Laser-powder bed fusion of silicon carbide reinforced 316L stainless steel using a sinusoidal laser scanning strategy

Andre Mussatto^{1, 2, 3, a}, Robert Groarke^{1, 2, 3}, Rajani K. Vijayaraghavan^{2, 3, 4}, Muhannad Ahmed Obeidi^{1, 2, 3}, Patrick J. McNally^{2, 3, 4}, Valeria Nicolosi^{5, 6, 7}, Yan Delaure^{1, 2, 3}, Dermot Brabazon^{1, 2, 3}

⁷ School of Chemistry, Trinity College Dublin, Dublin, Ireland

^a Corresponding author: <u>andre.mussatto2@mail.dcu.ie</u>

Highlights

- A novel hatching pattern for laser-powder bed fusion of metal matrix composites is introduced.
- A method for improving mechanical properties and achieving texture control is investigated
- The tribological and mechanical properties of naturally formed oxide films are studied.
- A method for reducing process lead time and energy consumption is presented.

Abstract

Laser-powder bed fusion was identified as a promising technique for manufacturing metal matrix composites. However, over a decade later, little progress has been made in addressing the persisting issues hindering the wider exploitation and industrial usage of metal matrix composites. Therefore, the present study proposes the implementation of a feasible solution to accomplish research advancements in metal matrix composites. Where, the issues concerning their performance and cost have driven this study towards the development of a novel scanning strategy. A hatching system based on a sine wave was successfully developed and employed in the printing of metal matrix composites. Composites printed using the sinusoidal hatching exhibited an enhanced yield strength and ductility owing to the resultant grain refinement and texture. The dabber mode formation of material tracks promoted the growth of highly oriented intragranular cellular structures. Apart for playing an important role at the obtained hardness, this control over the cellular growth could also be used towards improving composite toughness. Additionally, besides improving composite performance, the sinusoidal hatching was also effective in reducing manufacturing lead time and process energy consumption.

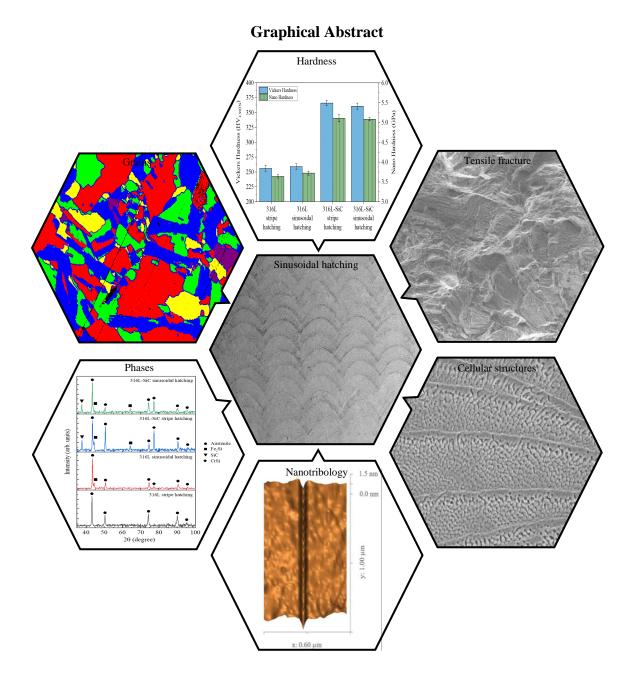
¹ School of Mechanical and Manufacturing Engineering, Dublin City University, Glasnevin, Ireland

² I-Form Advanced Manufacturing Research Centre, Dublin City University, Glasnevin, Ireland

³ Advanced Processing Technology Research Centre, Dublin City University, Glasnevin, Ireland
⁴ School of Electronic Engineering, Dublin City University, Glasnevin, Ireland

⁵ I-Form Advanced Manufacturing Research Centre, Trinity College Dublin, Dublin, Ireland

⁶ Centre for Research on Adaptive Nanostructures and Nanodevices (CRANN) and Advanced Materials and Bioengineering Research (AMBER), Trinity College Dublin, Dublin, Ireland



Keywords: powder bed fusion; sinusoidal scanning strategy; metal matrix composite; microstructure; nanotribology

1. Introduction

In many modern technological applications, components are very often required to have properties which are only possible by combining different materials, as against monolithic options individually [1]. Metal matrix composites (MMCs) are considered advanced materials due to the growing interest on them in the past decades in various high-performance applications such as those in the automotive and aerospace sectors [2,3]. Although MMCs have been used and are excellent candidate materials for advanced engineering systems, several aspects are hindering their further applications [3]. To which two of the possible reasons are their ductility and toughness achievements to date, and these properties' behavior in the presence of and with the increasing amount

of reinforcement [4]. Technical challenges including inhomogeneous reinforcement dispersion, low wettability, deleterious interfacial reaction, weak interfacial bonding, porosity and micro defects are well known to have a major impact on the properties of MMCs [5–11]. Another challenge of MMCs is the availability of a suitable processing technique to extract the maximum reinforcement benefits [12]. Ease of processing, adequate economic efficiency, low energy consumption and complex net-shape ability are also process related aspects to consider [3,13–15]. Even though substantial research is still needed, in specific applications, the MMCs property-profiles are distinguished from metals and alloys by offering a gain in performance. Yet, as far as industries are concerned, the viability of MMCs depends on the balance between their performance and cost [16].

As aforementioned, the production of MMCs via conventional manufacturing techniques face several challenges and limitations. Rapidly becoming more and more popular, laser-powder bed fusion (L-PBF), which is suitable for obtaining ultrafine, gradient and pointwise-controlled microstructure, has been used for the production of both in-situ and ex-situ MMCs [13,3,17]. L-PBF offers an exceptional flexibility for producing functional geometrically complex dense composites, besides allowing unprecedented freedom of design and customisation [18]. It provides also an opportunity for producing MMCs with a homogeneous dispersion of reinforcement and material pairings free from limitations which is extremely important when developing new materials [19,20]. Recently, L-PBF was identified as the most promising technique to process MMCs as it has been showing the potential of addressing several of the current issues concerning MMC production, including manufacturing costs, waste and lead time [21,22]. However, while there is promises, and the fact that L-PBF is still in its infancy, its true and full potential for MMCs are yet to be discovered.

To date, little research into L-PBF of silicon carbide (SiC) reinforced 316L composites exists. From these, it was reported that SiC additions affect microstructural morphology and texture, and improve the composite strength due to the grain boundary, Orowan, dislocation and load transfer strengthening mechanisms [23]. However, densification was reported to be affected by the SiC addition because of porosity. High residual stresses due to coefficient of thermal expansion mismatch between phases caused micro cracks, hence reduced the ductility in composites [23]. The decomposition of SiC encouraged the formation of iron and chromium silicide phases, and these were found to help to decrease the mechanical strength of MMCs [24]. The decomposition of dispersed SiC particles is a common phenomenon in L-PBF synthetised iron-based composites [25–27]. Although the decomposition of SiC is generally seen as deleterious for the strength properties, modification of the matrix with Si and C atoms can activate processes like solid solution strengthening [28]. On the other hand, the incorporation of high amounts of SiC can reduce its decomposition as the 316L matrix saturates with Si and C atoms [29]. However, in this case the ductility of the matrix is compromised and the microstructure is more prone to cracks and porosities [23,30].

In L-PBF, the spatial moving pattern of the energy beam is regarded as scanning strategy. This parameter is known to have an influence on the building time and properties of printed components [31–33]. The most common, not necessarily commercially available, filling patterns are stripes, islands (i.e. based on stripes, hexagons and chessboard), grid

zigzag, square spiral and contour offset [34–37]. Additionally, despite their limitations, fractal patterns such as the Hilbert and Peano-Gosper have been used as a method of reducing thermal gradients and residual stresses [38,39]. Unfortunately, all of these aforementioned infill patterns have one thing in common and this is the fact that their resulting scanning paths are linear. Hence, the generation of the nature inspired and custom scanning strategies, which are very often more suitable for components with complex geometries/features or is required to avoid printing defects and or to achieve a desirable component property value, is limited by this factor. Unfortunately, current commercial slicer softwares do not implicitly enable generation of such laser paths. Bo et al. [40] developed a helix scanning strategy based on the Voronoi diagram of the model slice and the recursive generation of toolpath algorithm for an engine impeller aiming to reduce the shape deformation magnitude and residual stress profiles. Similarly, the equidistant scanning algorithm was claimed to improve processing efficiency and component quality [41]. In fact, several other path generation algorithms that might be suitable for developing new scanning strategies can be found in the literature [37,42–46].

In this work, a specially developed powder mixture having a high flowability and spreadability performance was used for L-PBF of MMCs. A novel scanning strategy, here named sinusoidal hatching, was developed and used during the printing process in an attempt to improve the composites tensile properties and control crystallographic texture. Additionally, this scanning strategy was also explored as a feasible solution to some of the aforementioned issues faced by MMCs.

2. Experimental

2.1 Material and Sample Preparation

The characteristics and performance of the powder mixture developed for this work is presented in our previous studies [47,48]. The powder mixture is composed of a free-flowing gas atomised (35-50 μ m) stainless steel 316L powder and a high purity (45-65 nm) SiC powder, obtained from Mimete S.r.l and US Research Nanomaterials Inc. Figure 1 shows the prepared powder mixture (316L + 1 wt.% of SiC) which is characterised by the cohesive SiC nanoparticles adhering and evenly decorating the surface of 316L particles.

