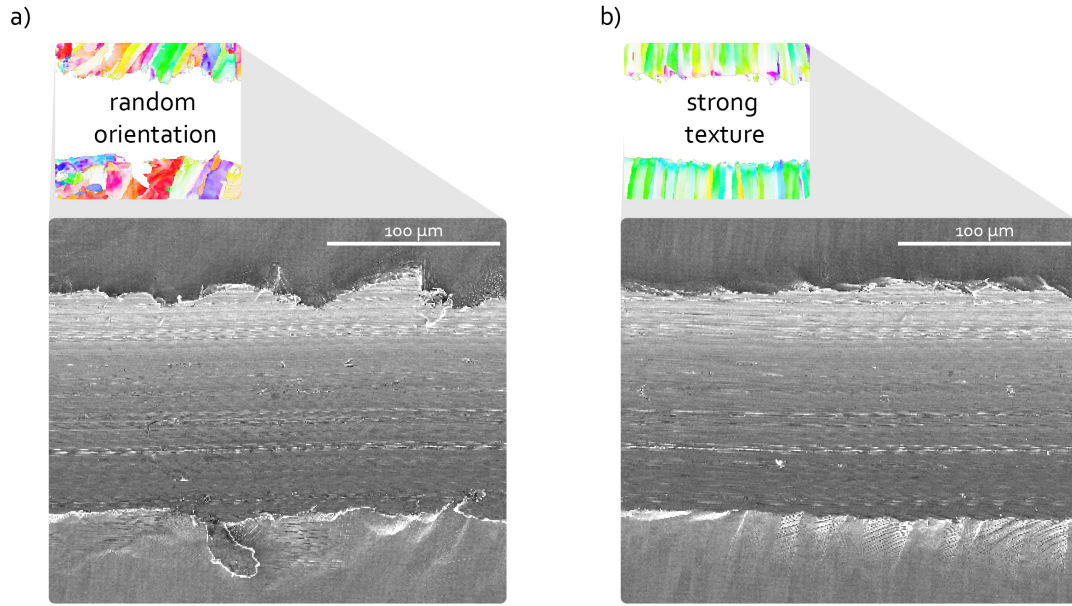


Figure 6.12: The impact of crystallographic texture strength on wear. a,b. A comparison of wear between randomly oriented crystals and $\langle 011 \rangle$ -textured surfaces along the same scratch. a. Surface with randomly oriented crystals exhibited higher susceptibility to generation of wear debris due to heterogenous formation of ridges. Whereas, b. $\langle 011 \rangle$ -textured surface showed no traces of wear debris due to homogenous ridge formation.

In a general sense, these results show that the anisotropic wear can be modified by (crystallographic) texture control. Although Yang et al. [82] reported that wear behaves isotropically at greater loads, namely at 5 N, it was shown here that wear continues to behave anisotropically at a load three times of that reported by Yang et al. As such, the control of tribological anisotropy in SLM alloys remains a subject worth investigating.

6.3 Chapter conclusion

In this study, microstructure control was used to produce multigrade tribosurfaces comprising various crystallographic textures. Experimental tribology was used to further underscore the tribological anisotropy induced by microstructure control in 316L manufactured by selective laser melting. The results showed the tribological anisotropy (stemming from the microstructure) is active over a wide-load spectrum ranging from 0.01 to 15 N. In terms of friction, the results suggest an inverse relationship with the Taylor orientation factor accounting for the orientation stiffness. Specifically, as the lattice's Taylor factor increases, friction decreases, and vice-versa. In terms of wear, the results suggest that wear debris may be mitigated by effective control of crystallographic texture. Considering the multitude of SLM-process parameter and alloy combinations, research concerning the tribological anisotropy of these alloys will likely continue to unfold over a long period. This research can be enabled via multigrade tribosurfaces similar to those used in this work.

