Thermal behavior of the molten pool, microstructural evolution, and tribological performance during selective laser melting of TiC/316L stainless steel nanocomposites: Experimental and simulation methods

Bandar AlMangour\textsuperscript{a,⁎}, Dariusz Grzesiak\textsuperscript{b}, Jinquan Cheng\textsuperscript{c}, Yavuz Ertas\textsuperscript{d}

\textsuperscript{a} School of Engineering and Applied Sciences, Harvard University, Cambridge, MA, 02138, USA
\textsuperscript{b} Department of Mechanical Engineering and Mechatronics, West Pomeranian University of Technology, Szczecin, Aleja Piastów 17, Poland
\textsuperscript{c} Composite Solutions and Digital Manufacturing LLC, Chandler, AZ, 85248, USA
\textsuperscript{d} Department of Bioengineering, University of California, Los Angeles, CA, 90095, USA

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\section*{ABSTRACT}

Bulk-form nanocomposites of TiC-reinforced 316L stainless steel matrix were fabricated by selective laser melting (SLM), an emerging powder-bed additive manufacturing technique which allows the direct fabrication of usable end-products, using various volumetric laser energy densities (\(\eta\)). The microstructural features of the distribution and sizes of TiC nanoparticles, as well as the grain sizes and tribological performances of the SLM-processed nanocomposite parts, were sensitive to the applied \(\eta\). Increasing \(\eta\) enhanced the dispersion state of nanoscale TiC owing to intensified Marangoni flow and the corresponding capillary force, which prevented TiC aggregation and promoted a uniform dispersion of reinforcements in the solidified matrix. However, with increasing \(\eta\), the TiC particle size also increased, and some nanoparticles lost their initial nanostructure because of significant thermal accumulation within the molten pool. Increasing \(\eta\) also caused increases in the grain sizes of the fabricated nanocomposite because of the decreasing cooling rate. A simulation model was developed to enhance understanding of the manufacturability of these new materials, as well as to predict the temperature evolution and thermal behaviors of the molten pool under various \(\eta\). The simulation modeled the effects of various \(\eta\) values on the temperature distribution evolutions and the corresponding effects of Marangoni convection during the SLM process. The temperature distribution was significantly influenced by the applied \(\eta\); the maximum temperature gradient within the molten pool was increased significantly with increased \(\eta\). The simulation results validated the experimental results and the underlying physical mechanism of the molten pool. The microhardness of the SLM nanocomposite decreased sharply with increased grain size due to the lower cooling rate, but increased with further increases in \(\eta\) because of the enhanced densification degree. Nanocomposites processed under the optimum condition of \(\eta = 200 \text{ J/mm}^2\) showed the lowest wear rates accompanied by the formation of adherent and strain-hardened tribolayers on the worn surfaces of the nanocomposites, suggesting improved tribological performance.

\section*{1. Introduction}

Many material-dependent sectors, including aircraft, spacecraft, biomedical devices, and electronics, require materials with extraordinary combinations of features not found in traditional pure ceramics or metal alloys. Therefore, various composite materials have recently been developed with improved material properties. Metal alloys can be reinforced by homogenous dispersions of second-stage hard and fine ceramic particulates; when nanoscale reinforcement particles are present, the resulting materials are called nanocomposites. The strengthening mechanism involves both interparticle interactions and dislocations within the matrix. Pagounis and Lindroos (1998) demonstrated that stainless steel-matrix composites (SMCs) strengthened with stiffer and harder ceramic particles exhibited promising features, including high specific strength, specific stiffness, and wear resistance.

Numerous manufacturing methods have been reported for producing nanoparticle-reinforced metal matrix composites (MMCs). Sheikhzadeh and Sanjabi (2012) successfully synthesized MMCs by mechanical alloying, while Sahib et al. (2012) and Chen et al. (2013) effectively used powder metallurgy and stir-casting, respectively. However, Viswanathan et al. (2006) and Moya et al. (2007) indicated that obtaining a homogenous distribution of nanoscale reinforcing...
particles in the matrix can be very challenging because extensive van der Waals interactions, coupled with the large surface areas of nanoparticle reinforcements, can cause considerable microstructural inhomogeneity. Moreover, Kim et al. (2004) and Lee and Kang (2006) reported difficulties in preserving the nanostructures of loose powders during the consolidation of composites because of extreme grain growth during high-temperature processes such as casting, sintering, and hot isostatic pressing. In addition, fabricating customized functional complex parts for high-tech applications by these conventional processing methods is quite challenging. Therefore, it is important to develop a novel, non-traditional nanocomposite fabrication method that can overcome these complex processing issues.

Selective laser melting (SLM) is an emerging additive manufacturing process that provides substantial flexibility in the production of parts with complex shapes that are suitable for various end uses. Parts can be synthesized directly from loose powders that cannot be processed readily by conventional processing methods of casting and sintering, as discussed by Gu et al. (2012b). SLM allows the fabrication of high-performance MMC parts with fine grain sizes because of the extremely high solidification rates stimulated by non-equilibrium laser scanning, as reported by Das et al. (2010) and Pei et al. (2005). However, the SLM process involves multiple modes of heat, mass, and momentum transfer, and is thus, as noted by Kruth et al. (2007), a complex metallurgical process with strong Marangoni effects induced by thermal capillary forces and very high cooling rates. The arrangement of nanoparticles and dispersion state are considerably influenced by fluid flow. However, existing knowledge mostly relies on experimental results, which remain lacking; moreover, little theoretical work has been performed. Obtaining the transient temperature variation and understanding the mechanisms underlying nanoparticle movements are difficult in experimental approaches. Therefore, numerical simulation methods are necessary to analyze and understand the influence of the laser energy density on the thermal behavior and temperature distribution evolution, as well as the fluid flow driven by surface tension gradients and Marangoni convection, in the molten pool during SLM.