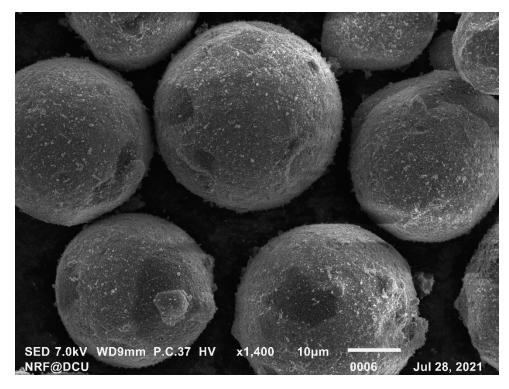


Figure 1 Micrograph of the prepared Nano-SiC satellited 316L feedstock powder.

The scanning strategies used in this work are illustrated in Figure 2. For generating the stripe hatching, models of the specimens were designed using SolidWorks and then the hatching was generated using a commercially available slicing tool, Netfabb Autodesk. While for the sinusoidal hatching, the three-dimensional design of the specimens and slicing were generated using a sine function, Excel Macro and JavaScript, then converted into Common Layer Interface. The inert gas (argon) flow was set perpendicular to hatching. Additionally, in order to reduce heat concentration zones, unidirectional scanning of the laser paths was considered.

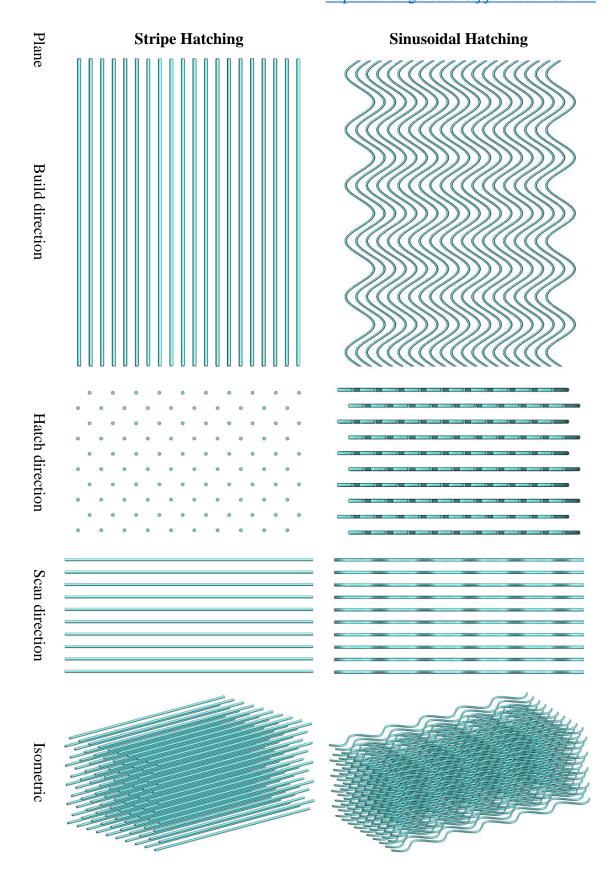


Figure 2 Illustration of the hatching systems used in this work.

Using an Aconity Mini (Aconity GmbH, Germany), cuboids and pins were printed based on the following design: 316L stripe hatching, 316L sinusoidal hatching, 316L-SiC stripe

hatching and 316L-SiC sinusoidal hatching. The prints were repeated three times to enable verifying the results and to estimate the experimental variability. To avoid property variability, caused by the location parameter, specimens were only printed in the centre of the build platform [49]. The laser power and scanning speed for each individual hatching were established from a processing window obtained prior printing the specimens through single track experiments. Categorised by the hatching system, the processing parameters used during the printing of the specimens are listed in Table 1. As the printed specimens were to be either machined or ground (and polished) for characterisation, the contour laser scanning was not employed in this study.

Table 1 Processing parameters.

Parameter	Stripe Hatching	Sinusoidal Hatching
Laser power (W)	180	50
Scanning speed (mm/s)	600	10000
Laser beam diameter (µm)	50	50
Layer thickness (µm)	50	50
Hatch spacing (µm)	75	75
Hatch translation per layer (µm)	37.5	37.5
Amplitude (µm)	-	50
Period (µm)	-	200

2.2 Sample Characterisation

The density of the specimens was determined by means of densimetry based on Archimedes' principle (density scale Avery Berkel FA215DT and Sartorius YDK01 Density Determination Kit). Deionised water (at room temperature) was used as the immersion liquid while determining the density. The Vickers hardness was measured using a Leitz microhardness tester and the measurements were performed according to ASTM E92 standard [50]. A nanoindenter (Bruker Hysitron TI Premier, USA) equipped with a standard Berkovich diamond indenter was used for nanohardness measurement of the specimens. An array of 6x14 nanoindentations was performed with 10 mN load and intervals of 30 µm between indentations. The horizontally printed pins were machined to dimensions according to the ASTM E8 standard round Specimen 4 [51]. The tensile test was performed using a Zwick Z050 (Zwick/Roell GmbH, Germany) fitted with Zwick TestXpert software and equipped with an Epsilon clip-on extensometer model 3542 of a 10 mm gauge length. The used tensile testing speed was 3 mm/min. Nanoscratch tests were performed in a Bruker Dimension Icon atomic force microscope equipped with a NanoScope V controller and using a single crystal diamond tip (AD-40-SS) from Adama Innovations. A micrograph of the tip is available in Figure 3. First, the probe's deflection sensitivity of 60.47 nm/V and its cantilever spring constant of 54.13 N/m were determined by the thermal tune method. Then, single pass scratches were performed in contact mode with an applied normal force of 1 µN and tip lateral velocity of 1 µm/s. A Zeiss Evo LS15 and a JEOL JSM-IT100 scanning electron microscope (SEM) were used to obtain microstructural data. The microstructure was also investigated using a tripleaxis Jordan Valley Bede D1 high resolution X-ray diffraction (HR-XRD) system with a copper (λ = 1.5405 Å) radiation source operated at 45 kV and 40 mA, and a Zeiss Supra 40 field emission scanning electron microscope equipped with a Bruker e-FlashHR electron backscatter diffraction detector. Lastly, a Zeiss EVO LS-15 SEM equipped with a Xplore 15 detector from Oxford Instruments was utilised for elemental analysis.

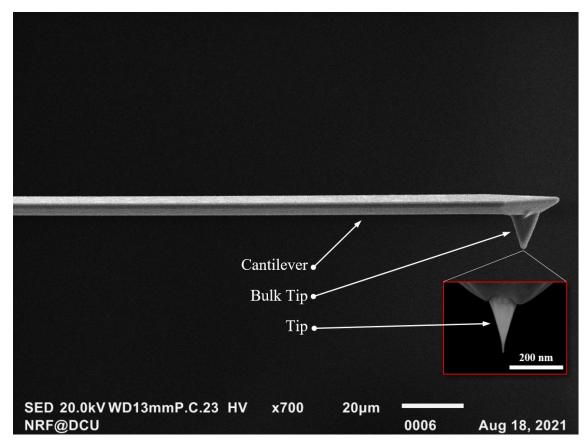


Figure 3 Single crystal diamond atomic force microscopy probe used in the nanoscratch testing.

3. Results

3.1 Density

The density of the printed specimens is presented in Figure 4. In order to properly assess the specimen's densities, the density of the virgin powders was also measured. Using a Micromeritics AccuPyc 1330 Helium pycnometer, densities of 7.75 and 3.16 g/cm³ were confirmed for the 316L and SiC powder. Based on this it would be expected a 100 % dense unreinforced and reinforced specimen to have 7.75 and 7.70 g/cm³. However, the densities reported in Figure 4 suggest the presence of internal defect in the specimens. It is clear that, independently of the hatching system, the presence of nano SiC particles have promoted specimen densification. It is also seen that in comparison with the stripe hatching, those specimens printed using sinusoidal hatching showed a drop in density.

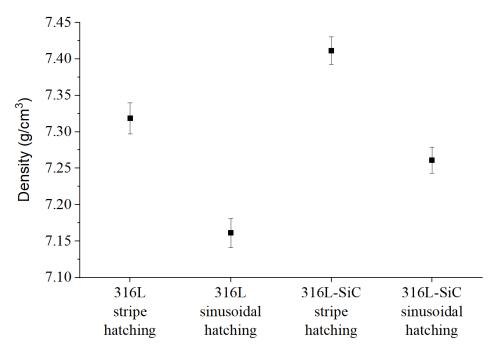
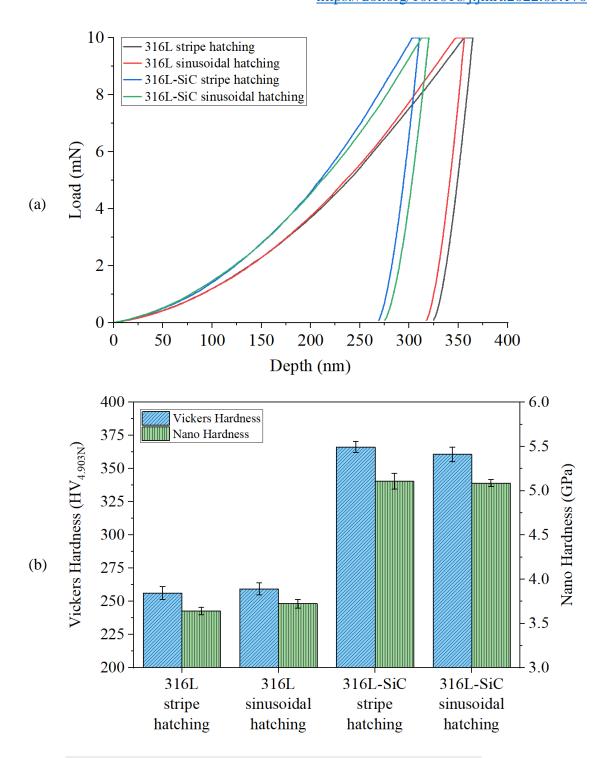


Figure 4 Compares the densities measured from the printed specimens. The data whiskers represent the 95% confidence interval.