Chapter 7

Concluding chapter

7.1 Concluding remarks

The studies collectively underscored and revealed the critical and complex role the SLM-induced microstructure plays in the tribological behavior (first aim of the thesis). In particular, it was demonstrated that wear mechanisms, which in turn impact the wear performance, are indeed anisotropic in SLM-manufactured steel (Chapter 4). Hence, it was argued that ‘hardness’ and ‘porosity’ should not solely be used as predictors for wear performance when comparing additively manufactured tribosurface with a conventionally manufactured one. Rather, a holistic approach considering the nuances of the microstructure should be implemented. We then observed the anisotropic tribological contact evolution which revealed several unique roles the SLM-induced microstructure plays in the tribological behavior (Chapter 5). For instance, we showed that the increased dislocation density in as-built SLM steel leads to a slight increase in its steady-state coefficient of friction compared with conventionally manufactured annealed steel. Furthermore, we saw that the Taylor factor (corresponding to the directional stiffness of the crystal) is correlated with the type of wear debris.

With the above insights, it was evident that microstructure had a critical role in the tribological behavior of SLM-manufactured steel. Therefore, it was viable to exploit SLM’s ability of deliberate microstructure design to examine the prospect of designing multigrade tribosurfaces (Chapter 6). Several tribological experiments revealed that it is possible to ‘control’ the tribological properties by deliberate microstructure design (second aim of the thesis). These experiments revealed additional mechanistic insights which were particularly facilitated via the fabricated multigrade tribosurfaces. Such as an inverse relationship between the Taylor factor and the coefficient of friction; and a correlation between the strength of crystallographic texture and the susceptibility to generating wear debris. These findings confirm that controlling the anisotropic response through deliberate microstructure design is viable.

7.2 Recommendations for future work

The results in this work underscored the importance of scrutinizing the microstructure of additively manufactured steel in relation to the tribological properties. Such effort can be expanded to cover a wide range of alloys available via additive manufacturing, such as Ti-based alloys, CoCrMo-alloys, Al-alloys, etc. Furthermore, the insights from this thesis can be helpful in aiding the analysis of complex dual-phase microstructures such as duplex-grade steels. Moreover, the microstructure control shown here can be leveraged to the extensive selection of alloys aforementioned here. Ultimately, it would be desirable to produce site-specific tribological properties custom built to fit the specific tribological system at hand.

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Glossary

DMLS Direct metal laser sintering is synonymous with selective laser melting (SLM). It is a powder-bed-fusion process which relies on full melting of metallic powder to produce parts.. 53

EBM Electron beam melting is a powder-bed process which high-energy electron beam to melt from a wide range of metallic alloys.. 53

LENS Laser engineered net shaping is a trade name for a process similar to LMD (see LMD below).. 53

LFR Laser forming repair deposits a powder via a nozzle which is simultaneously melted with a laser beam upon deposition. Powder material primarily consists of metallic alloys.. 53

LMD Laser metal deposition is a direct energy deposition (DED) process by which a nozzle deposits melted material onto a workpiece and/or build platform. LMD is synonymous with the trade name 'LENS', which uses powdered metal alloys, ceramics, and composites.. 53

SLS Selective laser sintering is strictly a sintering process which does not require full melting of the material substrate. Lasers are used to sinter powder material such as Nylons.. 53

Permissions

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12	2.16	(top)	Elsevier	5441890590158	Dec 12, 2022

* schematics

[†] SE micrographs

Appendices

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Concept Mlab Cusing R technical specification sheet

10/2016 (Subject to technical changes.
Machine layout (handling station) not shown into
Photos: user-mueller-laser.de
Artwork: brandnew-design.de

Mlab cusing R TECHNICAL DATA

Build envelope LaserCUSING®	50 x 50 mm² (x, y) 70 x 70 mm² (x, y) 90 x 90 mm² (x, y) z = 80 mm
Layer thickness LaserCUSING®	15 – 50 µm
Production speed LaserCUSING®	1 – 5 cm³/h (depending on material)
Laser system	Fibre laser 100 W (cw)
Max. scanning speed	7 m/s
Focus diameter	approx. 50 µm
Fixation of the building plate	mechanical (clamping system being prepared)
Connected loads	Power consumption max. 1.5 kW Power supply 1/1/PE AC 230 V, 16 A 1 gas connection provided / Nitrogen or Argon
Inert gas supply	approx. 0.6 – 0.8 l/min*
Inert gas consumption	
Dimension machine	705 x 1848 x 1220 mm (W x H x D)
Dimension handling station	729 x 1391 x 628 mm (W x H x D)
Weight machine	approx. 600 kg
Weight handling station	approx. 100 kg
Operating conditions	15 – 35°C
LaserCUSING® materials	CL 20ES Stainless steel (1.4404) CL 31AL Aluminium (AlSi10Mg) remanium® star CL Cobalt-chromium alloy (Dentaurum) rematitan® CL Titanium alloy (Dentaurum) CL 41Ti ELI Titanium alloy (TiAl6V4 ELI) CL 42Ti Commercially Pure Titanium Grade 2 CL 80CU Bronze Yellow gold 18 carat 3N*** Rose gold ** 18 carat 4N*** Red gold ** 18 carat 5N*** Platinum 950 ‰ Platinum alloy*** Silver alloy 930 ‰ Silver alloy***