Few studies have reported the possibility of processing Fe-matrix and 316L stainless steel-matrix nanocomposites by SLM. Song et al. (2014a) studied the SLM behavior of SiC/Fe bulk nanocomposites formed directly from a mixed powder of micro-sized Fe and SiC, reporting that the addition of SiC to the Fe matrix caused significant increases in the local melt instability and viscosity, as well as enhanced tensile properties. Hao et al. (2009) and Wei et al. (2015) processed hydroxyapatite/stainless steel composites by SLM for medical applications and reported the microstructural characteristics, element distribution, and mechanical properties of the composites. The authors of the current study, AlMangour et al. (2016a; and 2016b), previously reported progressive improvements in the mechanical and tribological performance of 316L nanocomposites with increasing TiC and TiB2 volume contents. More recent publications by the authors, AlMangour et al. (2017 and 2018), analyzed the densification behavior, microstructural evolution, and compression properties of TiC/316L nanocomposites processed using various SLM scanning methods and energy densities. Nanoscale TiC was selected as the reinforcement based on its excellent thermodynamic stability and wettability in molten steel, as well as its high hardness and elastic modulus as reported by Abenojar et al. (2002). In this study, the thermal behaviors and physical mechanisms of the molten pool are investigated, as are the evolutions of the TiC dispersion state and particle size and the TiC/316L nanocomposite grain size during SLM at various applied energy densities. A simulation model is constructed to predict the thermal behaviors of the SLM processing. The resulting variations in the microstructures and corresponding tribological performances of the nanocomposites are then analyzed. A process–microstructure–properties correlation was clarified to facilitate the effective production of SMC components.

2. Experimental procedures and simulation setup

2.1. Powder preparation and SLM processing

In this study, 316L spherical gas-atomized powder with an average particle diameter of 45 μm (Fig. 1a) and nearly spherical TiC nanopowders with an average particle size of 50 nm were used as starting materials (Fig. 1b). Mechanical mixing of the TiC and 316L powders comprising 15 vol.% TiC was performed in a Fritsch Pulverisette 4 Vario-Planetary mill, according to the milling conditions discussed in AlMangour et al. (2016a). The milled nanocomposite powders were severely deformed, with TiC nanoparticles embedded within the 316L matrix powders (Fig. 1c).

A commercial SLM machine (MCP HEK REALIZER II) was used, consisting of a Yb-fiber laser, automatic powder layering system, inert Ar gas protection system, and a computer system for process control. Before starting the SLM process, a steel substrate was placed on the building platform and leveled; then the building chamber was sealed and filled with Ar gas to minimize oxidation (an O2 content of < 1% was used to process the feedstock powder). The main SLM processing parameters were kept constant with a laser power (P) of 100 W and a spot size of 180 μm. An alternate-hatching scanning pattern (with 90° rotations between successive layers) was applied. The powder was applied by spreading it over the building platform using a wiper. The building platform was lowered to a distance equal to the layer thickness, and the resulting space was filled with the powder. The SLM parameters of powder layer thickness (d) and scan line hatch spacing (h) were kept constant at 50 μm and 120 μm, respectively. To alter the processing conditions during processing, different scanning speeds (v) of 250, 133, 83, and 55.6 mm/s were set using the SLM control program. Thus, four varying volumetric laser energy densities of \( \eta = \frac{P}{v \cdot d} \) , defined by Gu et al. (2012a) as:

\[
\eta = \frac{P}{v \cdot d}
\]

were used to evaluate the laser energy input to the powder layer being processed.

2.2. Microstructural characterization

Specimens for microstructural characterization were ground and
polished in accordance with standard metallographic practices, and then etched using Marble’s reagent for 10 s. The relative densities of the SLM-processed nanocomposites were estimated based on the Archimedes principle, and the average value of three readings was tabulated for each set of processing conditions. The microstructure of the SLM-processed nanocomposite samples was examined at their cross section (i.e., parallel to the building direction) using a Nano 230 scanning electron microscope (SEM). An FEI T12 transmission electron microscope (TEM) at 120 kV was used to examine the internal microstructures of the fabricated nanocomposites. TEM samples were prepared via focused-ion beam (FIB) milling. The grain orientations of the specimens were characterized using electron backscatter diffraction (EBSD) at 15 kV using an FEI Quanta 600F SEM with a step size of 40 nm at low magnification to collect broader statistical data. The specimens were polished using a diamond suspension and colloidal silica (100-μm step sizes to 0.01 μm) for approximately 8 h prior to the EBSD measurements.

2.3. Modeling approach and numerical simulation

2.3.1. Physical model description and governing equations

A layerwise additive manufacturing model using the relevant Layerwise Additive Manufacturing Predictions and Simulations (LAMPS) software was used to analyze the full-scale temperature field response and quantitatively explain the experimental results. The full-scale layerwise AM model is based on the governing behavior of conductive heat transfer in a laminated structure and the track-by-track and layerwise deposition of AM, as schematically shown in Fig. 2. Here, the heat transfer-governing equation for each layer in the laminated plate can be described by

\[
\frac{\partial}{\partial x} \left( K \frac{\partial T(x, y, z, t)}{\partial x} \right) + \frac{\partial}{\partial y} \left( K \frac{\partial T(x, y, z, t)}{\partial y} \right) + \frac{\partial}{\partial z} \left( K \frac{\partial T(x, y, z, t)}{\partial z} \right) + q_{int} = \rho C \frac{\partial T(x, y, z, t)}{\partial t}
\]