3.2 Hardness

The hardness of the specimens measured from the plane perpendicular to the build direction is presented in Figure 5. The lower penetration depth, in the load-depth plot, corresponds to higher hardness, Figure 5a. It is worth noting that the micro and nanohardness have shown similar trend. The hardness of the 316L sinusoidal hatched specimen is slightly higher than that of the stripe hatched specimen. Regardless of this, based on previous studies, here both the micro [52,53] and nanohardness [54,55] holds a degree of improvement. The addition of 1 wt. % SiC has improve the hardness of the base material by approximately 30 %. A similar study also reporting the L-PBF of 316L-SiC claimed 347 HV for their 1.278 wt. % reinforced composite [23]. Therefore, despite using less reinforcement, the composites here printed presented higher hardness. Clearly, the hardness of the specimens can be related to their yield strength and elastic modulus, as well as their reduced modulus, Figure 5c. Factors such as grain size and the presence of residual stresses associated to the L-PBF process could be contributing to the seen hardness improvements.

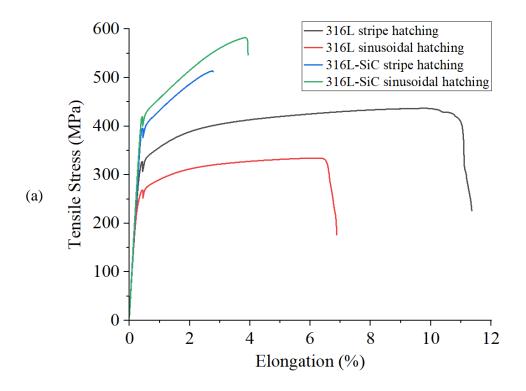


	Specimen	Reduced Modulus (GPa)	
	316L stripe hatching	$194.05_{\pm 1.81}$	
(c)	316L sinusoidal hatching	$195.07_{\pm 2.45}$	
	316L-SiC stripe hatching	$206.91_{\pm 1.44}$	
	316L-SiC sinusoidal hatching	$204.81_{\pm 2.03}$	

Figure 5 Measured Vickers and nanoindentation data: (a) nanoindentation load-displacement curves, (b) micro and nanohardness and (c) reduced modulus. The data bounds noted are 95% confidence intervals.

3.3 Tensile Properties

The tensile properties extracted from the specimens while applying tension along the scanning direction are shown in Figure 6. Figure 6a shows that the 316L specimen printed using the stripe hatching exhibited the highest elongation at fracture. In comparison to the 316L sinusoidal hatching, this specimen also holds superior tensile properties, Figure 6b. The addition of SiC led to an increase in yield strength, ultimate tensile strength and elastic modulus of the base material. However, the enhancement of these properties was accompanied by a loss of ductility. Notably, the sinusoidal hatching system provided a positive effect on the tensile properties of the composite. Where in contrast with the composite printed using the stripe hatching, this composite presented superior tensile strength and ductility. Despite the used input process parameters had been established through single track screening, one should note that the tensile properties of the specimens were influenced by their low density, which is related to the presence of porosities and lack of fusion defects. Future research should therefore adopt a statistical approach for further optimisation of the input process parameters. A recent study reported that tighter overlap of melt pools (hatch spacing) is required to ensure enough heat accumulation for bonding between tracks, and so to increase specimen densification [56]. Also, the preheating of the powder bed to a temperature of 200 °C can be used to improve the heat absorptivity of the powder, aiding to the complete melting of powder. Additionally, laser rescanning (remelting) was reported to improve specimen densification [57]. Therefore, for specimens in which density may be critical for crack formation and propagation, an increased melt pool overlap, the incorporation of powder bed preheating and layer remelting may be employed.



	Specimen	UTS (MPa)	YS (MPa)	E (GPa)
	316L stripe hatching	$431.82 {\scriptstyle \pm 3.68}$	$343.37_{\pm 4.80}$	$131.93_{\pm 2.36}$
(b)	316L sinusoidal hatching	$330.17 {\scriptstyle \pm 7.87}$	$278.78 \scriptstyle{\pm 2.67}$	$126.86 \scriptstyle{\pm 2.04}$
	316L-SiC stripe hatching	$514.48_{\pm 3.26}$	$414.25{\scriptstyle\pm2.73}$	$137.08 \scriptstyle{\pm 2.51}$
	316L-SiC sinusoidal hatching	$561.01_{\pm 15.86}$	$445.79_{\pm 4.05}$	$147.67_{\pm 2.79}$

Figure 6 Tensile performance of the specimens: (a) tensile stress versus percent elongation curves and (b) ultimate tensile strength, yield strength and elastic modulus. The data bounds noted are 95% confidence intervals.

Fractural analysis on the specimens was conducted using SEM observation, as shown in Figure 7. The existing porosities was considered as a particularly adverse factor for the tensile performance of the specimens as they contributed to and accelerated void growth and crack propagation causing premature fractures and failure under the tensile loads. The specimen of Figure 7a shows a ductile fracture in the presence of shear lips, whereas the specimen of Figure 7b shows almost no necking and a slanted fracture path indicating failure by ductility tearing from void coalescence induced microcracks [58]. Both of these specimens exhibited relative uniform dimpled fracture surfaces, with small dimples (around 1 µm in diameter) visible throughout the surfaces. Therefore, this suggests the existence of a subgrain structure, which affected the nucleation and growth of microvoids. The fracture morphologies of the composite specimens are shown in Figure 7c and 7d. The seen cleavage facets indicate that a brittle fracture process was developed during the failure due to the presence of the reinforcing brittle second phases. Despite the presence of brittle second phases, the composite of Figure 7d printed using the sinusoidal hatching also shows the presence of dimple patches, hence both brittle and ductile fracture characteristics were observed in this composite. Therefore, this resulted in the Journal of Materials Research and Technology Volume 18, 2022, Pages 2672-2698 https://doi.org/10.1016/j.jmrt.2022.03.170

consumption of more plastic deformation, which is also reflected on the maximum elongation before failure between the two hatching systems seen in Figure 6 for the composite specimens.

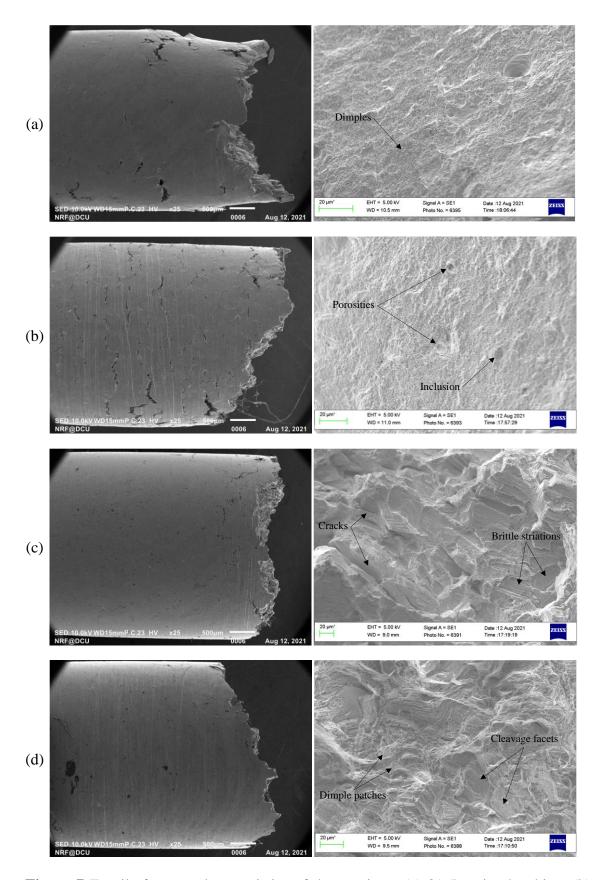


Figure 7 Tensile fracture characteristics of the specimen (a) 316L stripe hatching, (b) 316L sinusoidal hatching, (c) 316L-SiC stripe hatching and (d) 316L-SiC sinusoidal hatching.

3.4 Microstructure

Figure 8 shows the microstructure of the specimens with reference to the build plane, which was revealed after electrolytic etching for 30 s at 0.3 A/cm² with a 50 % HNO₃ solution. The etching also revealed the meltpools and their behaviour, but only in the unreinforced 316L specimens. Fine columnar grain bands observed from the specimen of Figure 8a were abruptly terminated at meltpool boundaries. Hence, it is possible that they have grown oriented towards the build direction. On the other hand, specimens of Figure 8a-c contained more complex columnar structures. Internal structure of columnar grains consists of colonies of cells [59]. In which, depending on growth direction, cells were either equiaxed or elongated [60]. This cellular structure is better observed in the high magnification micrographs of Figure 8, and they were formed because of complex heat transfer and large temperature gradients in the meltpool inherited from the ultrafast melting and solidification rates during laser processing. The highest temperature gradient is located on the bottom surface of concave shaped meltpools. Where, solidification of cellular structures starts perpendicular from the boundaries towards the centre of meltpools. Hence, cells are most likely to be oriented with its longitudinal direction parallel to the build direction. Whereas cells formed near the top of the meltpool boundaries can grow horizontally in response to the maximum heat flux direction. However, typically the structures formed on the top of meltpools are remelted during the fusion of the consecutive layer. Fluid dynamics of the meltpool driven by gas expansion and material evaporation, resulting from the recoil pressure [61] and the Marangoni flow effect on heat and mass transfer [62], can alter the heat flux direction. This is one of the reasons for the seen horizontal and arbitrary oriented colonies of cells. An example of a such complex growth behaviour was also observed on discontinuous meltpools, Figure 8a. Interestingly, colonies near the meltpool tip are oriented radially perpendicular to the tip planar boundary. A similar growth behaviour was also observed in Pham et al. single track study [63]. Therefore, it can be confirmed that the cells growth axis is closely perpendicular to the solid-liquid interface due to higher local thermal gradients and the maximum heat flux [64,65].