*Inert gas consumption during the building process with N₂.
** The material is currently being prepared.
Other materials on request.
*** Parameter from Concept Laser GmbH, material procured directly from precious metal supplier, contact details available on request.

m lab
cusing

Concept Laser GmbH An der Zeit 8 D 96215 Lichtenfels	T: +49 (0) 95 71 16 79 200 F: +49 (0) 95 71 16 79 299 info@concept-laser.de	Concept Laser Inc. 1000 Texan Trail, Ste 150 Grapevine, TX 76051 USA	T: +1-817-328-6500 info@conceptlaserinc.com www.conceptlaserinc.com
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www.concept-laser.de

SLM 500 technical specification sheet

Efficient, Reliable, Repeatable

SLM[®] 500

Selective Laser Melting Machine



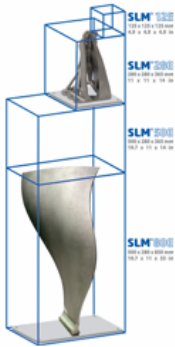
The image shows the SLM 500 machine system with four numbered components: 1. Main SLM 500 unit with a control panel and monitor; 2. Intermediate processing unit; 3. PSV (Powder Supply Valve) unit; 4. Powder handling unit.

Technical Specifications

Build Envelope(L x W x H)	900 x 280 x 365 mm reduced by substrate plate thickness
3D Optics Configuration	Twin (2x 400W or 2x 700W) Quad (4x 400W or 4x 700W) IPG fiber laser
Real Build Rate	up to 171 cm ³ /h*
Variable Layer Thickness	20 µm - 90 µm, more available on request
Minimum Feature Size	150 µm
Beam Focus Diameter	80 - 115 µm
Maximum Scan Speed	10 m/s
Average Inert Gas Consumption in Process	16 l/min (Argon)
Average Inert Gas Consumption in Purging	250 l/min (Argon)
E-Connection / Power Input	400 Volt 3NPE, 63 A, 50/60 Hz, 8 - 10 kW
Compressed Air Requirement	ISO 8573-1:2010 [1:43] 7 bar
Machine Dimensions (L x W x H)	6080 x 2530 x 2620 mm

*depending on material and build part geometry

Build Chamber Sizes



The diagram shows three build chambers of different sizes, each containing a part. The sizes are: SLM 500 (1000 x 1000 x 1000 mm), SLM 500 (1000 x 1000 x 1000 mm), and SLM 500 (1000 x 1000 x 1000 mm).

Appx 2

EBSD sample preparation procedure

Appx 3

#	Type	Solution/suspension	Load	RPM rel.motion	Time
1	P320 $\approx 46\ \mu\text{m}$	H ₂ O	5 N	150 $\curvearrowright \curvearrowright$	90 s
2	P600 $\approx 26\ \mu\text{m}$	H ₂ O	5 N	150 $\curvearrowright \curvearrowright$	90 s
3	P1200 $\approx 15\ \mu\text{m}$	H ₂ O	5 N	150 $\curvearrowright \curvearrowright$	90 s
4	————— H ₂ O + soap $\rightarrow$ rinse $\rightarrow$ Ethanol (optional) $\rightarrow$ air dry —————				
5	Nylap cloth	9 μm diamond suspension	5 N	150 $\curvearrowright \curvearrowright$	90 s
6	Plano cloth	6 μm diamond suspension	5 N	150 $\curvearrowright \curvearrowright$	90 s
7	Alpha cloth	1 μm diamond suspension	5 N	150 $\curvearrowright \curvearrowright$	90 s
8	Alpha cloth	0.04 μm OPUS (non crystallizing) colloidal silica	5 N	150 $\curvearrowright \curvearrowright$	90 s
9	————— H ₂ O + soap $\rightarrow$ rinse $\rightarrow$ Ethanol (optional) $\rightarrow$ air dry —————				
10	Electro-etch	10% Oxalic	5 V	N/A	30-90 s

