Combined strengthening of multi-phase and graded interface in laser additive manufactured TiC/Inconel 718 composites

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Combined strengthening of multi-phase and graded interface in laser additive manufactured TiC/Inconel 718 composites

Dongdong Gu\textsuperscript{1,5}, Chen Hong\textsuperscript{3}, Qingbo Jia\textsuperscript{1}, Donghua Dai\textsuperscript{1}, Andres Gasser\textsuperscript{2}, Andreas Weisheit\textsuperscript{2}, Ingomar Kelbassa\textsuperscript{2,3}, Minlin Zhong\textsuperscript{4} and Reinhart Poprawe\textsuperscript{2,3}

\textsuperscript{1} College of Materials Science and Technology, Nanjing University of Aeronautics and Astronautics (NUAA), Yudaow Street 29, 210016 Nanjing, People’s Republic of China
\textsuperscript{2} Fraunhofer Institute for Laser Technology ILT, Steinbachstraße 15, D-52074 Aachen, Germany
\textsuperscript{3} Chair for Laser Technology LLT, RWTH Aachen, Steinbachstraße 15, D-52074 Aachen, Germany
\textsuperscript{4} School of Materials Science and Engineering, Tsinghua University, 100084 Beijing, People’s Republic of China

E-mail: dongdonggu@nuaa.edu.cn

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Abstract

Laser metal deposition (LMD) additive manufacturing of TiC particle reinforced Inconel 718 composite parts was performed. The influence of laser energy density (LED) on densification, microstructures and wear behaviour of LMD-processed composites was studied. It showed that using a LED of 280 J mm\textsuperscript{-3} produced \textasciitilde5% porosity in LMD-processed composites, caused by the aggregation of reinforcing particles. A further increase in LED above 350 J mm\textsuperscript{-3} yielded near-full densification. Two categories of reinforcing phases, i.e. the substoichiometric TiC\textsubscript{x} particles and the in situ (Ti, M)C (M = Mo, Nb and Cr) carbide having 7–10 at% Nb and Mo contents, were formed in the matrix of LMD-processed composites. The TiC\textsubscript{x} reinforcing particles changed from an irregular poly-angular shape to a smoothened and refined structure as the LED increased. An increase in LED resulted in a larger amount of phase formation and an enhanced degree of crystal growth of the in situ (Ti, M)C reinforcement. The interfacial graded layer with thickness of 0.2–1.2 µm, which was identified as (Ti, M)C (M = Mo, Nb and Cr) carbide with 5–6 at% Mo and Nb contents, was tailored between the TiC\textsubscript{x} particles and the matrix. At an optimal LED of 420 J mm\textsuperscript{-3}, a considerably low coefficient of friction of 0.38 and resultant low wear rate of 1.8 \times 10\textsuperscript{-4} mm\textsuperscript{3} N\textsuperscript{-1} m\textsuperscript{-1} were obtained in sliding tests, due to the combined strengthening of the interfacial graded layer and the multiple reinforcing phases. The wear resistance decreased at an excessive LED because of the coarsening of reinforcement crystals and the decrease in microstructural uniformity of composites.

Keywords: additive manufacturing, laser metal deposition (LMD), metal matrix composites (MMCs), particle reinforcement, graded interface

(Some figures may appear in colour only in the online journal)

1. Introduction

Additive manufacturing (AM) is currently one of the rapidly developing advanced manufacturing techniques in the world [1–3]. Unlike the material removal method in conventional machining processes, AM is based on an entirely opposite materials incremental manufacturing (MIM) philosophy. AM implies layer-by-layer shaping and consolidation of feedstock (typically powder materials) to arbitrary complex configurations, normally using a computer-controlled laser as the energy resource. As a primary step of AM, the computer-aided design (CAD) model of the part to be built is mathematically sliced into thin layers. The part is then
produced by the controlled consolidation of the deposited material layers with a scanning laser beam. Each shaped layer represents a cross-section of the sliced CAD model. AM technology accordingly offers a unique potential for freeform fabrication of complex shaped parts that cannot be easily produced by conventional processes.

Laser metal deposition (LMD), being capable of processing a wide range of metals, alloys and metal matrix composites (MMCs), is presently regarded as one of the most versatile AM processes [4]. LMD process originates from the laser cladding technology, which has demonstrated its capability to locally tailor the substrate surface to the designed macro/microstructures with designed properties [5]. In addition to being a two-dimensional (2D) coating technology, LMD has also exhibited a revolutionary 3D manufacturing capability [6]. LMD can be applied to coat, build and rebuild components having complex geometries, sound material integrity and dimensional accuracy [7–9]. As a natural development from 2D coating to 3D manufacturing, the recent research focus of LMD is to produce complex shaped end-use metallic parts that are hard to process by other conventional methods, in order to meet the demanding requirements from aerospace, automotive, rapid tooling and biomedical industries [10].