As illustrated in Figure 8b, the rapid rastering of the laser beam trough the sinusoidal path led to the formation of meltpools which have a "stack of dimes" type of look resembling weave bead patterns sometimes seen in metal welding [66-68], where each semicircle corresponds to a dab of filler formed during the welding [69]. The seen overlapping dabs resulted, therefore, from a dabber mode formation of material tracks. Factors such as laser, scanning and layer parameters and physical properties of the molten material such as surface tension and viscosity contribute to the spacing of the dabs. In contrast with the continuous mode, this interrupted action allowed for a faster cooling causing grain refinement and strong vertical texture. However, some grains persisted to go through multiple dabs, implying that the dabber mode does not completely prevent grains from elongating. Nevertheless, the colonies seen in this specimen are several orders of magnitude smaller than those of Figure 8a. The nonhomogeneous size of the refined structures seen in Figure 8c and 8Figure 8d resulted from the addition of SiC to 316L. Where, the degree of constitutional undercooling set by the composite composition, the distribution of SiC and thermal profiles in the liquid were associated with this phenomenon [70-72]. Therefore, based on the unreinforced specimens, here, those colonies decorated with slightly large structures correspond to a reinforcing phase. The observed porosity is probably associated with typical L-PBF defects, 316L, SiC and contaminants [73,74]. Therefore, the causes of porosity formation were either from entrapped gases (or absorption of surrounding gases), evaporation of certain elements, meltpool instabilities, shrinkage during solidification or other influencing factors such as powder humidity and oxygen content in the processing chamber. It is worth noting that pores were only found between grains, and where they resided there was a lack of grain boundary. Generally, solidification crack occurs along the grain boundaries and precipitated phases, aided by existing internal defects. Here, the presence of porosities made the specimens less ductile and offered an alternative pathway for crack to propagate. Cracking along cellular boundary is convoluted and often consumes more energy. In accordance with a previous study [75], fewer cracks were observed when using a relative high scanning speed. The 10000 mm/s scanning speed reduced the solidification time, which constrained aggregation of nano SiC particles and consequently reduced the degree of cracks. However, based on the density results of Figure 4, the increase in scanning speed correlated with an increase in porosity. Nevertheless, the tensile results of Figure 6 confirmed that higher scanning speed reflects on improvements in composite tensile strength and ductility.

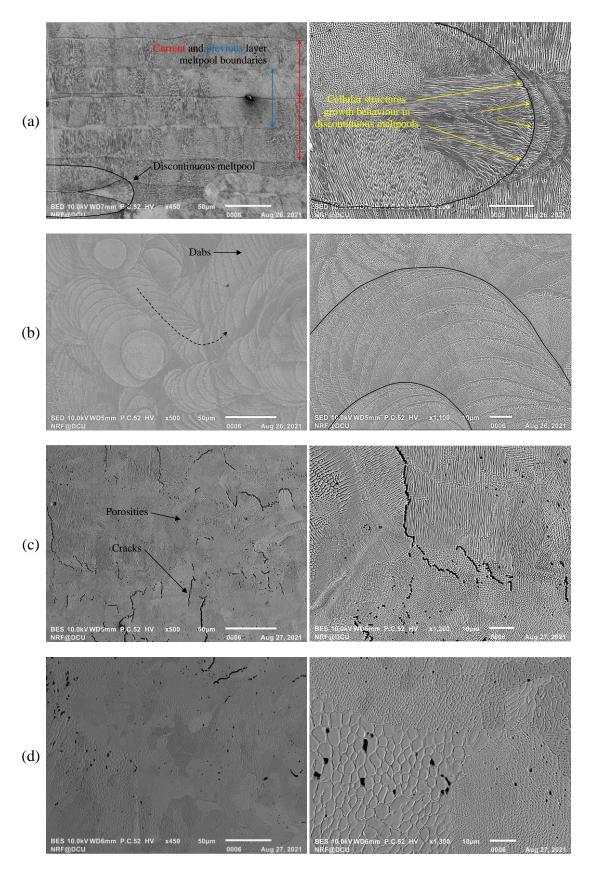


Figure 8 Microstructure of the specimen (a) 316L stripe hatching, (b) 316L sinusoidal hatching, (c) 316L-SiC stripe hatching and (d) 316L-SiC sinusoidal hatching.

3.5 Nanotribological Properties

The performance of metal matrix composite components such as engine pistons, valves and gas turbine blades also depends on the asperities, friction, wear, corrosion and lubricant properties of theirs exposed or contact surfaces with other metals, fluids and gases [76,77]. The surface of stainless steel is chemically reactive forming oxide layers in air and in other environments (i.e. chlorides, sulphides and nitrides). Some oxides are very tenacious, and the surface become passivated with no further oxidation. However, others like Fe₂O₃ can continue to grow in a humid air environment. Typically, the oxide layer ranges from 1 to 10 nm, which is established within a few minutes of exposure of atomic clean surface. Tribological oxidation is said to reduce the shear strength of the interface, which reduces wear and lower friction, and may effectively separate two contact surfaces [78]. However, at a high load and temperature, the oxide film may be penetrated. Then, transition to a high friction and wear occurs. Therefore, to develop fundamental understanding of the friction and wear properties of the naturally formed oxide films on the surface of the specimens, the nanoscratching technique was used.

In the process of nanoscratching, vertical and tangential forces are involved in the deformation of surfaces. As the relationship between these two forces is not governed by the Coulomb law of friction, other influencing factors must also be considered. Bowden et al. [79] proposed that friction resulted from the sum of two components: (i) a physical component which is the adhesion force required to shear the contacting junctions for sliding to occur; and (ii) the mechanical component due to the plastic deformation of the contacting surface arising from the ploughing. According to the Hertzian elastic contact theory [80] and the Bowden et al. [79] model of adhesive friction force, the adhesive friction coefficient becomes:

$$\mu_a = \tau_s . \pi . \left(\frac{3r}{4E_r}\right)^{\frac{2}{3}} . F_n^{\frac{-1}{3}}$$
 (1)

where r is the tip radius, E_r the reduced modulus F_n the normal force and τ_s the interfacial shear strength [79]. Bowden et al [79] as well as Goddard et al. [81] proposed expressions for the ploughing friction coefficient. However, both expressions assumed that the contact area is a half disc, and their expressions were not corrected for elastic-plastic or viscoelastic contact [82–85]. Subsequently, Lafaye et al. [86] incorporate into the Goddard et al. [81] expression a term, which takes into account elastic recovery, assuming a real tip that is conical and truncated by a spherical cap at the extremity. Therefore, the equation, which allows to evaluate the ploughing friction coefficient is:

$$\mu_p = \frac{2}{a^2} \frac{\beta^2 \sin^{-1} \left(\frac{a \cos w}{\beta} \right) - a \cos w \sqrt{r^2 - a^2}}{\pi + 2w + \sin 2w}$$
 (2)

where h the scratch depth and H the specimen hardness. The contact radius a, a fictive radius of the tip β and the angle made by the contact at the back of the sliding tip ω were calculated according to the geometrical expressions of [87,88].

$$\begin{cases} a = \sqrt{2hr - h^2} \\ \beta = \sqrt{r^2 - a^2 \sin^2 w} \end{cases}$$

$$w = \sin^{-1} \sqrt{\frac{2H(r - h)}{aE_r}}$$
(3)

The nanoscratch topography and cross section profile of the specimens are respectively shown in Figure 9. Higher penetration depths were achieved on the unreinforced specimens. Also, minimal, but noticeable variations in depths resulted from the scanning strategy and its respective process parameters (compare: Figure 9a against Figure 9b and Figure 9c against Figure 9d). The scratch cross section profile of the composite specimens is v-shaped perfectly symmetrical mirroring the profile of the tip. In conjunction with their lower penetration depths, this suggests that the existing films are possibly of low plasticity. Differently, the seen apparent smaller widths and the convex walls from the depth cross section profile of the unreinforced specimens are evidences of elastic recovery (occurring behind the scratch-tip) on the films.

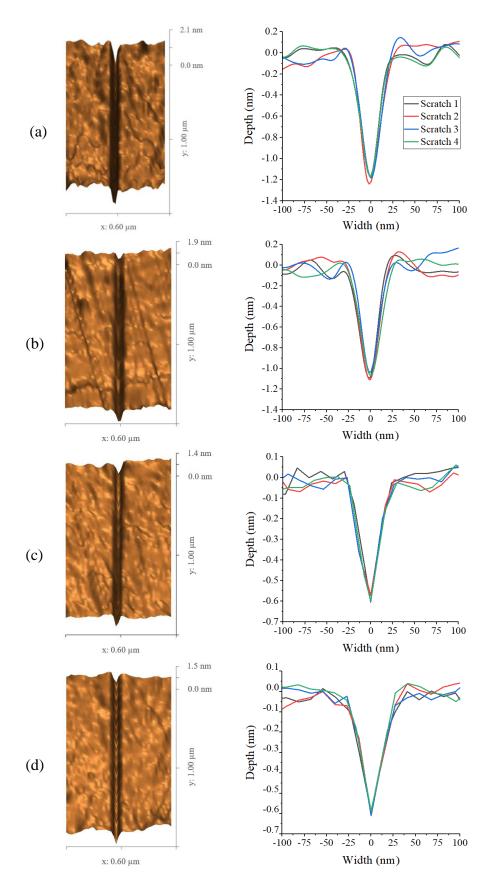


Figure 9 Atomic force microscopy scratches and the scratches cross section profiles of the specimen (a) 316L stripe hatching, (b) 316L sinusoidal hatching, (c) 316L-SiC stripe hatching and (d) 316L-SiC sinusoidal hatching.