Wyko NT9100 WLI specification sheet

Wyko NT9100 Optical Profiling System

The Wyko NT9100 Optical Profiling System embodies convenience, affordability, and performance in non-contact three-dimensional surface metrology. This ninth-generation system utilizes a patent-pending dual-LED illumination source to provide superior non-contact measurements on the widest variety of super-smooth or very rough samples, from sub-nanometer surface roughness to millimeter high steps. The NT9100 has been specifically designed to provide the industry's most reliable and repeatable tabletop characterization for R&D, wear, failure analysis, process control, and monitoring.

As the bench-top model of the NT9000 Series optical profilers, the NT9100 shares many of the performance attributes with the larger systems, including: easy measurement setup, fast data acquisition, comprehensible and extensible data analysis, and angstrom-level repeatability. An X-Y stage automation option brings programmability to the NT9100, a first for a Wyko tabletop profiler.

The NT9100 provides performance on par with the other profilers in the NT9000 series, yet its foot print and flexible configuration provides a convenient foundation to grow with your needs. The system comes complete with the industry-leading Wyko Vision software package for advanced 2D and 3D data analysis and visualization. Vision provides over 200 built-in analyses, as well as automated measurement sequences (recipes), data logging, and pass-fail criteria for real-time process feedback and SPC. An optional MATLAB® package enables end users to develop custom Vision analyses for other specialized applications as well.



COVER APPLICATION IMAGES:

(Top) Copper Indium Gallium Selenide (CIGS) boundary region with different grain structures on either side of the boundary, 640x480 array (Image courtesy A. Hall/A. Rockett, Dept. of Materials Science & Engineering, Univ. of Illinois).

(Bottom) 2.7mm stitched measurement of 60-degree slope on machined threads, 20x magnification.

SPECIFICATIONS

Measurement Capability	Non-contact, three-dimensional, topography and film thickness
Objectives	1.5X, 2.5X, 5X, 10X, 20X, 50X for magnifications from 0.75X to 100X; Long working distance objectives available; Optional motorized turret; Optional Through Transmissive Media objectives
Field-of-View Multipliers	0.55X, 0.75X, 1X, 1.5X, 2X
Measurement Array	640 x 480, non-interlaced; Optional 1392 x 1040 camera
Light Source	Long-lifetime green and white LEDs
Optical Assembly	Dual LED illuminator; Three-position FOV turret, closed-loop, 10mm scan
Stages	Automated 100mm Z-axis; $\pm 6^\circ$ tip/tilt stage; 100mm XY manual stage, standard; 150mm XY auto stage, optional
Computer System	Latest Dell® with 24/7 support line;
Software	Vision running under Microsoft® Windows XP® Professional; Production mode, built-in databasing with pass/fail for any parameter; Optional Stitching, MATLAB/TCPIP, Film Analysis, Optical Analysis, and SureVision
Vertical Measurement Range	0.1nm to 10mm std.
Vertical Resolution ¹	<0.1nm
RMS Repeatability ²	0.05nm
Vertical Scan Speed	User-selectable up to 24µm/sec
Lateral Spatial Sampling	0.1 to 13.2µm (≤160nm with high resolution camera)
Optical Resolution ³	0.49µm min.
Field-of-View	7.68 x 5.76mm max, 0.06 x 0.05mm min with std-resolution camera; 7.84 x 5.87mm max, 0.09 x 0.07 min with high-res camera;
Reflectivity	<1 to 100%
Step Height	0.8% accuracy; <0.1% at 1σ repeatability
Profiler Footprint	320mm W x 510mm D x 645mm H; 70kg (excluding peripheral electronics and air table)
Certification	CE, NRTL

1. As demonstrated by a PSI difference measurement on a SiC reference mirror with nulled fringes and 10 averages.

2. As demonstrated by taking the one sigma Rq value of 30 PSI repeatability measurements on a SiC reference mirror.

3. Based on Sparrow Criteria at 535nm.

Note: Performance specifications are typical and subject to change without notice.