or

\[V \cdot (KVT) = f \text{ on } V\]  

with the relevant boundary conditions as follows:

\[T = T_0 \text{ on } S_1\]  

\[K \cdot V T = q_{in} \text{ on } S_2\]  

\[-K \cdot \nabla T = h(T - T_{in}) + r(T' - T_{in}') \text{ on } S_3\]

where \(T(x, y, z, t)\) is the temperature of the plate, \(K, \rho, C\) are the location-dependent thermal conductivity, density, and specific heat capacity of the plate, respectively. \(q_{int}\) is the internal heat source and \(q_s\) is the applied surface heat flux. Further details on the fundamentals of the theoretical model are given in the LAMPS user’s manual (CSDM, 2017). Since the LAMPS software is a full-scale transient simulation tool, it provides detailed information on the effects of the processing parameters on the temperature field from track-to-track and layer-to-layer.

2.3.2. Physical properties

The geometries of the full substrates of 6061 Al alloy and 316L stainless steel are shown in Fig. 3 and were used to predict and analyze the real deposition. The thermophysical properties of the substrate are listed in Table 1. The effective thermophysical properties of the TiC nanoparticle-reinforced steel were obtained based on the sub-component volume fraction. The effective thermophysical properties of the powder bed were estimated in terms of the powder-bed densification and volume fractions of the TiC and steel particles.

2.3.3. Simulation parameters

Because the numerical analysis is intended to clarify the effect of varying \(n\), only the strip deposition with 8 mm length and 0.00102 mm width under the specified processing condition was modeled to reduce the computational work and save time. Considering that the powder particles are surrounded by or embedded in TiC particles, the absorptivity of \(q\) was set as 0.82. The processing parameters are listed in Table 2 for LAMPS simulation (Yuan and Gu, 2015). Since the LAMPS software considers the effect of the machine movement acceleration and deceleration, here both the acceleration and deceleration were set as 5 m/s².

2.4. Hardness and wear testing

A Leco LM800AT micro-hardness tester was used to measure the Vickers hardness values at a load of 0.2 kg and indentation period of 10 s. Each sample was subjected to 15 indentations, and the average Vickers hardness was considered. Dry rotary wear tests were performed according to the ASTM G99 standard using a T50 ball-on-disk tribometer in air at room temperature. The specimen surfaces were ground and polished before wear testing to ensure identical surface roughness values. The counterface material was a 3-mm-diameter 52,100 chrome steel ball, applied with a test load of 3 N. The friction unit was rotated at 840 rpm for 20 min with a fixed rotation radius of 2 mm. A ST 400 light profilometer with a vertical resolution of 8 nm, accuracy of 80 nm, and lateral resolution of 2 μm was used to measure the volume of materials lost (V). The wear rate (w) of the samples was then determined by:

\[w = \frac{V}{FL} \text{ (mm}^3 \text{ N}^{-1} \text{ m}^{-1})\]  

(3)

Fig. 2. Schematic of the laminated layerwise additive manufacturing model.
where $F$ is the contact load and $L$ is the total sliding distance.

3. Results and discussion

3.1. Effects of applied $\eta$ on the distribution state of TiC

During the solidification of the molten pool in SLM, the laser beam moves, and the thermal energy dissipates quickly to the substrate or the previously solidified layer. Niu and Chang (1999) and Gu and Shen (2009) both attributed this to the higher thermal conductivity of the solid relative to that of the surrounding powder, which creates temperature gradients within the molten pool. Simchi et al. (2001) suggested that gradients of temperature or chemical composition in the molten pool, which are strongly influenced by the applied $\eta$, may generate a surface tension gradient and corresponding Marangoni convection, thus inducing a non-steady-state solidification process. Fig. 4 shows SEM images of the microstructures of the SLM-processed nanocomposite parts, demonstrating that the dispersions of TiC nanoparticles are significantly influenced by the applied $\eta$. At low $\eta$, the TiC nanoparticles tend to aggregate into clusters, forming microscale agglomerates of several nanoparticles formed in the matrix (Fig. 4a and e). As the applied $\eta$ is increased to 125 J/mm$^3$, the distribution of TiC nanoparticles is improved in uniformity and the TiC particles bond together as continuous ring-like structures (Fig. 4b). The high-magnification image, however, shows the presence of some inhomogeneous TiC agglomerates in the matrix (Fig. 4f). When $\eta$ is increased further to 200 J/mm$^3$, the TiC reinforcements are uniformly and homogenously distributed throughout the matrix (Fig. 4c) with no visible aggregation of TiC nanoparticles within the SLM-processed part, even when observed under high magnification (Fig. 4g). The high-magnification image, however, shows the presence of some inhomogeneous TiC agglomerates in the matrix. Simchi and Asgharzadeh (2004) and Gu et al. (2007) reported that Marangoni convection within the molten pool, generated by high temperature and high surface tension, induced a liquid capillary force that caused rearrangement of TiC nanoparticles. Yuan and Gu (2015) found that Marangoni convection was clearly sensitive to the applied $\eta$. Using $\eta \geq 125$ J/mm$^3$, the dispersion state of the TiC reinforcements was enhanced (i.e., uniform and homogenous distribution in the matrix) but at the expense of the formation of comparatively coarse grains. By decreasing the laser scan speed, and thus the cooling rate, SLM-processed parts with increasingly coarse microstructures were obtained (e.g., Fig. 4g). This is further discussed in Section 3.3.