Inconel 718 is a high-strength, corrosion-resistant nickel chromium based alloy used at a high temperature up to ~700°C. The ease and economy with which Inconel 718 can be fabricated, combined with good tensile, fatigue, creep and rupture strength, have favoured its use in a wide range of applications, e.g. components for liquid fuelled rockets, rings, and aircraft and land-based gas turbine engines [11–13]. Nevertheless, the limited hardness and wear resistance of Inconel 718 is a serious concern for the application environments where abrasive and erosion phenomena exist [14]. The preparation of Inconel based MMCs is hoped to solve this problem via the reinforcement of the material with harder and stiffer ceramic particles [15–18]. Nickel-based alloys are normally reinforced with transition metal carbides, e.g. TiC [14], WC [19] and CrC [20], to improve the strength, hardness and wear resistance. These MMC parts are typically produced by powder metallurgy and liquid metal processing methods (e.g. stir casting, infiltration casting), showing a favourable flexibility of achievable compositions [21, 22]. However, the insufficient densification rate and inhomogeneous microstructure caused by the segregation of reinforcing particles are most likely to occur in these conventionally processed MMCs.

LMD, due to its flexibility in materials and shapes, is expected to create new technological opportunities for net shaping Inconel 718 based MMC parts with unique microstructural and mechanical properties. Nevertheless, one significant challenge that occurs when MMCs are produced with LMD is the formation of interfacial microcracks or pores between the reinforcing particles and the matrix. As the wettability between ceramics and metals is usually poor, the limited particle/matrix interfacial bonding due to the microcracks or pores formation may result in the premature failure of MMCs during mechanical loading [23]. Furthermore, LMD involves a highly non-equilibrium, rapid melting and solidification process caused by a high-energy laser beam, which in turn influences the chemical concentration and resultant microstructural development within the molten pool [3]. Considerable research efforts are required to study how the microstructures of ceramic reinforcing phases and reinforcement/matrix interfaces are developed during LMD. The underlying role of microstructural evolution in the changes of mechanical properties of LMD-processed MMC parts should be clarified.

In this work, the TiC particle reinforced Inconel 718 MMCs parts with multiple reinforcing phases and unique microstructural features were prepared by LMD. The influence of laser energy density (LED) on the microstructural development, densification behaviour, and wear performance of LMD-processed MMCs was studied. A material–microstructure–property relationship was proposed that enabled a successful LMD production of Inconel 718 based MMCs parts with novel reinforcement architecture and elevated performance.

2. Experimental procedures

2.1. Powder preparation

The gas atomized, spherical Inconel 718 powder with the particle size distribution of 15–45 μm and 99.5% purity, irregular shaped TiC powder with the particle size distribution of 22.5–45 μm were used. The chemical compositions of Inconel 718 powder were 50–55 Ni, 17–21 Cr, 4.75–5.5 Nb, 2.8–3.3 Mo, 0.65–1.15 Ti, 0.2–0.8 Al, 0.08 C, and Fe balance (wt%). Through a series of preliminary LMD experiments on the TiC/Inconel 718 powder systems containing different contents of TiC particles, the TiC content was optimized at 20–30 wt% to obtain a favourable LMD processability and a sufficiently high densification response. In this study, the Inconel 718 and TiC powder components, according to a weight ratio of 75:25, were homogeneously mixed in a Fritsch Pulverisette 6 planetary ball mill using a ball-to-powder weight ratio of 5:1, a rotation speed of the main disc of 200 rpm, and a milling time of 4 h.

2.2. LMD process

The LMD system consisted of a Nd : YAG laser with a maximum output power of 3 kW and a spot diameter of 0.6 mm, a powder feeding system, a 5-axis CNC machine, and a standard optics equipped with a coaxial powder nozzle. The C45 carbon steel was taken as the substrate material. The as-prepared TiC/Inconel 718 powder mixture was injected into the molten pool through the nozzle with Ar as carrier gas, using a powder feeding rate of 2.4 g min⁻¹. The multiple tracks were cladded for each layer with the dimensions of 5 mm × 20 mm and multiple layers were deposited on the substrate to produce the desired 3D parts with a height of 8 mm. LMD fabrication was based on the line-by-line laser cladding, using a constant laser scan speed of 500 mm min⁻¹. Three main parameters were involved in laser operation, i.e. spot diameter (D), laser power (P), and scan speed
An integrated parameter ‘laser energy density’ (LED), which was defined by \( LED = \frac{P}{\pi (D/2)^2} \times v \), was used to estimate the laser energy input to the track being deposited [24]. Four different LEDs of 280, 350, 420 and 490 J mm\(^{-3}\) were set periodically to study the influence of different LMD parameters on the processability and attendant microstructural and mechanical properties of LMD-processed parts.