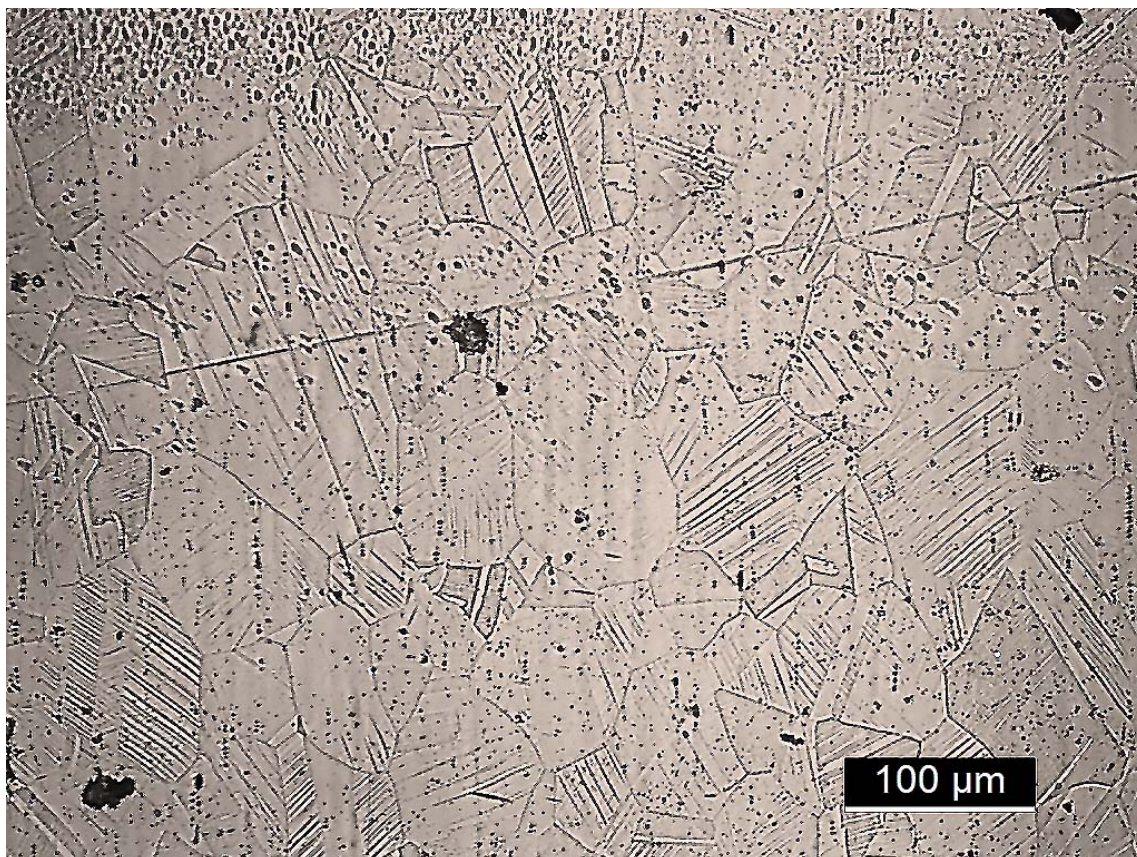


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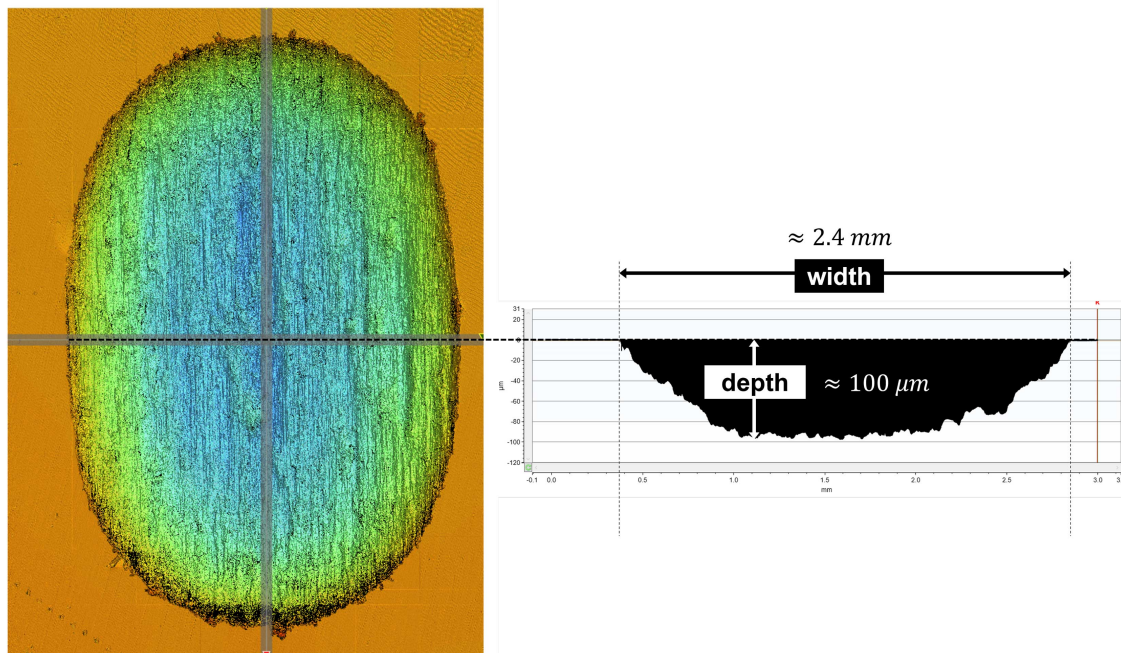
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Etched surface of conventionally wire-drawn specimen (WD)