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$$Ma = \frac{\eta}{\nu}$$

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where $\eta$ is the contact load and $L$ is the total sliding distance.
considerably shorter time for thermal Marangoni of the liquid front in the molten pool is increased; therefore, there is a weakening both the SLM temperature gradient and the associated capillary forces. Moreover, an inadequate laser energy input appears to be insuﬃcient for TiC grain growth, leading to the favorable formation of reﬁned TiC nanoparticles (Fig. 4d). By examining the TEM image in Fig. 6a, we can conﬁrm that TiC nanoparticles approximately maintain their initial reﬁned size of 60–100 nm (Fig. 6c).

Conversely, at the lowest scan speeds in this study, η is high; hence, the temperature in the molten pool increases, resulting in signiﬁcant thermal accumulation within the molten pool. The longer molten time and lower cooling rate signiﬁcantly enhance the activity of grains within TiC nanoparticles in the molten pool, allowing grain growth for some TiC particles. The statistical results show that some TiC particles in the fabricated nanocomposite with η of 300 J/mm\(^3\) become very coarse and lose their initial standard nanostructure, reaching a value of 400 nm (Fig. 6b).

In addition, the matrix of the composite shows an increased dislocation density (Fig. 6d). Orowan bypassing of hard ﬁne particles by dislocations, which contributes to composite strengthening, is clearly revealed by the TEM image (Fig. 6b and d). Grain boundaries and particle/matrix interfaces are major sources of dislocations because of the large differences in the thermal expansion coefﬁcients between TiC and 316L. At higher η, the particles are more homogeneously distributed, creating a larger number of particle-matrix interfaces. Under these conditions, all particle clusters are broken, further increasing the number of such interfaces. At higher energy densities, more grain boundaries are available, increasing the total number of dislocation sources.

Fig. 4. SEM images showing the evolution of nanoscale TiC reinforcement dispersion states in SLM-processed TiC/316L nanocomposites at various applied η: (a,e) η = 67 J/mm\(^3\); (b,f) η = 125 J/mm\(^3\); (c,g) η = 200 J/mm\(^3\); (d,h) η = 300 J/mm\(^3\).

Ma = \(\frac{\Delta \sigma L}{\mu v_k}\) \(\tag{4}\)

where Δσ is the surface tension difference of Marangoni flow, L is the length of the free surface, \(\mu\) is the dynamic viscosity, and \(v_k\) is the kinematic viscosity. The high temperature in the SLM process induced by high η causes a sharp decrease in \(\mu\), which intensiﬁes Marangoni ﬂow and turbulence within the molten pool, as well as the magnitude of the resulting capillary force. This, in turn, increases the rate of nanoscale TiC rearrangement in the melt, preventing TiC aggregation and promoting uniform dispersion in the solidiﬁed matrix (Fig. 4c and d).

Fig. 5 depicts simple mechanisms for the varied distribution states of the TiC nanoparticles under decreased laser scan speeds and other constant processing parameters. In the laser-induced molten pool comprising TiC and 316L particles developed by SLM, a sharp temperature gradient forms between the edge and center of the molten pool. This produces a surface tension gradient and subsequent Marangoni ﬂow, which, as reported by Simchi (2006), induces a liquid ﬂow for capillary forces. Whenever capillary forces act on the TiC nanoparticles, a torque is generated around the particles, which Simchi (2008) attributed to particle center misalignment. The torque causes the rotation of the TiC nanoparticles within the pool, enabling rearrangement of the TiC distribution. Ma et al. (2013) reported that the size of the temperature gradient determined the strength of the capillary forces. Moreover, an inadequate laser energy input appears to weaken both the SLM temperature gradient and the associated capillary force intensity; the TiC nanoparticles within the molten pool cannot be suﬃciently rearranged at low η. In this condition, the solidiﬁcation rate of the liquid front in the molten pool is increased; therefore, there is a considerably shorter time for thermal Marangoni ﬂow to rearrange the TiC nanoparticles. As a result, the TiC nanoparticle distribution is signiﬁcantly non-uniform because of the formation of clusters of reinforcing particles at a comparatively low η of 67 J/mm\(^3\) (Fig. 5a).

When η is increased by decreasing the scanning speed, the rate of rearrangement of TiC reinforcing nanoparticles inside the molten pool is increased because of the intensiﬁcation of Marangoni ﬂow (Eq. 4). The torque force acts constantly on the TiC nanoparticles, which congregate around the core of the Marangoni ﬂow pattern and form a ring-like structure (Fig. 5b and c). Meanwhile, repulsive forces tend to act between TiC reinforcing particles when an adequate quantity of 316L melt develops within the molten pool, as reported by Kruth et al. (2010). Although the converging ﬂow pushes the TiC nanoparticles toward the center, the collective impact of repulsion forces and signiﬁcantly intensiﬁed Marangoni ﬂow results in a much more well-developed ring-like structure in the solidiﬁed matrix when a sufficiently high η (\(\geq 200\) J/mm\(^3\)) is applied (Fig. 5c). Under this condition, good wetting of TiC by the liquid 316L alloy enhances the TiC dispersion state. Nonetheless, when η is increased to the highest value of 300 J/mm\(^3\), the TiC reinforcement ring structure shows obvious coarsening (see Fig. 4d) because of considerable thermal accumulation within the molten pool, as well as subsequent grain growth and the large increase in the TiC particle size (as characterized in Sections 3.2 and 3.3).