2.3. Microstructural and mechanical properties characterization

Cross-sections of LMD-processed TiC/Inconel 718 composite parts were cut, ground and polished according to standard procedures to prepare the metallographic samples. To reveal the etched microstructures, an etchant consisting of HCl and \( \text{H}_2\text{O}_2 \) with a volume ratio of 10:3 was used, with the etching time of 25 s. Microstructures were characterized using an LEO 1550 field emission scanning electron microscope (FE-SEM) (Carl Zeiss NTS GmbH, Germany) at an accelerating voltage of 5 kV. A UTHSCSA ImageTool 3.0 image processing and analysis program was used to acquire the sizes of reinforcing particles. Chemical compositions were determined by an EDAX energy dispersive x-ray spectroscope (EDX) (EDAX Inc., USA), using a super-ultra thin window (SUTW) sapphire detector.

The density of LMD-processed TiC/Inconel 718 composite parts was determined based on the Archimedes principle. Based on the ASTM G99 standard, the dry sliding wear tests on the polished cross-sections of LMD-processed parts were conducted in a HT-500 ball-on-disc tribometer at room temperature 15°C. A \( \Phi 3 \) mm bearing steel GCr15 ball with a mean hardness of HRC60 was taken as the counterface material, using a test load of 6 N. The friction unit was rotated at 560 rpm for 30 min and the rotation radius was 2 mm. The coefficient of friction (COF) was recorded during sliding. The wear volumes \( (V) \) of specimens were determined gravimetrically using \( V = M_{\text{loss}}/\rho \), where \( M_{\text{loss}} \) was the weight loss of specimens after tests and \( \rho \) was the density. The wear rates \( (\omega) \) were calculated by \( \omega = V/(WL) \), where \( W \) was the contact load and \( L \) was the sliding distance.

3. Results

3.1. Densification behaviour and particle dispersion

Figure 1(a) depicts the change of densification levels of LMD-processed TiC/Inconel 718 composite parts with the applied LEDs. At a relatively low LED of 280 J mm\(^{-3}\), a full densification was not realized after LMD, leaving \( \sim 5\% \) porosity in LMD-processed composites. On increasing the LED \( \geq 350 \) J mm\(^{-3}\), the obtainable densification rates of LMD-processed parts were generally larger than 98.5% theoretical density, implying the near-full densification response of TiC/Inconel 718 composites during LMD process. The typical surface morphologies of LMD-processed composite parts at various LED are illustrated in figure 1(b).

As a relatively low LED of 280 J mm\(^{-3}\) was used, the laser-processed surface consisted of individual scan tracks with the limited inter-track bonding coherence. As the applied LED increased to 420 J mm\(^{-3}\), the neighbouring scan tracks were sufficiently bonded, forming a smooth and dense surface after LMD process. At an even higher LED of 490 J mm\(^{-3}\), although the entire surface remained dense, the laser-processed surface became rough because of the formation of the relatively wide scan tracks at a higher laser energy input (figure 1(b)).

Low-magnification FE-SEM images on the polished cross-sections of LMD-processed TiC/Inconel 718 composite parts at different LED are revealed in figure 2. At a relatively low LED of 280 J mm\(^{-3}\), a fraction of TiC reinforcing particles were aggregated into clusters in local areas of the matrix, as selectively indicated in figure 2(a). As the LED increased above 350 J mm\(^{-3}\), the dispersion state of the TiC reinforcing particles in the matrix became much more homogeneous, showing no apparent aggregation of particles (figures 2(b) and (c)). Generally, an insufficient densification activity of MMCs during laser processing was
Figure 2. FE-SEM images showing the dispersion state of TiC reinforcing particles on the polished cross-sections of LMD-processed TiC/Inconel 718 composite parts at different LEDs: (a) LED = 280 J mm$^{-3}$; (b) LED = 420 J mm$^{-3}$; (c) LED = 490 J mm$^{-3}$.

mainly ascribed to (i) the gas entrapment and resultant formation of large-sized pores; and (ii) the aggregation of reinforcing particles and resultant formation of interfacial micropores and/or microcracks between the accumulated particles [25]. In this study, even for the TiC/Inconel 718 composites processed at a relatively low LED, there are no apparent large-sized pores formed in the LMD-processed parts (figure 2(a)), implying less gas entrapment phenomenon occurred during LMD. The incomplete densification due to the residual $\sim 5\%$ porosity (figure 1(a)) was mainly caused by the aggregation of reinforcing particles (figure 2(a)).

3.2. Microstructures and compositions of multiple reinforcing phases and graded interfacial layer

The average sizes of TiC reinforcing particles in the matrixes of LMD-processed composites at various LED were acquired by accounting $\sim 200$ particles using the SEM image processing and analysis program. The characteristic morphologies of TiC particles are illustrated in figures 3(a), (c) and (e), respectively. On increasing the LED from 280 to 420 J mm$^{-3}$, the mean size of TiC reinforcing particles decreased sharply from 37 to 22 $\mu$m. The microstructures of particles changed from an irregular poly-angular shape to a round structure, showing a significant refinement and smoothening of the particles (figures 3(a) and (c)). A further increase in LED to 490 J mm$^{-3}$ led to a slight decrease in the average size of TiC reinforcing particles to 15 $\mu$m. The particles had a round and smooth structure, showing no apparent change in particle morphology (figure 3(e)).