The friction coefficient and specific wear rate of the specimens (wear volume per unit applied normal force per unit scratching distance) obtained from the nanoscratched surfaces are listed in Table 2. It is of interest to note the interrelationship of friction with wear. Specially, the disparities in friction coefficient and specific wear rate between the unreinforced and reinforced specimens. Which suggests that their films differ in physical and mechanical properties as well as chemistry. It is well known that 316L is a high corrosion resistant alloy due to its naturally formed protective passive film. Species of Fe (i.e. FeO₂ and Fe₃O₄), Cr (i.e. Cr₂O₃ and CrO₂), Mo (i.e. MoO₂ and MoO₃) and Ni (i.e. Ni(OH)₂ and NiO₂) formed from reactions with intrinsic elements (i.e. O, H, C, N), other metallic elements and impurities (i.e. sulphides and phosphides) are typically found within oxide films [89–96]. In fact, an oxide film formed on the surface of 316L can be composed of three layers: an inner layer of metallic Nickel at trace amount (if present), an intermediate layer of Fe species (existence of Cr species is also possible) and an outer layer of Fe and Mo species [96–100]. Unfortunately, if the oxide film exceeds a critical thickness, it can act as an abrasive during its contact with other surfaces, but if the thickness is less than a critical limit, then the oxide acts as a protective tribological film [101,102]. Additionally, when the hardness of the oxide is close to that of the bulk material and the oxide film thickness is of the order of 10 nm, the film will adhere well to the surface below and hence acts as a solid lubricant [103]. However, oxide films are susceptible to fretting as mechanical friction may destroy them more rapidly than they can grow [104,105], therefore introducing depassivation which will have a direct impact on corrosion, wear and friction.

Table 2 Nanoscratch friction coefficient and specific wear rate of the specimens. The data bounds noted are 95% confidence intervals.

Specimen	Friction Coefficient	Specific Wear Rate x10 ⁻¹² (m ³ /N.m)
316L stripe hatching	$0.239_{\pm 0.0074}$	$30.28_{\pm0.85}$
316L sinusoidal hatching	$0.224 {\scriptstyle \pm 0.0084}$	$26.35 \scriptstyle{\pm 0.81}$
316L-SiC stripe hatching	$0.149_{\pm 0.0063}$	$15.39_{\pm0.71}$
316L-SiC sinusoidal hatching	$0.150_{\pm 0.0064}$	$16.49_{\pm 0.53}$

To gain further understanding on the formed oxide films, deconvolution of their mechanical properties were investigated via in-situ nanoindentation. Graphs illustrating the evolution of nanohardness and reduced modulus within the oxide films are available in Appendix A. At 2 nm depth into the oxide films, both the nanohardness and reduced modulus were found to be much higher in the composites than in the unreinforced specimens. Also, it is worth noting that at this depth the nanohardness follows an expected trend reflecting on the specific wear rate values of Table 2. The depth into the oxide film which the nanohardness was closely related to the intrinsic nanohardness of the bulk material was: 20 nm for the 316L sinusoidal hatching, 30 nm for the 316L stripe hatching and 70 nm for the composite specimens. Therefore, this suggests the existence of a thicker oxide film on the composite specimens. Here, both the nanohardness and the reduced modulus of the films increased with the growing indentation depth. In fact, such a trend is characteristic of many oxides [106–108]. Since a more in-depth investigation

is beyond the scope of this study, it can be speculated that discrepancies among the reported properties of oxide films are reasonably explained by the various growth environments.

3.6 Crystallography

The crystalline phase composition present in the specimens was evaluated by means of x-ray diffraction. Figure 10 confirms a fully austenitic structure on the 316L specimen printed using the stripe hatching. Apparently, the sinusoidal hatching led to a different heating and cooling rate in the meltpool, which failed to fully prevent the suppression of solute redistribution and resulted in a portion of the austenitic phase transforming into Fe₂Si and CrSi as confirmed by the JCPDS cards 83-1259 and 65-3298. An evident diffraction peak of SiC was observed in the composites spectrum and confirmed by the JCPDS card 89-1396. The presence of SiC in the microstructure suggests that complete dissolution of SiC particles was avoided under the used set of processing parameters and inherent rapid cooling rates of L-PBF. However, at a temperature of 883 K decomposition of SiC into Si and C atoms began, and according to the Fe-Si binary phase diagram, the phase transformation temperature decrease with increasing content of Si [30,109,110]. Therefore, Si atoms could have diffused into the lattice of molten Fe and consequently de-stabilise the austenite. In this regard, it is clear that displaced Si from SiC reacted with the matrix and contributed to the precipitation of silicide phases.

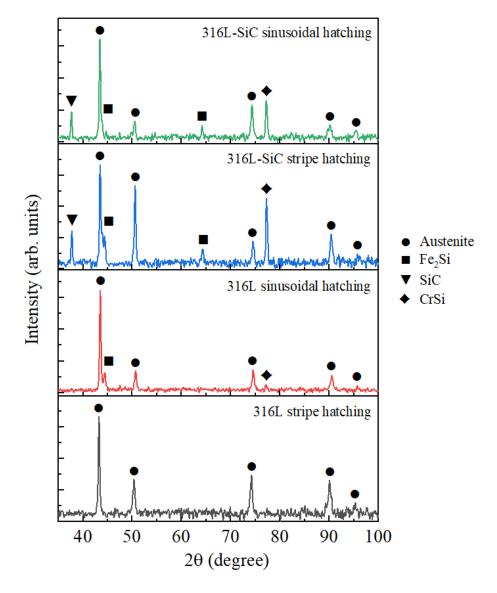


Figure 10 X-ray diffraction patterns of the printed specimens.

Electron backscatter diffraction grain orientation maps normal to the build direction and corresponding discrete pole figures of the austenitic phase are presented in Figure 11. It is observed that the specimens printed in this study are characterised by different crystallographic texture. A moderate {100} and {111} texture with preferable [101] and [001] grain growth was revealed in the pristine (unreinforced) 316L stripe hatching. In contrast, both composite specimens showed similar but a less strong texture, spread rather towards the {100} crystallographic family of planes. Nevertheless, the 316L-SiC sinusoidal hatching specimen presented a distinct fraction of [001] oriented grains. SiC and precipitated silicide phases had a weak contribution to the specimen's crystallographic texture. It was observed for SiC and CrSi a texture incline to the {100} pole, especially for SiC with orientation parallel to the hatching direction. In both printed composites, it is also observed that SiC grains were preferably oriented between [001] and [101]. On the other hand, grains of Fe₂Si were predominantly [210] and [120] oriented in the specimen 316L sinusoidal hatching, while the Fe₂Si phase present in the composites showed no clear contribution to texture. Supplementary pole figures are available in Figure B1 of Appendix B.

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The electron backscatter diffraction analysis also showed that the total contribution of silicide phases to each specimen (316L sinusoidal hatching, 316L-SiC stripe hatching and 316L-SiC sinusoidal hatching) was less than 1 % (from this, approximately ¼ being of CrSi). As expected, most of these precipitated silicides (Fe₂Si and CrSi) were distributed along the grain boundaries of the austenite phase. This is because at grain boundaries the concentration of vacancies tends to be high, thus permitting easy diffusion of atoms. Additionally, as grain boundaries are potent nucleation sites, precipitating particles may have removed sufficient solute from the adjacent matrix such that the region in the proximity of the grain boundary remained free of precipitates [111–113].

There is clear evidence that both the addition of SiC and the used sinusoidal hatching promoted grain refinement. Large austenite grains underwent approximately 10 μm reduction in response to the sinusoidal hatching, bringing the largest size down to 55 μm in the unreinforced specimen and down to 36 μm in the composite specimen. The sinusoidal hatching system also impacted on the size distribution range of the remaining grains, for example in the unreinforced specimens it was reduced from 1-43 μm to 2-27 μm range. The 316L-SiC sinusoidal hatching presented the most refined microstructure of the four specimens, and this is supported by its low average 20 μm grain size, narrower grain size distribution, and lower as well as more homogeneous area fraction grain size. Electron backscatter diffraction grain maps and grain size distribution plots are available in Figure B2 of Appendix B.

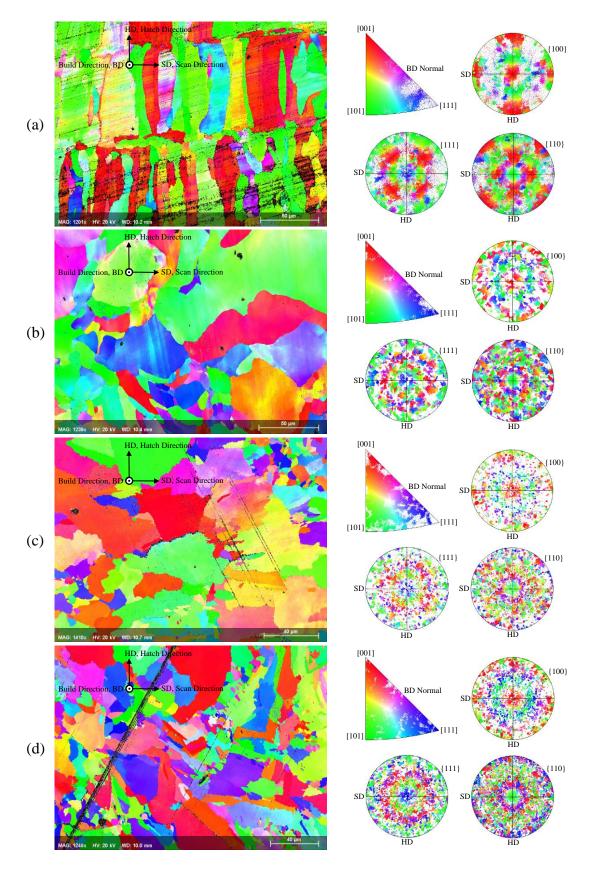


Figure 11 Electron backscatter diffraction discrete pole figures and inverse pole figures for (a) 316L stripe hatching, (b) 316L sinusoidal hatching, (c) 316L-SiC stripe hatching and (d) 316L-SiC sinusoidal hatching.