3.2. Eﬀects of applied η on TiC particle size

In addition to aﬀecting the TiC dispersion state within the matrix, the applied η also aﬀects the evolution of nanoscale TiC particle size by changing the undercooling degree and the resulting solidiﬁcation rate. Fig. 6a and b show TEM micrographs of the nanocomposites fabricated under low and high η and the effect of applied η on TiC particle size. At high scan speeds, η is low, which increases the solidiﬁcation rate of the liquid front within the molten pool, causing large temperature gradients within the pool and high degrees of undercooling in the melt. Typically, the time available before solidiﬁcation is insuﬃcient for TiC grain growth, leading to the favorable formation of reﬁned TiC nanostructures (Fig. 6a). By examining the TEM image in Fig. 6a, we can conﬁrm that TiC nanoparticles approximately maintain their initial reﬁned size of 60–100 nm (Fig. 6c).

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3.3. Effects of applied $\eta$ on grain size

EBSD orientation maps of the nanocomposites using various $\eta$ values, taken from the top views of the fabricated cylinders, are shown in Fig. 7. Analysis of the cross-sectional surfaces (i.e., parallel to the building direction) was undertaken in our previous study (AlMangour et al., 2018). $\eta$ clearly influences the thermal history and liquid flow by altering the solidification rate, which in turn determines the final solidified microstructure.

During the solidification process, the local growth velocity of the solidification front, $v_s$, can be calculated directly by measuring the angle ($\theta$) between $v_s$ and the laser scan speed $v$ using the following equation proposed by Liu et al. (2004):

$$v_s = v \cos(\theta)$$  

(5)

The thermal undercooling $\Delta T_t$ is expressed as follows defined by Schwarz et al. (1997):

$$\Delta T_t = \frac{\Delta H_f}{c_p} F_I P_e$$  

(6)

where $P_e = \frac{v L}{k_0 v_s}$ is the thermal Péclet number, $\Delta H_f$ is the heat of fusion (J mol$^{-1}$), $c_p$ is the specific heat of the liquid (J mol$^{-1}$ K$^{-1}$), $F_I$ is the Ivantsov function, $R$ is the radius of curvature of the crystal tip (m), and $D_T$ is the thermal diffusivity (m$^2$ s$^{-1}$).

The kinetic undercooling $\Delta T_k$ within the pool, defined by Schwarz et al. (1997), is expressed as:

$$\Delta T_k = \frac{v_s}{\lambda}$$  

(7)

where $\lambda = \frac{\Delta H_f}{k_0 T_L}$ is the interfacial kinetic coefficient, $v_s$ is the speed of sound (m s$^{-1}$), $k_0$ is the Boltzmann constant, and $T_L$ is the liquidus temperature (K). At low $v$, the input $\eta$ during line-by-line scanning is high, causing an elevated thermalization of the energy and a higher SLM temperature. With increased $v$, the solidification rate of the liquid front within the pool is increased (Eq. 5), thereby enhancing both $\Delta T_t$ (Eq. 6) and $\Delta T_k$ (Eq. 7). Significantly enhanced temperature gradients within the pool further refine the grain size (Fig. 7a).

The TiC nanoparticles have smaller grain sizes and higher melting points than those of the 316L stainless steel matrix. Smallman and Ngan (2013) reported that these remained partially melted as impurity phases, and acted as nucleation sites for the molten stainless steel, facilitating heterogeneous nucleation and consequently causing further grain refinements. The main driving force in the molten pools of the SLM process is, as reported by Niu and Chang (1999), convection formed by a combination of surface tension gradients, viscous shear stress, and buoyancy forces. Furthermore, during SLM, laser scanning is performed line-by-line, followed by layer-by-layer. These sequential passes influence the grain formation in multiple directions, as observed in the EBSD images, because of their corresponding thermal flows. As the temperature gradient decreases, the grain size increases.

Microstructural development in these alloys is dependent on the thermal history experienced by different regions of a component. In the solidification process, $G$ is the temperature gradient in the liquid phase and $R$ is the growth rate of the interface, or the solidification rate. These
Fig. 6. TEM images showing the TiC particle size of SLM-processed TiC/316L nanocomposites obtained at (a,c) $\eta = 67 \text{ J/mm}^3$; and (b,d) $\eta = 300 \text{ J/mm}^3$. Note the size of the nearly spherical TiC particles distributed in the 316L matrix, as well as the increased dislocation density of the 316L austenitic matrix.

Fig. 7. EBSD orientation maps of SLM-processed TiC/316L nanocomposites obtained at (a) $\eta = 67 \text{ J/mm}^3$; (b) $\eta = 125 \text{ J/mm}^3$; (c) $\eta = 200 \text{ J/mm}^3$; (d) $\eta = 300 \text{ J/mm}^3$. Note the increase in grain size.
two factors determine the grain morphology. Zhang et al. (2014) reported that a high G and low R caused columnar growth, while a low G and high R caused equiaxed growth. Rapid solidification and hence, high R values, is inherent to the SLM process. The strategy used in this study of alternating the hatched laser scans results in shorter scan tracks, which, as noted by Vrancken et al. (2014), produce lower temperature gradients (low G). Therefore, the low G/R ratio in this study produces equiaxed grains in the SLM-processed nanocomposite (Fig. 7a).

In addition to columnar grains, fine equiaxed grains are also observed. The fine grains surround the unmelted TiC particles, growing parallel to the surface of the particles due to the high thermal conductivity of TiC relative to that of 316L. This generates a higher G surrounding the TiC particles, as heat dissipates faster from the liquid phase along the TiC particles. When the melt tracks overlap, G is further increased by re-melting, resulting in the formation of bimodal grains in the solidified nanocomposite. Zhu et al. (2014) attributed the formation of these directional microstructures to the Gaussian laser heat source, defined by a non-uniform power distribution and a fluctuating energy output. This created multiple temperature gradients in different directions within the molten pool. Because of the coupling of multiple nucleation sites in the molten pool and the different thermal conductivities in the liquid, solid, and powder, sub-structural anisotropy was generated.