On the other hand, high-magnification FE-SEM characterization revealed that the interfacial graded layers were generally formed between the TiC reinforcing particles and matrixes for the given LED, as shown in figures 3(b), (d) and (f). The quantitative elemental determination by EDX area scan indicated that 57.03 at% C and 28.92 at% Ti, as the elements from TiC particles, were detected in the interfacial layer. Meanwhile, the Mo, Nb and Cr elements from the Inconel 718 matrix were identified, among which the Nb and Mo demonstrated relatively high elemental concentrations above 5 at%. The atomic ratio of the C element and the metallic elements (57.03 at% versus 46.93 at%) was close to 1 : 1 (figure 4). It was accordingly reasonable to consider that the Mo, Nb and Cr, as the strong carbide-forming elements [26], reacted with the dissolved Ti and C elements on the surface of TiC particles, forming the (Ti, $M$)C ($M$ = Mo, Nb and Cr) carbide interfacial layer between the reinforcement and the matrix. Furthermore, it was noted that the applied LED played a significant role in influencing the thickness of the carbide graded layer. A thin graded layer with a mean thickness of 0.2 $\mu$m was present at a relatively low LED of 280 J mm$^{-3}$ (figure 3(b)). On enhancing the LED from 420 to 490 J mm$^{-3}$, the (Ti, $M$)C carbide graded layers showed an apparent increase in the mean thickness from 0.5 to 1.2 $\mu$m (figures 3(d) and (f)).

Figure 5 shows the characteristic microstructures on the etched cross-sections of LMD-processed TiC/Inconel 718 composites using different LEDs. The corresponding high-magnification microstructures in the interfacial areas are provided in figure 6. Generally, two categories of
Figure 3. FE-SEM images showing the characteristic morphologies of TiC reinforcing particles and the graded interfacial layer between reinforcement and matrix on the polished cross-sections of LMD-processed TiC/Inconel 718 composites at various LED: (a), (b) LED = 280 J mm$^{-3}$; (c), (d) LED = 420 J mm$^{-3}$; (e), (f) LED = 490 J mm$^{-3}$. In order to obtain suitable micrographs to display the morphologies of TiC reinforcing particles, (a), (c) and (e) are taken at different magnifications.

Figure 4. EDX analysis of elements within area A in figure 3(f), showing the chemical compositions of the interfacial graded layer between the reinforcement and the matrix.

reinforcing phases were observed in the Ni–Cr matrix of Inconel 718. Table 1 depicts the chemical compositions of these two reinforcing phases detected by the EDX method. The first category of reinforcement was the exteriorly added TiC$_x$ carbide phase (area 1, figure 6(b)), showing a substoichiometric ratio of Ti and C elements of 1 : 0.6 (table 1). The substoichiometric TiC$_x$ carbides are stable over a large range of carbon contents, from TiC$_{0.50}$ to TiC$_{0.97}$ [27]. The second kind of reinforcement was the significantly refined and well dispersed (Ti,M)$_C$ ($M = \text{Mo, Nb and Cr}$) carbide phase (area 2, figure 6(b)). The atomic ratio of the C element and the Ti, Mo, Nb and Cr metallic elements was near 1 : 1. It was believed that the released Ti and C atoms from TiC$_x$ particles reacted with the Mo, Nb and Cr elements from Inconel 718 during LMD, leading to the in situ formation of (Ti,M)$_C$ carbide reinforcing phase throughout the matrix. As relative to the (Ti,M)$_C$ interfacial graded layer (figures 3(f) and 4), the chemical concentrations of Mo and Nb elements in the in situ carbide reinforcing phase showed a slight increase (table 1). Meanwhile, it was noted that the microstructural features of the two categories of reinforcements were significantly influenced by the applied LED. At a relatively low LED of 280 J mm$^{-3}$, the TiC$_x$ reinforcing particles had a relatively large and irregular shape and the distribution density of the in situ formed (Ti,M)$_C$ carbide reinforcing phase was comparatively low (figure 5(a)), implying the insignificant melting of TiC particle surface and the attendant limited formation of in situ (Ti,M)$_C$ reinforcing phase. As the LED increased to 420 J mm$^{-3}$, the microstructure of LMD-processed composites became more homogeneous. The TiC$_x$ reinforcing particles were refined in the size and the in situ (Ti,M)$_C$ reinforcing phase was well developed into a dense network distribution (figure 5(b)). At an even higher LED of 490 J mm$^{-3}$, although the TiC$_x$ reinforcing particles underwent an apparent smoothening and refinement in their morphology, the in situ (Ti,M)$_C$ reinforcing phase aggregated severely, thereby producing a heterogeneous composite structure in this instance (figure 5(c)).