3.7 Chemical Analysis and Phase Mapping

Figure 12 illustrates secondary electron micrographs with corresponding overlaid energy dispersive x-ray spectroscopy mapping of silicon and carbon obtained from each of the specimens. Figure 12 also contrasts the electron backscatter diffraction phase map of the specimens. The observed elemental concentration increases from Figure 12a and 12b to Figure 12c and 12d is in agreement with the incorporated 1 wt.% SiC. The dispersion of silicon and carbon in the unreinforced specimens was apparently uniform, whereas in the reinforced specimens several carbon alignments were observed. The obtained electron backscatter diffraction phase constitution agrees with the x-ray diffraction results of Figure 10. However, as illustrated in Figure 12, SiC is the dominant reinforcing phase in the printed composites. Similarly to conventionally manufactured 316L-SiC composites [47,114], the composite printed with the stripe hatching system exhibited the aggregation of SiC grains, see the phase map in Figure 12c. On the other hand, the sinusoidal hatching system avoided the formation of large aggregates and showed an improved SiC dispersion in the 316L matrix. The impact of this can be related to the observed lesser microstructural cracking defects (Figure 8d) and overall refined microstructure (Figure B1d) when compared to the 316L-SiC stripe hatched specimen.

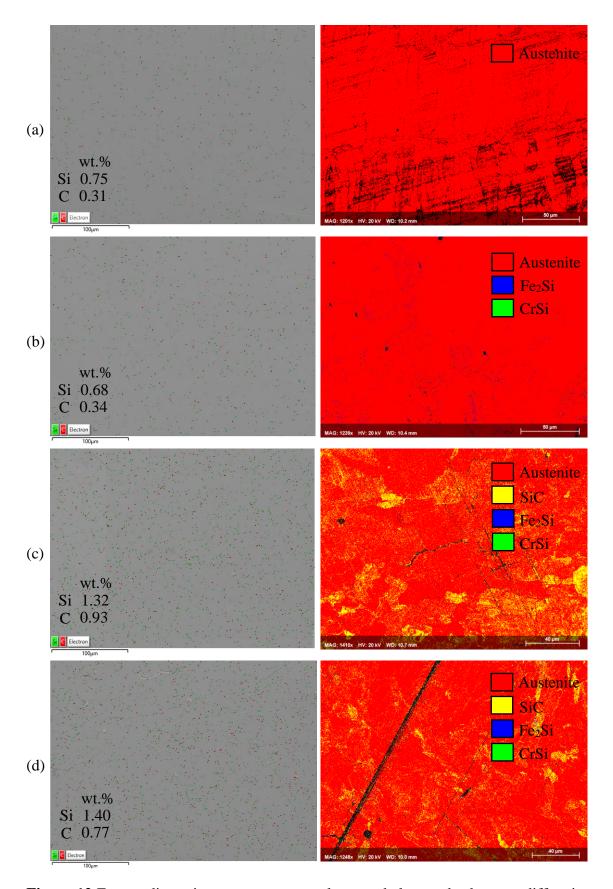


Figure 12 Energy dispersive spectroscopy analyses and electron backscatter diffraction phase map of (a) 316L stripe hatching, (b) 316L sinusoidal hatching, (c) 316L-SiC stripe hatching and (d) 316L-SiC sinusoidal hatching.

4. Discussion

4.1 Physical and Mechanical Properties of the Specimens

According to the density results, all the specimens possessed a relative low density. Yet, microstructural micrographs showed no evidence of lack of fusion defects, but there is a likelihood that lack of fusion defects could be present at other cross-section depths than those analysed. On the other hand, the micrographs do reveal the existence of severe porosity (pores smaller than 5 µm in diameter) within the specimen's microstructure, and these were located specially at grain boundaries. In this case, its typically said that the presence of moisture and entrapped gas from the feedstock powder, inappropriate shielding gas flow rate/velocity and excessive turbulence in the meltpool are the frequent causes of porosity [115–118]. However, it is most likely that instead, also supported by the observed spattering from the meltpools, a high energy input was used [119,120]. Therefore, the melting mechanism was dominated by either a transition or keyhole mode. The keyhole mode represents a highly unstable regime due to typical local temporal fluctuations of evaporation and flow imbalances along the wall of the keyhole cavity [121]. Therefore, it has a large potential of forming small gas bubbles that can get trapped within their meltpool during its rapid solidification. Nevertheless, the keyhole instability can be controlled [122–125]. The reason for this study not using the conductive mode of melting was linked to its productivity limitations, and in fact the keyhole mode is much more appreciated by the industry [126].

It is well known that lesser porosity and higher densification are prerequisites for obtaining greater hardness [127,128]. Despite their density and porosity, specimens printed in this study showed outstanding hardness. It is therefore clear that addressing these two issues additional hardness improvements are possible. A more detrimental impact of porosity was evidenced from the tensile results. As pores, crack initiation sites, drove the specimens to a premature failure in addition to affecting the yield and tensile strength attributed to loss of resistance area resulting from the presence of pores. During solidification, molted metal shrinks due to thermal contraction and in L-PBF this is impeded by the previously solidified layers underneath, which then forms strong compressive stresses at the interface layer. Similarly, the cyclic thermal stress could result in crack in response to residual stress release. Yet, the inherited residual stresses were not sufficient for generating cracks at the unreinforced specimens due to the high toughness and ductility of the 316L alloy. While in the composite specimens, the coefficient of thermal expansion mismatch between the SiC and 316L let to the generation of thermal stress in the SiC phase, and along with the accumulated residual stresses, this was the driving force of crack formation. Here, crack propagation followed the networks of grain boundaries, which resistance was weakened by the presence of pores and silicides. In the printing of composites with high scanning speeds such as that used for the sinusoidal hatching system, very high heating and cooling rates exists. Even though cracks were present in the composites printed with both low and high scanning speeds, it could be expected that the high scanning speed would introduce higher thermal shock and stress, which would then promote and accelerate crack initiation and propagation. However, this was not confirmed in this study. Here, a promising solution for inhibiting the initiation and propagation of crack is to attain further grain refinement to increase the microstructural barriers. Additionally, the preheating (at an optimal temperature) of the

build substrate could retard the temperature gradient and cooling rates and reduce the magnitude of shrinkage stress. Therefore, this could be an effective mean of mitigating residual stress and solidification cracking.

4.2 Strengthening Mechanisms

The presented results demonstrated that superior mechanical properties were obtained from the SiC reinforced 316L composites. The enhanced strength can be explained by the role of nano SiC particulates, which activate multiple strengthening mechanisms. SiC particulates can be located both intergranularly and transgranularly within the 316L matrix. In the case of L-PBF 316L-SiC composites, several have reported the preferential distribution of SiC to grain boundaries [23,129,130], and this was assumed to be responsible for the inhibition of grain growth confirmed by Figure B2. Therefore, the composites strengthening can be explained by grain refinement according to the Hall-Petch effect and grain boundary strengthening. The addition of SiC to 316L also caused the precipitation of Fe₂Si and CrSi, and these precipitates are known for their strengthening effect [26,47,131]. The resulting precipitates confirmed the decomposition SiC into Si and C atoms. Therefore, the migration of C (and Si) into the austenitic matrix could possibly have taken place and so introduced solid solution strengthening effects. Additionally, partially the dispersed SiC nanoparticles could also have been retained and promoted heterogeneous nucleation during the solidification process, hence playing a role of dispersion strengthening. Therefore, the fine closely spaced hard SiC (and Fe₂Si and CrSi) particles at the austenite grain boundaries could account for dislocations pinning (Orowan strengthening [132,133]) and contribute to the strengthening of the 316L-SiC composites.

4.3 Tribological and Mechanical Properties of the Naturally Formed Oxide Films

In MMCs, understanding the nanotribology occurring at the interface of two contacting surfaces during their relative motion is necessary to develop fundamental understanding to many technological problems, including wear, friction and lubrication occurring at the nanoscale. Also, the importance of this is that it can lead to understanding tribology on the microscale. Adding up to the discussions accompanying the results, it is observed that despite having a much thicker oxide film, the composite specimens showed almost twofold reduction in friction coefficient and specific wear rate. In fact, the in-situ nanoindentation support these results with its reported nanohardness and reduced modulus. However, it should be noted that this is specifically for the 2 nm depth into the oxide film. The analysis made on the evolution of nanohardness and reduced modulus suggested that further into the oxide film, at a specific depth, different trend for friction and wear to that of Table 2 could exist. Ultimately, the influence of the naturally formed oxide films on the nanotribological response was quite different. Mechanically softer oxide layers exhibited an elastoplastic deformation behavior, eventually leading to higher wear. In contrast, harder and stiffer oxide layers led to a reduction in wear and friction. The broad implication of the presented study is that tuning the properties and characteristics of oxide films can therefore be beneficial for many applications to improve tribological performance.