3.4. Thermal behavior prediction

Because LAMPS simulated the transient temperature field response track-to-track and layer-by-layer for the whole structure system, only some time-step results were extracted for comparison and discussed. Figs. 8 and 9 show the transient temperature field changes of the first seven tracks in the first layers deposited at η = 300 J/mm$^3$ and 67 J/mm$^3$, respectively. A higher η value corresponds to a higher peak temperature, as shown in Figs. 8 and 9.

The peak temperature can surpass 3200 K at the boundary between the deposited zone and loose powder zones; this is because of the large differences in the thermophysical properties of these zones, which hinder the transfer of laser energy between zones. Based on Takamichi and Roderick’s results, the dynamic viscosity μ of the melt liquid (unit: Pa s) is defined by (Yuan, 2015):

$$\mu = \frac{16\gamma}{15} \sqrt{\frac{m}{k_B T}}$$  \hspace{1cm} (8)

where γ is the surface tension (N m$^{-1}$), m is the atomic mass, $k_B$ represents the Boltzmann constant, and $T$ is the liquid temperature. The dynamic viscosity decreases with an increase in the temperature of the liquid. Thus, in the current study, a higher η induces higher temperatures in the molten pool. The higher temperatures allow decreases in the liquid dynamic viscosity and increases in the fluid flow, which is governed by the following equations:

$$-\mu \frac{\partial u}{\partial x} = \frac{\partial y}{\partial T} \frac{\partial T}{\partial x} \hspace{1cm} (9a)$$

$$-\mu \frac{\partial v}{\partial y} = \frac{\partial y}{\partial T} \frac{\partial T}{\partial y} \hspace{1cm} (9b)$$

where $u$ and $v$ are the fluid flow velocities (m s$^{-1}$) along the x and y directions.

From Eq. (9a,b), the fluid flow velocities show strong dependencies on the dynamic viscosity $\mu$, surface tension temperature coefficient $\frac{\partial y}{\partial T}$, and the temperature gradients $\frac{\partial T}{\partial x}$ and $\frac{\partial T}{\partial y}$. In general, the surface tension temperature coefficient is an inherent constant for a given material. The temperature gradient becomes the main factor dominating the molten pool fluid-flow velocity. Here, Fig. 10 shows the detailed effects of η on the temperature gradient changes during the deposition. Clearly, higher η creates higher temperature gradients. Fig. 11 clarifies this effect by illustrating the relationship between η and the maximum temperature gradient in three directions. As indicated in Eq. (9a,b) for the fluid flow velocity, the higher temperature gradient permits higher fluid flow velocity under the given conditions.

Since LAMPS simulates the whole deposition procedure, the molten pool size changes from location to location because of the edge effect and structural geometry. However, the simulations confirm that the molten pool size for higher laser energy densities is larger than that for lower densities. Fig. 12 displays the change in molten pool size at one simulation step for different laser energy densities.

Furthermore, since the TiC melting point is much higher than that of 316L steel at 3430 K, the TiC particles are not melted and are instead suspended in the molten pool. Thus, the TiC particles must be a buoyancy effect. Assuming a TiC particle with a radius $r$ in the molten pool, the speed of the flotation $v_{buoy}$ or buoyancy-driven flow, is given by Stokes’ law as (Bird et al., 1960):
\[ v_{\text{buoy}} = \frac{2g}{9} \left( \rho_{\text{melt}} - \rho_{\text{TIC}} \right) \frac{r}{\mu_{\text{melt}}} \]  

where \( \rho_{\text{melt}} \) is the density of the 316L melt, \( \rho_{\text{TIC}} \) is the density of the TIC particle with the positive direction being against gravity \( g \), and \( \mu_{\text{melt}} \) is the dynamic viscosity of the molten pool.

On the other hand, the Marangoni-driven flow \( v_{\text{Mar}} \) of a TIC particle is presented as (Bergman et al., 1988):

\[ v_{\text{Mar}} = \frac{2}{3} \left( \frac{r}{3 \mu_{\text{melt}} + 3 \mu_{\text{TIC}}} \right) \frac{\partial \mu}{\partial X} \frac{\partial T}{\partial X} \]  

where \( \mu_{\text{TIC}} \) is the viscosity of the TIC. For a solid TIC particle, it is equal to zero.

From Eq. (10), the buoyancy effect always allows TIC to flow to the surface, because the density of TIC is much lower than that of the molten pool. From Eq. (11), the flow speed and direction depend on the temperature coefficient of surface tension and the temperature gradient. For a given condition and component materials, Eq. (11) shows that the speed of the Marangoni-driven flow depends only on the temperature gradient, which means that higher temperature gradients cause higher flow speeds. The temperature coefficient of surface tension depends on many factors. Because the effects of TIC nanoparticles on the surface tension of molten pools are unclear, further study is necessary to clarify the flow speed. Regardless, there are only two cases for the temperature coefficient of surface tension: a negative-value case and a positive-value case.