In order to further study the microstructural characteristics of the particle/matrix interface and the in situ formed (Ti,M)$_C$ carbide reinforcing phase, high-magnification FE-SEM analysis was performed, as shown in figures 6 and 7, respectively. As a relatively low LED of 280 J mm$^{-3}$ was applied, the in situ (Ti,M)$_C$ carbide reinforcing phase was dispersed sparsely within the matrix, exhibiting a considerably refined, fish-bone type structure with a size below 2 $\mu$m (figure 7(a)). Furthermore, it was noted that the interfacial graded layer between the TiC$_x$ reinforcing particles and the matrix, as disclosed on the polished cross-section of LMD-processed composites (figure 3(b)), was well maintained after etching treatment of the sample (figure 6(a)), implying the strong bonding ability of the graded interfacial layer with the reinforcing particles. On enhancing the LED to 420 J mm$^{-3}$, a larger amount of in situ (Ti,M)$_C$ carbide reinforcing phase was developed, leading to a dense distribution throughout the matrix (figure 6(a) versus (b)). The in situ formed
carbide remained in a refined fish-bone-type microstructure, with crystalline size of \( \sim 2 \mu m \) (figure 7(b)). In this instance, the graded interfacial layer was also maintained in the etched sample, acting as a coherent and efficient transition area between the TiC\(_x\) reinforcing particles and the matrix (figure 6(b)). At an even higher LED of 490 J mm\(^{-3}\), the considerably coarsened \textit{in situ} (Ti,M)C carbide phase with crystalline size larger than 5 \( \mu m \) was present, showing a severe aggregation of the reinforcement in LMD-processed composite structure (figure 7(c)). As to the interface, although an interfacial graded layer with relatively high thickness was formed between the TiC\(_x\) particles and the matrix (figure 3(f)), the interfacial layer was not well retained in the etched sample (figure 6(c)), which indicated the relatively weak bonding coherence of the interfacial layer with the reinforcing particles.

### 3.3. Wear performance—COF and worn surface morphology

The variations of COFs and wear rates of LMD-processed TiC/Inconel 718 composites with the applied LED are depicted in figure 8. For comparison, the pure Inconel 718 parts without any reinforcement were prepared using the LED of 420 J mm\(^{-3}\) and the sliding wear tests on the Inconel 718 bulk-form samples were performed under the same test conditions. Generally, the LMD-processed TiC/Inconel 718 composite parts had much lower COFs (the average value below 0.55) as relative to the LMD-processed pure Inconel 718 alloy parts (the mean COF value of \( \sim 0.75 \)) and the conventionally processed Inconel 718 alloy without any surface treatment (the average COF value of \( \sim 1.1 \)) [28]. Furthermore, it was noted that the local fluctuation of the COFs of composite parts was apparently smaller than that of pure Inconel 718 part (figure 8(a)), which indicated the significantly elevated wear performance of composite parts. Meanwhile, the LED played an important role in affecting the wear performance of TiC/Inconel 718 composites. The mean COF and attendant wear rate of the part prepared at a low LED of 280 J mm\(^{-3}\) were comparatively high, reaching 0.52 \( \times 10^{-4} \) mm\(^3\) N\(^{-1}\) m\(^{-1}\) and 3.2 \( \times 10^{-4} \) mm\(^3\) N\(^{-1}\) m\(^{-1}\), respectively. On increasing the LED to 420 J mm\(^{-3}\), the average COF and wear rate decreased markedly to 0.38 \( \times 10^{-4} \) mm\(^3\) N\(^{-1}\) m\(^{-1}\) and 1.8 \( \times 10^{-4} \) mm\(^3\) N\(^{-1}\) m\(^{-1}\), respectively, showing 27% and 44% decrease upon the part at 280 J mm\(^{-3}\). At an even higher LED of 490 J mm\(^{-3}\), however, the mean COF of the part increased slightly to 0.42, increasing the resultant wear rate to 2.2 \( \times 10^{-4} \) mm\(^3\) N\(^{-1}\) m\(^{-1}\). By comparison, the LMD-processed composite part using the LED of 420 J mm\(^{-3}\) demonstrated the superior wear performance. Furthermore, it was interesting to mention that in the initial stage of sliding tests, the COFs of composite parts showed a certain degree of fluctuation and the COF values were relatively higher. With the sliding prolonged, the uniform distributions of COFs with considerably low values were obtained for the composites prepared at 420 and 490 J mm\(^{-3}\) (figure 8(a)). Furthermore, a close comparison revealed that the average wear rate of the LMD-processed pure Inconel 718 alloy parts during sliding
Figure 6. FE-SEM images showing the interfacial microstructures after etching treatment in LMD-processed TiC/Inconel 718 composites at various LEDs: (a) LED = 280 J mm$^{-3}$; (b) LED = 420 J mm$^{-3}$; (c) LED = 490 J mm$^{-3}$.