4.4 Resulting Crystallographic Textures

Typically, the highest temperature gradient is located at the bottom of the meltpool and decreases nonlinearly towards the meltpool upper surface. Therefore, in response to the maximum heat flow, columnar grains grow predominantly parallel to the build direction. However, the results suggested that several grains also grew closely align with the hatching direction in the 316L stripe hatching specimen and in the remaining specimens rather closely aligned with the scanning direction. This is most likely consequential to the adopted keyhole mode of melting as high meltpool temperatures can lead to less aligned crystal growth with the build direction [134] due to the keyhole's meltpool promotion of multiple heat dissipating directions in response to the presence of irregular meltpool boundary morphologies. Therefore, the aforementioned along with the used processing parameters explain the absence of strong crystallographic textures.

The stripe hatching is the simplest and most commonly used hatching system in L-PBF. Additionally, several authors have reported it to develop strong <001> columnar grain alignments parallel to the build direction [61,135–138]. Instead, complex hatching systems such as islands [139–145] and fractals [38] have a more profound influence on grain morphologies and development of crystallographic textures. Accordingly, it was demonstrated in [63] [146] [147] that it is possible to control grain orientation as per hatching patterns. Therefore, this suggests that it is possible to grow grains with the sinusoidal hatching system which form sinusoidal patterns. With a such achievement, it would be interesting to verify the implications of sinusoidally oriented grains on the ductility of MMCs when applying tension along the hatching.

The existence of intragranular cellular structures in L-PBF 316L has been reported in a number of studies [148–157]. From these, it is worth noting that neighboring cellular colonies typically are misoriented with each other. As a matter of fact, the resulting misorientation obtained with the stripe hatching was consistent with these reports. However, the sinusoidal hatching system led to the development of cellular colonies highly oriented with the build direction. As contrasted in Figure 13, the rastering of the laser at 10000 mm/s along the sinusoidal path caused periodic oscillation of the molten along the scanning vector hence resulting in the solidification of short spaced overlapping dabs which influenced the orientation of cellular structures. In this respect, observations confirmed that the maximum temperature gradient controlling the cellular growth direction was vertically, and this being consistent between colonies. Reminding that a colony of cells is a group of cellular structures with the same morphological orientation and cell spacing.

One of the implications of having misoriented rather than highly oriented colonies of cellular structures is on the tensile performance of specimens. As shown by [157], tension perpendicular to the cells axis caused less deformation in response to accumulation of nano voids in the cellular microstructure, and additional stress then enlarged the nano void sizes breaking cellular boundaries. Under the same conditions, cells which tension was applied along their long axis were void free and the stress was relieved by cell deformation. In both scenarios, cells could only deform to a certain limit until the high tensile stress teared them apart. Therefore, based on these findings and as seen from the comparison of the tensile performance between the composites, the sinusoidal hatching

can be used as a mean of collectively improving tensile strength and ductility. Additionally, the cellular microstructure resulting from the sinusoidal hatching could be used towards increasing composites toughness by deviating crack direction to longer paths. Even though both composites showed similar outstanding hardnesses, owing mainly to the barriers of dislocation motions imposed by cellular structures, it was observed that the 316L-SiC stripe hatching composite had a slightly higher hardness. Clearly, this is most likely to be related to the randomisation of the cellular colonies orientations which could potentially induce higher densities of entanglement dislocations. However, the colonies in the 316L-SiC sinusoidal hatching specimen were highly oriented and the reported hardness for this specimen was measured from the transversal axis of the cellular structures. This denotes that the hardness of this specimen with reference to the longitudinal axis of the cellular structures could be different.

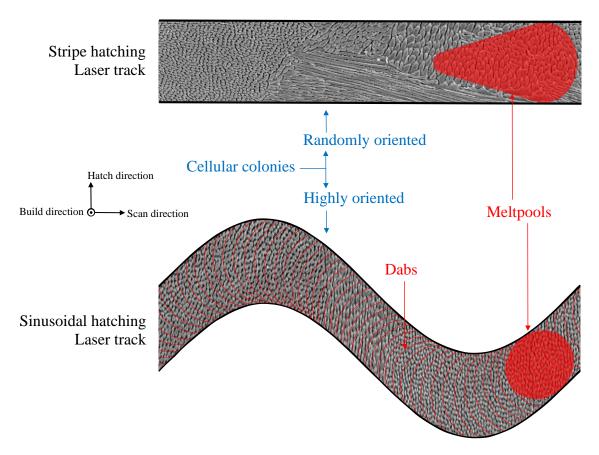


Figure 13 Schematic representation of the hatching systems and their resultant cellular structures.

4.5 Assessment of the Sinusoidal Hatching System

The sinusoidal hatching emerged as an important hatching system for L-PBF of MMCs. It proved to be capable of contributing to improvements in hardness, strength and ductility in composites. However, the existing gas porosity and solidification cracking within the composite microstructure prevented the exploitation of its full potential, therefore further research is recommended. Contrasting with the stripe hatching, over sixteenfold increase in scanning speed and over threefold reduction in laser power was successfully implemented in the sinusoidal hatching. Therefore, besides improving

performance of composites, the sinusoidal hatching also helps to address the manufacturing lead time and process energy consumption issues concerning the L-PBF process [158,3].

The mechanisms which capacitated the sinusoidal hatching to promote suitable melting with such low laser power and extremely high scanning speed is speculated below. With the used scanning speed, the scanning of consecutive neighboring hatching paths was ultrafast. Therefore, the rate of heat flowing way from solidified zones because of argon flow convection and thermal radiation was reduced. Additionally, the sinusoidal profile of the laser paths is liked to have trapped pockets of hot argon and so reduced the efficiency of the gas flow as a cooling provider. Despite not using preheating, it was observed at the end of the printing job that the build substrate was approximately at 100 °C. Apparently, this temperature stabilised during the printing of the support structures in response to the argon flow and heat dissipation and transfer to the printer components. Therefore, it is clear that a much higher temperature than this in solidified neighboring hatching paths existed. In this respect, the stored thermal energy reflects well with the applied laser power. Another observation is the reduction of the meltpool width in contrast with the stripe hatching. Nonetheless, the width was very consistent along the sinusoidal paths and in fact it was found no clear evidence of lack of fusion defects.

5 Conclusions

A novel hatching strategy here named sinusoidal hatching was developed for L-PBF of MMCs. The presented investigational results provide a deep understanding regarding the effect of the sinusoidal hatching on the microstructure and mechanical properties. Comparison with the conventional stripe hatched composite shows that an increase in yield strength and ductility can be achieved by implementing the sinusoidal hatching. The tensile properties of the composites were compromised by the existing porosities and cracks which caused premature fractures and failure under the tensile loads. Their existence was attributed to trapped gas bubbles resulting from an unstable keyhole mode of melting and thermal expansion mismatch between the matrix and reinforcement along with accumulated residual stresses. Fortunately, these defects were less detrimental to the hardness. Confirmed by the literature, both composites showed an outstanding hardness. Yet, a slightly higher hardness was measured from the stripe hatched composite which was attributed to the randomised directional growth of cellular structures.

Friction and wear properties of the naturally formed oxide films and deconvolution of their mechanical properties were also investigated in this study. At approximately 1 nm depth into the oxide film, the composites already presented low plasticity, in contrast, elastic recovery was evidenced in the matrix specimens. As a matter of fact, these were consistent with the films nanohardness and reduced modulus, which also correlate with the twofold reduction in friction coefficient and specific wear rate measured from the composites. However, the depth into the oxide film which the nanohardness was closely related to the intrinsic nanohardness of the bulk material was 70 nm for the composites and 20 nm and 30 nm for the unreinforced stripe and sinusoidal hatched specimens. Additionally, the evolution of nanohardness and reduced modulus showed that further into the oxide film, at a specific depth, different trend for friction and wear to that reported could exist. Ultimately, mechanically softer oxide layers exhibited an elastoplastic

deformation behavior, eventually leading to higher wear. In contrast, harder and stiffer oxide layers led to a reduction in wear and friction. The broad implication of the presented study is that tuning the properties and characteristics of oxide films can therefore be beneficial for many applications in order to improve tribological performance.

The sinusoidal hatched composite exhibited a highly refined columnar microstructure having a moderate {100} texture as most of the grains solidified preferentially in the <001> direction with respect to the build direction. A unique molten flow and solidification condition was stablished by the sinusoidal hatching. As periodic oscillation of the molten along the scanning vector resulted in the solidification of short spaced overlapping dabs which formed small colonies of intragranular cellular structures with cells highly oriented with the build direction. The implications of this reflected on the obtained improvements in yield strength and ductility seen from the sinusoidal hatched composite. It was speculated that, apart from tailoring hardness, the sinusoidal hatching could be used towards increasing composites toughness by deviating crack direction to longer paths. Besides improving composite performance, the sinusoidal hatching also reduced manufacturing lead time and process energy consumption.

The bulk mechanical properties of the composites were negatively affected by the existing porosities and cracks. Future research should therefore identify the source of theses defects and apply mitigation measures to eradicate them from the composite's microstructure. In-situ process monitoring of temperature and melt flow as well as build substrate preheating for mitigation of crack formation and residual stress are recommended.

The current study helps to pave the path towards addressing several endemic issues concerning mechanical performance and cost effectiveness of MMCs. As there is an increasing demand for materials with supernormal performance, the sinusoidal hatching is worth further research.