For a negative temperature coefficient of surface tension \( \frac{\partial \mu}{\partial T} \), the Marangoni-driven flow is always directed upward to the surface and then outward to the molten pool edge. Because the buoyancy-driven flow speed is always positive, when driving the TIC particle toward the surface, both the buoyancy and Marangoni effects promote TIC particle flow in the same direction. However, for a positive temperature coefficient of surface tension \( \frac{\partial \mu}{\partial T} \), the Marangoni-driven flow is directed toward the molten pool center and downward to the bottom of the pool. In this case, the buoyancy effect partially or completely cancels the Marangoni effect. Thus, the speed ratio of the Marangoni- and buoyancy-driven flows can be introduced to analyze the TIC particle flow characteristics, as the ratio of Eq. (11) to Eq. (10):

\[ \frac{v_{\text{buoy}}}{v_{\text{Mar}}} = \frac{3}{2g} \left( \frac{\partial \mu}{\partial T} \right) \frac{\partial T}{\partial X} \frac{\partial X}{\partial T} \]  

From Eq. (12), the speed ratio depends strongly on the temperature gradient as well as other inherent factors. A particle can be suspended in the molten pool under a suitable temperature gradient; in SLM, the temperature gradient is controllable, depending on the processing parameters.

As mentioned above, the effects of TIC nanoparticles on the surface tension of molten 316L are poorly characterized. However, the existing data permits some basic analysis. The temperature coefficient of surface tension for 316L is \(-4.0 \times 10^{-4} \text{N m}^{-1} \text{K}^{-1}\) and the viscosity is \(6.7 \times 10^{-3} \text{kg m}^{-1} \text{s}^{-1}\) (Mills, 2002). The buoyancy-driven flow speed for a 60-nm-diameter TIC particle is 0.0280 m s\(^{-1}\); the respective speeds of the Marangoni-driven flow are shown in Table 3. From this data, the speed of particle movement is much lower than 0.05 m s\(^{-1}\). However, higher laser energy densities (lower scan speeds) entail increased molten pool sizes, providing sufficient time for Marangoni-driven flow to finish the flow cycle at lower scan speeds before solidification occurs.

### 3.5. Microhardness and tribological performance

Fig. 13 depicts the effect of the applied energy density on the microhardness values measured on polished cross-sections of SLM-processed TiC/316L parts. Clearly, the incorporation of TIC particles into the 316L matrix (average hardness of \(\sim 215 \text{HV}_0.2\)) increases the hardness values, irrespective of the applied \(\eta\). Hardness is influenced by variations in the cooling rates at various energy densities. The highest hardness values are obtained in samples produced at the highest scanning speed and lowest \(\eta\) (67 J/mm\(^3\)). This is associated with a finer microstructure, as observed in the EBSD images (Fig. 7a). Above \(\eta = 67 \text{J/mm}^3\), a sharp reduction in hardness occurs because of the increase in grain size (Fig. 7b).

In general, SLM processing at higher \(\eta\) causes two opposing effects on the hardness values of the processed parts (Fig. 13). The reduced scanning speed improves layer melting, which improves binding between the molten particles, creating higher densification and reduced porosity, and hence, better mechanical properties. The average relative
densities of the nanocomposites processed using various $\eta$ values are shown in Table 4; a detailed discussion of their densification behavior has been reported in an earlier study (AlMangour et al., 2018). However, the grain refinement caused by rapid solidification after laser melting enhanced the mechanical properties according to the Hall–Petch relationship. Song et al. (2014b) proposed that the high mechanical strength of SLM-processed specimens was related to the fine grains and high dislocation densities resulting from high cooling rates, while Abe et al. (2003) suggested that high porosities caused low ductility in SLM samples. In this study, the lowest energy density resulted in higher hardness, despite its low average relative density (Table 4).

At lower scanning speeds and higher $\eta$, the size of the molten pool increased, and the thermal gradient decreased, causing lower thermal and residual stresses, which decreased the hardness of the sample. The higher temperature of the molten pool and the lower cooling rate also increase the grain size due to the longer melt time, which reduces hardness. However, the homogenous distribution of TiC particles
which is attributed to the more limited densification level and pore formation relative to other samples. As observed in the SEM images, at this low $\eta$, the TiC nanoparticles form clusters within the 316L matrix, leading to poor microstructural homogeneity (Fig. 4a). The pores and nanoparticle aggregates are both prone to cracking due to stress concentration during sliding. This forms debris, which separate from the wear surface when the expanded cracks become connected, resulting in significant increases in the wear rate.

The characteristic morphologies of the worn surfaces of the tested samples are shown in Fig. 15. At low $\eta$, the wear surface is disrupted, showing deep parallel grooves, large agglomerates, and some ultrafine particles. The observed abrasive worn morphology in this condition, characterized by the presence of irregular fragments at the groove edges with severe local deformation and plowing of the surface during sliding, indicates a relatively high wear rate. By increasing $\eta$ to 125 J/mm$^3$, a significant amount of loose abrasive fragments remains on the worn surface, but many fewer large agglomerates are observed on the worn surface (Fig. 15b). The worn surface shows slightly shallower grooves with localized deep portions. This is accompanied by a corresponding wear rate of $2.959 \times 10^{-4}$ mm$^3$ N$^{-1}$ m$^{-1}$ (Fig. 14). For $\eta = 200$ J/mm$^3$, the worn surface of the SLM-processed sample becomes smooth, with reinforcing nanoparticles embedded in the adhesive tribolayer. An adherent and strain-hardened tribolayer completely covers the worn surface of the sample (Fig. 15c). This characteristic led to the lowest wear rate of $2.328 \times 10^{-4}$ mm$^3$ N$^{-1}$ m$^{-1}$ (Fig. 14) obtained in this study.

The TiC/316L nanocomposite part produced at the optimum $\eta$ of 200 J/mm$^3$ demonstrates superior wear performance. It is reasonable to suggest that, as the TiC reinforcing nanoparticles are homogeneously dispersed within the coherent adhesive tribolayer in the SLM-processed part, the abrasive wear mechanism during prolonged sliding changes to tribolayer adhesion. This transition to tribolayer formation is expected to reduce the wear rate in the sliding tests, as was suggested by Tjong (2007) to explain the uniform distribution of nanoscale TiC reinforcements with lower interfacial stresses in the matrix, and thus minimal nanocomposite breakage during sliding. Jain et al. (2010) reported that improved densification and increased hardness of a sample allows easy plastic smearing of the protective tribolayer on the worn surface.