Table 1. EDX elemental analysis within areas 1 and 2 in figure 6(b), showing the chemical compositions of two different categories of reinforcing phases.

<table>
<thead>
<tr>
<th>Position</th>
<th>Category of reinforcement</th>
<th>Collected elements</th>
<th>Weight fraction (wt%)</th>
<th>Atomic fraction (at%)</th>
<th>Error (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area 1, figure 6(b)</td>
<td>The added TiC reinforcing particles</td>
<td>Ti</td>
<td>86.97</td>
<td>62.60</td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>C</td>
<td>13.03</td>
<td>37.40</td>
<td>2.3</td>
</tr>
<tr>
<td>Area 2, figure 6(b)</td>
<td>The $\textit{in situ}$ formed reinforcement</td>
<td>Ti</td>
<td>32.38</td>
<td>25.05</td>
<td>1.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mo</td>
<td>24.60</td>
<td>9.50</td>
<td>1.6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Nb</td>
<td>18.69</td>
<td>7.45</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>C</td>
<td>17.28</td>
<td>53.27</td>
<td>3.3</td>
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<td>Ni</td>
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<td>2.26</td>
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<tr>
<td></td>
<td></td>
<td>Cr</td>
<td>3.47</td>
<td>2.47</td>
<td>0.3</td>
</tr>
</tbody>
</table>

tests was $6.1 \times 10^{-4}$ mm$^3$ N$^{-1}$ m$^{-1}$. The LMD-processed TiC/Inconel 718 composite parts generally had an apparently decreased COF and wear rate due to the incorporation of ceramic reinforcement in the matrix.

The characteristic morphologies of the worn surfaces of TiC/Inconel 718 composite parts at various LED are revealed in figure 9. At the relatively low LED of 280 and 350 J mm$^{-3}$, the worn surface was considerably rough, consisting of parallel grooves and granular wear debris (figures 9(a) and (b)). Such a microstructural feature illustrated that the specimen suffered severe abrasive wear during sliding tests, which in turn resulted in the relatively high wear rate (figure 8(b)). As an elevated LED of 420 J mm$^{-3}$ settled properly, the worn surface of LMD-processed TiC/Inconel 718 composite part became rather smooth. A continuously adherent, strain-hardened tribolayer was formed on the worn surface, without any significant fracturing or local plowing of the surface (figure 9(c)). It was accordingly reasonable to consider that as the LED increased, the mechanism of material removal during sliding tests changed from the abrasion to the adhesion of tribolayer. Such a transition was effective in stabilizing the COF and hence reducing the wear rate of LMD-processed composites, as revealed in figure 8. As the LED was further enhanced to 490 J mm$^{-3}$, although the worn surface was still remained dense and free of the apparent sliding grooves, the tribolayer on the surface was fragmented and the entrapped small-sized debris were formed, producing a rough worn surface (figure 9(d)) and resultant lower wear performance (figure 8).
4. Discussion

4.1. Densification and particle dispersion behaviour during LMD of composites

During LMD process, the high-energy laser beam creates a moving molten pool on the substrate into which the mixed TiC/Inconel 718 powder is injected. The Inconel 718 component, due to the relatively low melting temperature range of 1260–1336 °C, experiences a complete melting within the molten pool, producing a liquid/solid composite system containing TiC reinforcing particles. The cores of TiC particles, due to the considerably high melting point of 3067 °C, remain in solid state. With the sufficient wetting of the surrounding liquid in the pool, the edges of the starting irregularly shaped TiC particles become melted and, meanwhile, the free Ti and C atoms are released in the pool. According to [29], the viscosity of a solid–liquid mixture (µ) is dependent on LMD temperature (T) and is inversely proportional to T. As the applied LED increases, the operative T elevates within the pool, which in turn lowers the solid–liquid mixture viscosity µ. Accordingly, the rheological properties of the liquid in conjunction with solid reinforcing particles are elevated. In this situation, the capillary forces that are responsible for melt flow are easier to exert on the reinforcing particles by the surrounding liquid, leading to a sufficient wetting of the solid particles by the liquid. Therefore, an increase in the LED favours the significant smoothening and refinement of the initially poly-angular TiC reinforcing particles (figures 3(a), (c) and (e)). Furthermore, the rearrangement rate of the reinforcing particles is elevated under the influence of capillary forces exerted on them by the wetting liquid, favouring a high densification activity of the composite system (figure 1) without the apparent aggregation of reinforcing particles (figure 2(c)) after solidification of the molten pool.