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Appendix A

Nanoindentation on the oxide film of the specimens was performed to support the nanoscratch study, and to measure the depth into the oxide film which the nanohardness is closely related to the nanohardness of the bulk material. The existence of surface

defects, grain boundaries and phases affect the oxide film physical characteristics and chemistry. Here, each presented result is the average of six indents (2x3 matrix, 30µm spacing) obtained for the applied nanoindentation experimental design. The tip (a Berkovich diamond indenter) geometry and radius was calibrated with a fused quartz standard of known properties by running a series of tests that spans the load range of the instrument for area function determination and establishment of the compliance correction factor, both to be later utilised by the instrument software. Then, in-situ indentation mode of operation was chosen as it offered drift compensation capabilities for maximum accuracy. Prior performing each indent, a drift settle time of 300 s was applied while the tip waited on the specimen's surface in feedback prior to begin the drift correction procedure. This was to enable time for the motors and piezos to settle down. Then, the indentation procedures only began when the overall drift rate including thermal drift was within ± 0.05 nm/s. All indentations were performed at room temperature. Figure A1 shows the evolution of the nanohardness within the oxide film and Figure A2 shows the evolution of the reduced modulus within the oxide film. The nanoindentation loadunload behavior of the 2, 10, 20, 30, 40, 50, 60, 70 nm depths into the oxide film is illustrated in Figure A3 and Figure A4.

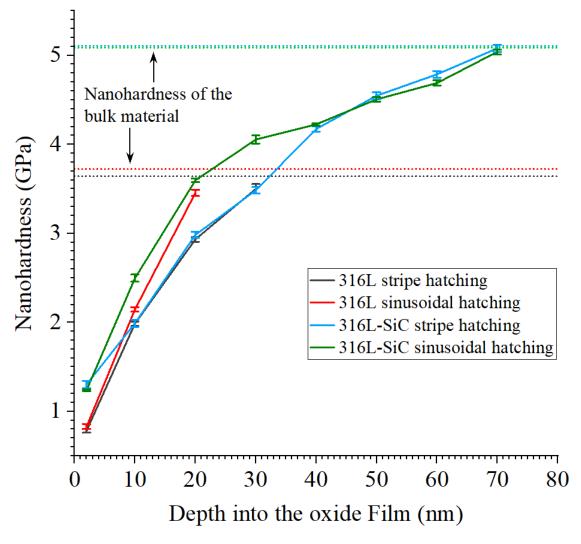


Figure A1 Evolution of the nanohardness within the oxide film. The data whiskers represent the 95% confidence interval.

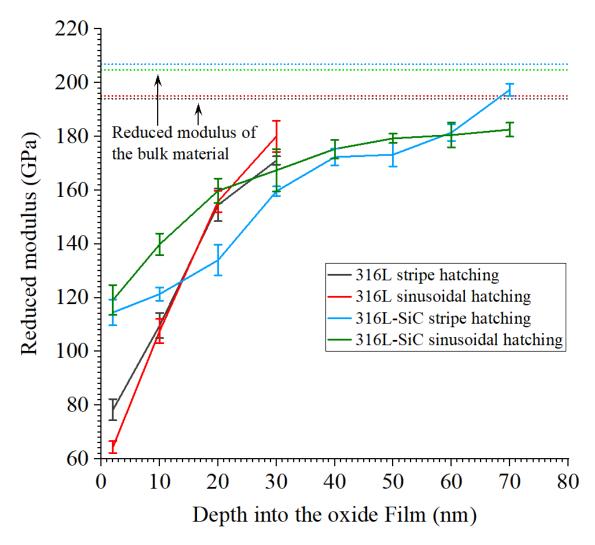


Figure A2 Evolution of the reduced modulus within the oxide film. The data whiskers represent the 95% confidence interval.

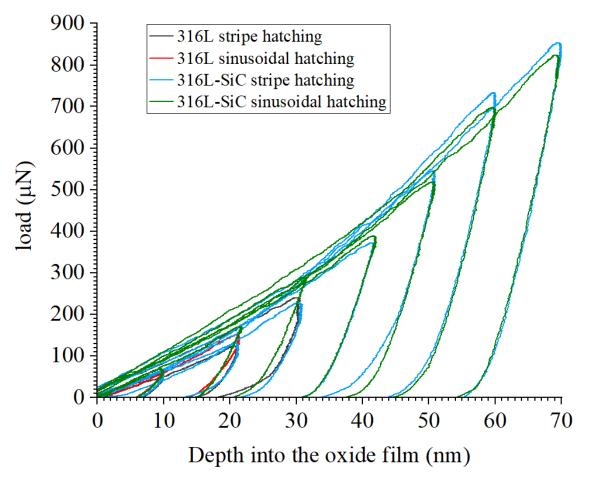


Figure A3 Nanoindentation load-unload behaviour of the 2, 10, 20, 30, 40, 50, 60, 70 nm depths into the oxide film.

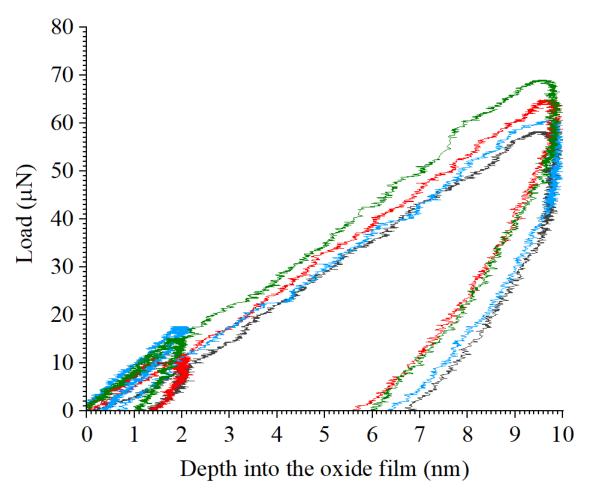


Figure A4 Supporting plot for Figure A3.

Appendix B

Inverse pole figures and pole figures of the precipitated silicides (Fe_2Si and CrSi) and SiC phase are available in Figure B1.

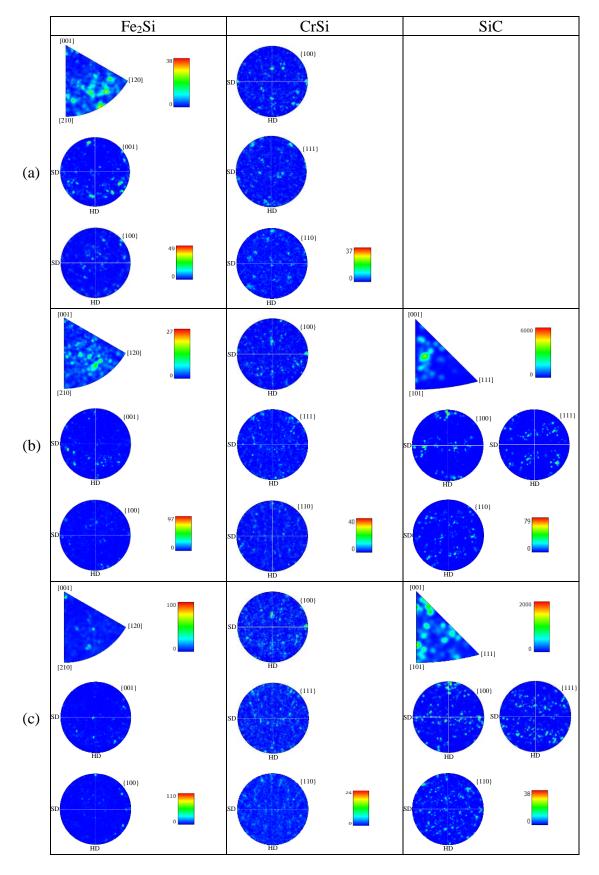


Figure B1 Additional electron backscatter diffraction build direction inverse pole figures and pole figures for (a) 316L sinusoidal hatching, (b) 316L-SiC stripe hatching and (c) 316L-SiC sinusoidal hatching.

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Electron backscatter diffraction grain map and grain size distribution plot for the printed specimens are given in Figure B2.

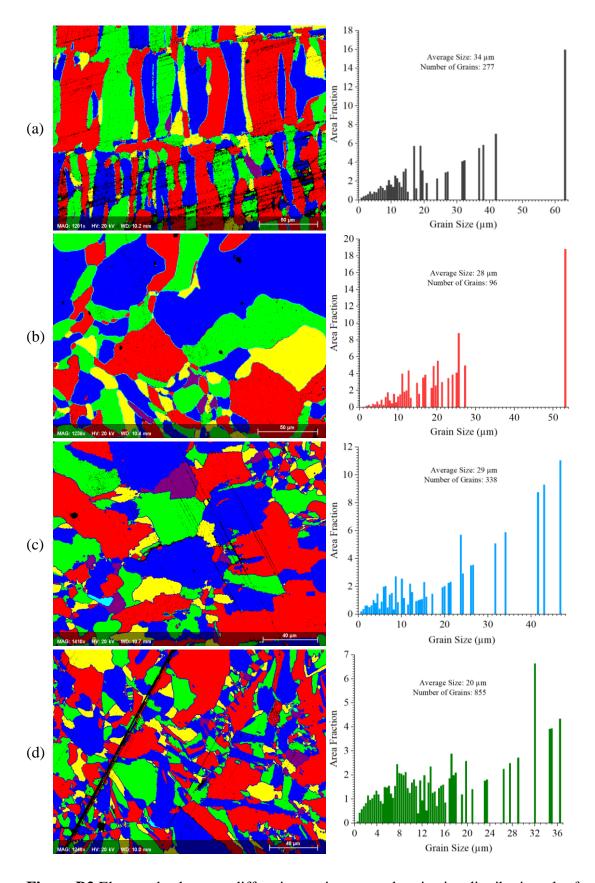


Figure B2 Electron backscatter diffraction grain map and grain size distribution plot for (a) 316L stripe hatching, (b) 316L sinusoidal hatching, (c) 316L-SiC stripe hatching and (d) 316L-SiC sinusoidal hatching.

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