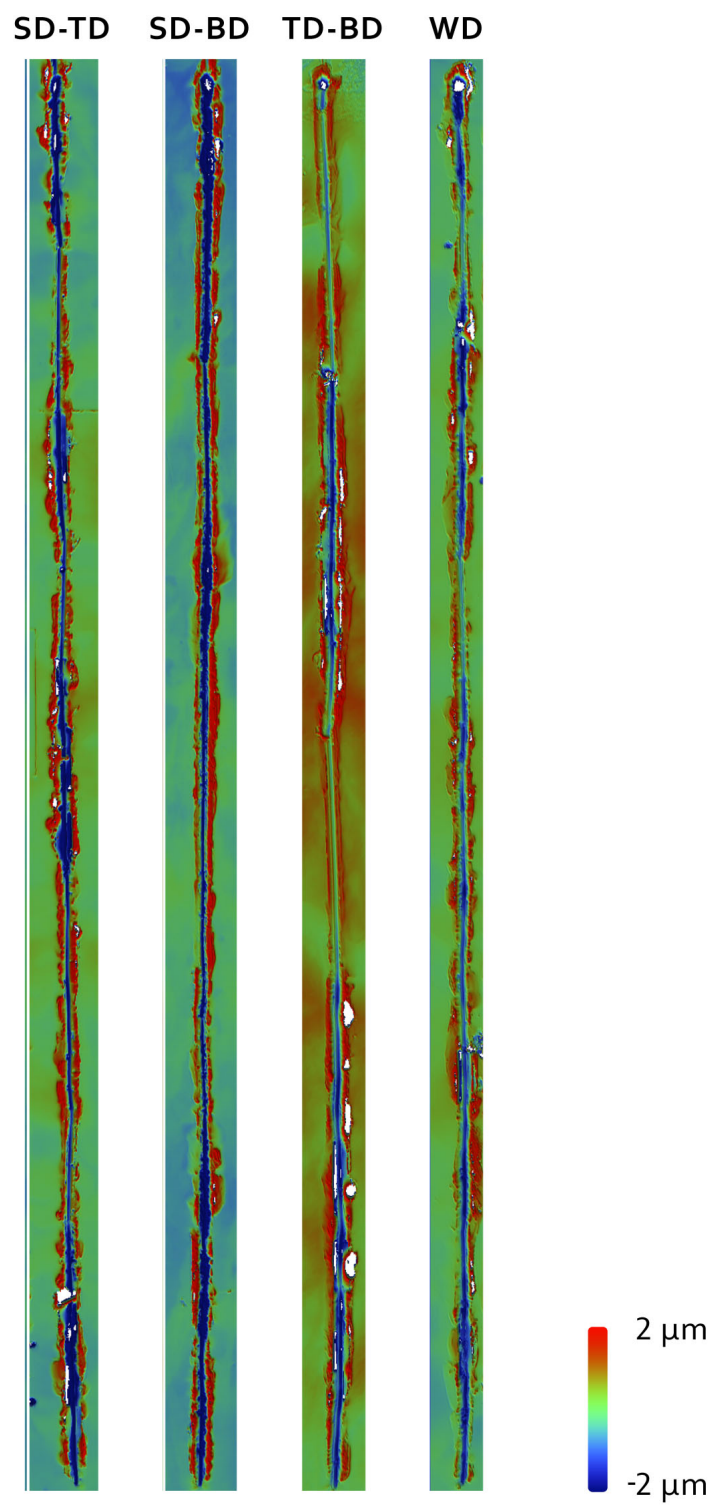
Appx 5

Interferometry measurement of full wear track

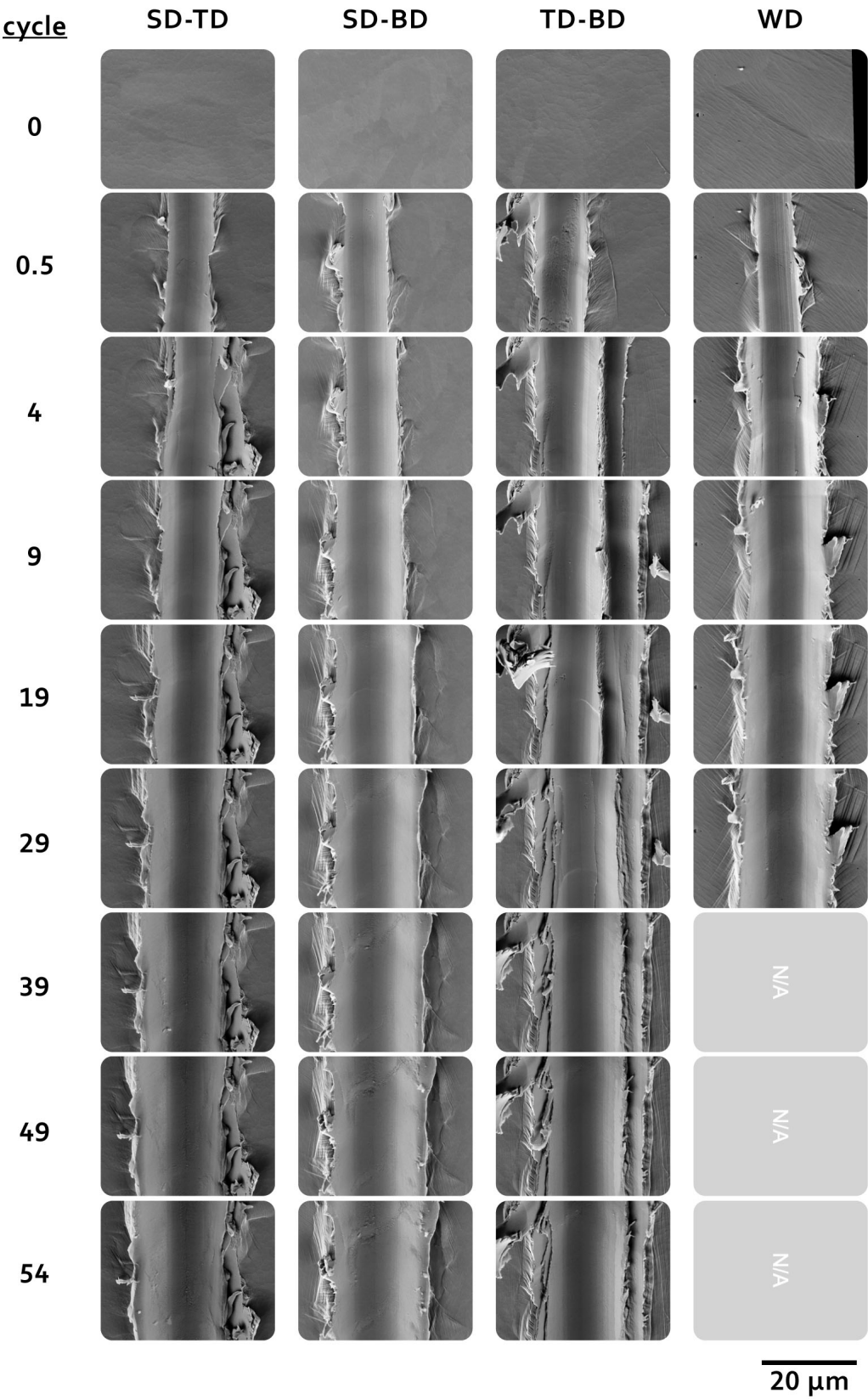