On the other hand, either chemical concentration or temperature gradient at the solid–liquid interfaces within the molten pool will generate the surface tension gradients and resultant Marangoni flow. The microscopic distribution of the reinforcing particles mainly depends on the interaction between the solid particles and the dynamic Marangoni flow. Based on [30], the intensity of Marangoni flow is inversely proportional to µ and, accordingly, a lower solid–liquid mixture viscosity obtained at a higher LED leads to an intensified Marangoni flow. The significant operation of the convective Marangoni flow within the pool accelerates the rearrangement of the solid particles, leading to a uniform distribution of the reinforcing particles without the local segregation in the regions finally solidified (figure 2(c)).

4.2. Formation mechanisms of multiple reinforcement and graded interfacial layer

Figure 10 depicts schematically the development mechanisms of the in situ formed reinforcing phase and interfacial graded layer in LMD-processed TiC/Inconel 718 composites. Due
to the melting of surfaces of TiC particles, the Ti and C atoms release from the melted surface and diffuse actively throughout the molten pool. The presence of the substoichiometric TiC$_x$ reinforcing particles after LMD (figure 6(b) and table 1) indicates the decomposition and diffusion of Ti and C elements from the particles to the melt. As the Inconel 718 melts completely during LMD, the main alloying elements Nb, Mo and Cr elements also become active in the pool. The Nb, Mo and Cr are the strong carbide-forming elements \[26\] and, therefore, they tend to react with the Ti and C elements on the surfaces of TiC particles to form the (Ti,$M$)C ($M$ = Mo, Nb and Cr) carbide interfacial graded layer between the reinforcing particles and the matrix (figures 2 and 3). The development of the (Ti,$M$)C reinforcing phase is identified as the in situ (Ti,$M$)C (figure 7(b)). Also, it is noted that the applied LED plays a key role in the crystal development of the in situ (Ti,$M$)C reinforcing phase (figure 7). An increase in LED tends to elevate the temperature of the molten pool markedly and, accordingly, a large amount of heat is accumulated around the tips of the growing (Ti,$M$)C crystals, which in turn provides significant internal energy and thermodynamic potentials for the coarsening of the finally developed in situ reinforcing phase (figure 7(c)).

Besides TiC$_x$ reinforcing particles and (Ti,$M$)C interfacial graded layer (figures 2 and 3), the second category of reinforcing phase is identified as the in situ (Ti,$M$)C ($M$ = Mo, Nb and Cr) carbide phase having relatively high concentrations of Mo and Nb elements (figure 7 and table 1). The reinforcement is in situ formed via a dissolution-precipitation mechanism by means of the heterogeneous nucleation of carbide nuclei and subsequent grain growth. Since LMD process has a highly non-equilibrium rapid solidification nature \[31, 32\] and the cooling rate can reach a high value of $10^6 \text{Ks}^{-1}$ \[33, 34\], the in situ (Ti,$M$)C reinforcing phase has an insufficient time and a limited space for grain growth, hence refining the crystalline size considerably as a reasonable LED is applied (figure 7(b)). Furthermore, different to the (Ti,$M$)C interfacial layer formed on TiC particle surfaces, the second kind of (Ti,$M$)C reinforcing phase is in situ developed within the matrix of Inconel 718, where the Mo and Nb elements, as the main alloying elements, have a relatively high concentration. Therefore, the contents of Mo and Nb elements detected in the in situ (Ti,$M$)C reinforcement (figure 6(b)) are relatively high (figure 4 versus table 1, figure 10). Also, it is noted that the applied LED plays a key role in the crystal development of the in situ (Ti,$M$)C reinforcing phase (figure 7). An increase in LED tends to elevate the temperature of the molten pool markedly and, accordingly, a large amount of heat is accumulated around the tips of the growing (Ti,$M$)C crystals, which in turn provides significant internal energy and thermodynamic potentials for the coarsening of the finally developed in situ reinforcing phase (figure 7(c)).

4.3. Relationship of microstructural characteristics and wear behaviour

In order to disclose the underlying mechanisms contributing to the enhancement of wear performance of TiC/Inconel 718 composites, a comprehensive relationship of SLM process, microstructures, and mechanical properties is determined. Firstly, the dispersion homogeneity of the reinforcing particles and resultant densification rate of composites plays a basic role in determining the wear performance. At the relatively low LED, the TiC reinforcing particles remain the initial poly-angular structure (figure 3(a)) and aggregate in local areas of the matrix (figure 2(a)). In this situation, the densification response of the composites after LMD is limited (figure 1(a)), resulting in a significant increase in COF and attendant wear rate (figure 8). Also, the non-uniform dispersion of TiC reinforcing particles is responsible for the apparent fluctuation of COF values during sliding test of the composites processed at a low LED of 280 J mm$^{-3}$ (figure 8(a)). On the other hand, it is revealed from figure 1(b) that the surface property is strongly influenced by the applied LED. Meanwhile, the surface property may influence the material behaviour during sliding tests and resultant wear and tribological performance. Either the insufficient laser energy input or the excessive laser energy input tends to make the laser-processed surface roughened and loosened (figure 1(b)). The sliding tests on these surfaces may result in the fluctuation of COF values and attendant high wear rate because of the unfavourable surface modification.
Figure 9. FE-SEM images showing the typical morphologies of worn surfaces of LMD-processed TiC/Inconel 718 composite parts at various LEDs: (a) LED = 280 J mm$^{-3}$; (b) LED = 350 J mm$^{-3}$; (c) LED = 420 J mm$^{-3}$; (d) LED = 490 J mm$^{-3}$.