When $\eta$ is increased further to 300 J/mm$^3$, although some localized tribolayer formation is observed on the worn surface, it shows signs of surface plowing. Parallel grooves as well as much debris were formed on the worn surface after sliding. The wear mechanism in this instance is micro-plowing rather than adhesion. Some reinforcing particles are also observed at groove edges on the worn surface. All of this explains the slight increase in the average wear rate of $2.558 \times 10^{-4}$ mm$^3$ N$^{-1}$ m$^{-1}$ (Fig. 14) relative to that of the sample processed at $\eta = 200$ J/mm$^3$. These characteristics of the worn morphology observed in this condition could be attributed to the coarse microstructure, as well as the resultant disappearance of nanoscale TiC reinforcement. TiC tends to split more easily under severe sliding wear, which in turn increases the wear rate. Similar observations were reported by Gu et al. (2014).

Under the optimum $\eta$, the nanoscale TiC reinforcements are uniformly distributed in the matrix (Fig. 4c and g). During sliding wear, the homogeneously dispersed ultrafine TiC particles stick together, driven by small reinforcement/matrix interfacial stresses. Friction on the surface causes plastic deformation, which produces a strain-hardened layer of refined TiC nanoparticles on the wear surface (Fig. 15c) and thereby improves the wear properties. At higher $\eta$, although the densification level is maintained (Table 4), TiC loses its nanostructure during SLM processing (Fig. 5). The coarsened TiC particles are thus prone to spalling and splitting under sliding. The plowing action between the TiC particles and 316L matrix occurs with a tangential force, producing the deep grooves on the surface that suggest limited wear performance (Fig. 15d).

The established relationships between SLM process parameters,
Fig. 12. Molten pool size at the 19th simulation step for different laser energy densities: (a) $\eta = 67 \text{ J/mm}^3$, (b) $\eta = 125 \text{ J/mm}^3$, (c) $\eta = 200 \text{ J/mm}^3$, and (d) $\eta = 300 \text{ J/mm}^3$.

Table 3
Maximum speed vs. laser energy density.

<table>
<thead>
<tr>
<th>Laser energy density (J/mm$^3$)</th>
<th>Maximum speed (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>x-direction</td>
</tr>
<tr>
<td>67</td>
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</tr>
<tr>
<td>125</td>
<td>$-0.000597612$</td>
</tr>
<tr>
<td>200</td>
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</tr>
<tr>
<td>300</td>
<td>$-0.000853731$</td>
</tr>
</tbody>
</table>

Fig. 13. Effect of applied $\eta$ during SLM processing of TiC/316L nanocomposite parts on microhardness.

Table 4
Average relative densities of the TiC/316L nanocomposite parts obtained using various energy density values during SLM.

<table>
<thead>
<tr>
<th>Energy density $\eta$ (J/mm$^3$)</th>
<th>Relative density (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>67</td>
<td>90.39</td>
</tr>
<tr>
<td>125</td>
<td>93.01</td>
</tr>
<tr>
<td>200</td>
<td>96.10</td>
</tr>
<tr>
<td>300</td>
<td>98.22</td>
</tr>
</tbody>
</table>

Fig. 14. Effect of applied $\eta$ during SLM processing of TiC/316L nanocomposite parts on their average wear rate.
solidification microstructure, and sample hardness assist in understanding the tribological performance of the TiC/316L nanocomposites. The SLM energy densities strongly influence the tribological performance of the TiC/316L nanocomposites. Laser processing parameters should be carefully selected from an optimum processing window to ensure refined microstructure with adequate densification in order to achieve the best tribological properties under harsh wear conditions.

4. Conclusion

(1) In this study, the distribution state and particle size of nanoscale TiC reinforcements, as well as the grain size of the nanocomposites in a 316L matrix, can be tailored by regulating $\eta$ used during SLM. Increasing $\eta$ from 67 to 300 J/mm$^3$ homogenized the dispersion of nanoscale TiC reinforcing particles from agglomerates, while causing partial disappearance of the reinforcement nanostructure and significant increase in grain size.

(2) Simulations of the dynamics of the molten pool showed that the maximum temperature gradient within the molten pool was increased significantly with increasing $\eta$.

(3) Both microhardness and wear rates were influenced by the degree of densification, grain coarsening due to lower cooling rates, and the size and dispersion state of TiC particles in the matrix. The highest microhardness was obtained at $\eta = 67$ J/mm$^3$, which was attributed to the finest grain structure. The nanocomposites processed under optimum processing conditions ($\eta = 200$ J/mm$^3$) showed the lowest wear rate of $2.328 \times 10^{-4}$ mm$^3$N$^{-1}$m$^{-1}$, and a strain-hardened adherent tribolayer formed on the worn surface.

(4) The experimental study, accompanied by the numerical simulation of temperature evolutions in SLM processing, may clarify the thermal behaviors active in this method of fabrication. Thus, the processing parameters during SLM can be optimized to obtain a desirable microstructure and good tribological performance.

Acknowledgments

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References


Fig. 15. SEM images showing typical morphologies of the worn surfaces of TiC/316L parts processed using (a) $\eta = 67$ J/mm$^3$, (b) $\eta = 125$ J/mm$^3$, (c) $\eta = 200$ J/mm$^3$, and (d) $\eta = 300$ J/mm$^3$.