Appx 6

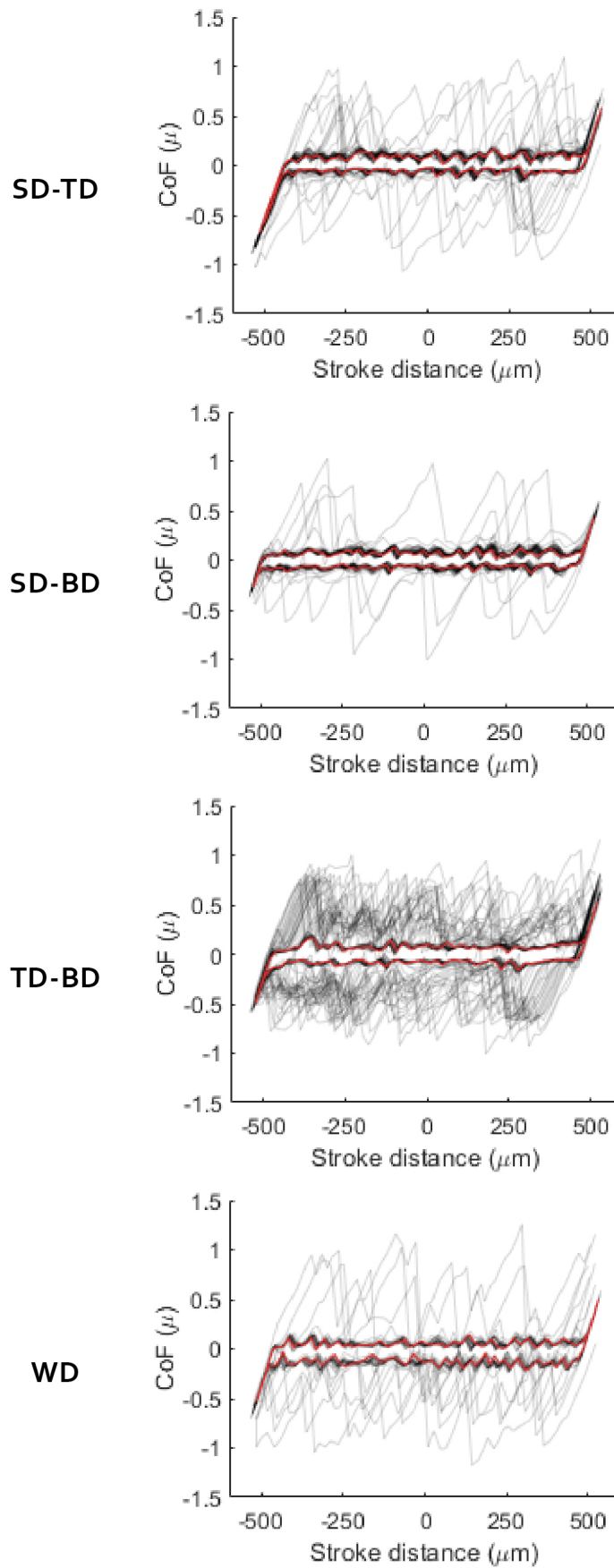
Interferometry measurement of entire strokes



Accessible format of Video 1



CoF data for multi-pass scratch testing



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End of thesis.