Secondly, the tailored formation of the interfacial graded layer between the reinforcing particles and the matrix improves the wear performance. The reinforcement/matrix interfaces are the weakest areas within the composites and, thus, demonstrate a high tendency for the pore formation or even cracking during sliding. The present study reveals that as the (Ti,$M$)C interfacial graded layer is formed and tailored during LMD, the TiC reinforcing particles and the matrix are coherently bonded without the formation of any interfacial pores or cracks (figures 3(b), (d) and (f)), thereby strengthening the interfaces within the composites. It is believed that for the strengthened interface itself, it is not easy to split during sliding. More important, such a graded interlayer layer is able to modulate the different deformation behaviours of the TiC reinforcing particles and the Ni–Cr matrix during sliding, yielding a stable wear behaviour with the even distribution of COF values and considerably decreased wear rate (figure 8).

Thirdly, the development of the uniformly dispersed, novel structured and considerably refined in situ (Ti,$M$)C reinforcing phase in the matrix (figures 5(b) and 7(b)) further enhances the wear resistance. During sliding tests, the counterface ball slides against the surface continuously. The worn surface experiences sufficient plastic deformation at a temperature below its recrystallization temperature. The material strengthening occurs because of the dislocation movements within the crystal structure of the material, typically in the sliding-treated layer, which is known as strain-hardened tribolayer, as experimentally disclosed in figure 9(c). The homogeneous and refined in situ (Ti,$M$)C reinforcement (figures 5(b) and 7(b)) is not easy to spall during sliding, but has a high tendency to adhere to each other and get strain-hardened, favouring the complete smearing of the protective tribolayer on the worn surface to enhance wear resistance (figure 9(c)). However, using an excessive LED of 490 J mm$^{-3}$ produces a heterogeneous composite structure (figure 5(c)) consisting of the apparently coarsened in situ (Ti,$M$)C reinforcing phase (figure 7(c)). The coarsening of crystals of reinforcement and resultant decrease in microstructural uniformity are...
responsible for the spalling of tribolayer (figure 9(d)) and, accordingly, a decrease in the obtainable wear performance (figure 8).

5. Conclusions

(1) The densification activity of TiC/Inconel 718 composite parts processed by laser metal deposition (LMD) was controlled by the applied laser energy density (LED). Using a LED of 280 J mm\(^{-3}\) produced ~5% porosity in LMD-processed composites, caused by the aggregation of reinforcing particles. On increasing the LED above 350 J mm\(^{-3}\), near-full densification larger than 98.5% theoretical density was achieved.

(2) Two categories of reinforcing phases were formed in the matrix of LMD-processed TiC/Inconel 718 composites, i.e. the exteriorly added TiC\(_x\) particles having a substoichiometric ratio of Ti and C elements and the \textit{in situ} formed (Ti,M)\(_x\)C (M = Mo, Nb and Cr) carbide having 7–10 at% Nb and Mo contents. The TiC\(_x\) reinforcing particles changed from an irregular poly-angular shape to a smoothened and refined structure as the LED increased. An increase in LED resulted in a larger amount of formation of \textit{in situ} (Ti,M)\(_x\)C reinforcing phase and an enhanced degree of crystal growth.

(3) The interfacial graded layer with the thickness of 0.2–1.2 \(\mu\)m, which was identified as (Ti,M)\(_x\)C (M = Mo, Nb and Cr) carbide with 5–6 at% Mo and Nb contents, was tailored between the TiC\(_x\) reinforcing particles and the matrix, due to the reaction of the strong carbide-forming elements Mo, Nb and Cr with the released Ti and C elements on the melted surfaces of TiC particles.

(4) At the optimal LED of 420 J mm\(^{-3}\), a considerably low average COF of 0.38 without any apparent fluctuation of COF values was obtained in sliding wear tests, decreasing the wear rate significantly to 1.8 \(\times\) 10\(^{-3}\) mm\(^2\) N\(^{-1}\) m\(^{-1}\). The combined strengthening of the interfacial graded layer and the multiple reinforcing phases favoured the formation of adherent strain-hardened tribolayer covered on the worn surface and, accordingly, enhanced the wear performance. However, the wear resistance decreased at an excessive LED because of the coarsening of reinforcement crystals and the decrease in microstructural uniformity.

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