Laser additive manufacturing of ultrafine TiC particle reinforced Inconel 625 based composite parts: Tailored microstructures and enhanced performance

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**A B S T R A C T**

Laser metal deposition (LMD) additive manufacturing process was applied to produce ultrafine TiC particle reinforced Inconel 625 composite parts. The effects of laser energy input per unit length (LEIPUL) on microstructure development, densification response, and mechanical performance including wear performance and tensile properties were comprehensively studied. A relationship of processing conditions, microstructural characteristics, mechanical performance, and underlying strengthening mechanisms was proposed for a successful LMD of high-performance Inconel based composite parts. It revealed that using an insufficient LEIPUL of 33 kJ/m lowered the densification behavior of LMD-processed parts, due to the appearance of residual large-sized pores in inter-layer areas of the parts. An increase in LEIPUL above 100 kJ/m yielded the near fully dense composite parts after LMD. On increasing LEIPUL, the TiC reinforcing particles became significantly refined and smoothened via the elevated melting of particle surfaces and the dispersion state of ultra-fine reinforcing particles was homogenized due to the efficient action of Marangoni flow within the molten pool. The dendrites of Ni–Cr–γ matrix underwent a successive change from an insufficiently developed, disordered microstructure to a refined, ordered microstructure with the increase of LEIPUL. However, the columnar dendrites of the matrix were coarsened apparently at an excessive LEIPUL of 160 kJ/m because of the elevated thermalization of the input laser energy. The formation of the refined columnar dendrites of Ni–Cr–γ matrix combined with the homogeneously distributed ultra-fine reinforcing particles contributed to the enhancement of wear performance of LMD-processed composites with a considerably low coefficient of friction (COF) of 0.30 and reduced wear rate of $1.3 \times 10^{-14}$ mm$^3$/N m. The optimally prepared TiC/Inconel 625 composite parts demonstrated a ductile fracture mode with a sufficiently high tensile strength of 1077.3 MPa, yield strength of 659.3 MPa, and elongation of 20.7%. The superior tensile properties of LMD-processed parts were attributed to the significant grain refinement effect of the matrix during laser processing and the efficient prohibition of ultrafine reinforcing particles on the mobility of dislocations.

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1. Introduction

In recent years, the demand for production of high-performance industrial components with geometrically complex structures has promoted the development of additive manufacturing (AM) (also known as 3D printing, 3DP) technology. Unlike the conventional material machining methods, AM/3DP possesses the advantages of creating dense parts directly from the feedstock (typically powder materials) in an entirely opposite philosophy, i.e., material incremental manufacturing [1–5]. Due to the high freedom of geometrical design and process manipulation, AM/3DP exhibits the outstanding capability of net-shaping user-defined three-dimensional parts having arbitrary configurations [6–10]. Laser metal deposition (LMD) (also known as laser engineered net
shaping, LENS\cite{15}, as a typical AM/3DP process combining laser cladding and rapid prototyping, has demonstrated its high feasibility in (i) rapid manufacturing and repairing/re-manufacturing parts with complex geometries; and (ii) preparing coatings to modify surface properties of components\cite{16,17,18,19}. During the LMD process, a high-energy laser beam is focused onto the top surface of the work piece to create a molten pool, wherein the powder delivered by the inert gas is steadily injected inside through a coaxial nozzle. The cross-sectional geometry of each layer of the desired part is built as the work piece moves in the X–Y direction by a computer-controlled driving system. The component is then created in a layer-by-layer fashion along the Z direction by means of the unique development method from 2D coating to 3D manufacturing. To date, a wide range of wide range of Ti-based alloys, e.g., Ti–Ni-based stainless steel and tool steel\cite{20,21}, and Ni-based superalloys\cite{22,23}, have been processed successfully by LMD for manufacturing complex-shaped parts.

Furthermore, recent studies have proved that the LMD process, due to its unique AM production manner and laser-induced non-equilibrium rapid melting/solidification mechanism, has a promising potential in net-shaping metal matrix composites (MMCs) parts with tailored microstructures and high performance. Schoenung et al.\cite{24,25} have produced the dense WC–Co and (Ti,W)C–Ni MMCs parts from nanostructured WC–Co and (Ti,W)C–Ni powder by using the LENS AM process. Both theoretical and experimental researches have been carried out to study the thermal behavior of the LENS process and the mechanisms for the development of microstructural and mechanical properties of LENS-processed composites. Lavernia et al.\cite{26,27} have deposited IN625-based and Ti6Al4V-based MMCs using LENS with Ni-coated TiC reinforcement particles. The integrity of the interface between matrix and TiC particles and the mechanical properties of LENS-deposited MMCs are generally improved effectively by using Ni-coated TiC particles. Bandyopadhyay et al.\cite{28,29} have applied the LENS process to create Ti–SiC composites on Ti to improve its wear resistance. Laser parameters have been found to have a strong influence on the dissolution of SiC, leading to the in-situ formation of Ti5Si3 and TiC with a high amount of SiC on the surface. In Banerjee et al.’s work\cite{30}, MMCs consisting of a complex quaternary Ti–35Nb–7Zr–5Ta alloy reinforced by borides have been deposited from a blend of Ti, Nb, Zr, Ta, and TiB2 powders by the LENS process. Wu et al.\cite{31} have fabricated Ti6Al4V/TiB MMCs using the LMD process by injection of premixed powders of TiB2 and Ti6Al4V. The modulus, yield and ultimate strength of Ti6Al4V are increased by the TiB reinforcement, but the ductility is decreased.

Nickel-based superalloys are widely used in applications like turbine blades and engine components where the combination of superior mechanical property and excellent workability are required. Inconel 625, a Ni–Cr–based austenite superalloy, is featured by an improved balance of creep performance, fatigue strength, tensile properties, and oxidation resistance, making it an attractive choice for diverse industrial applications. As a typical material for hot-end components, Inconel 625 has the merits of maintaining its outstanding mechanical strength and corrosion resistance at both moderate service temperature or up to 650 °C \cite{32,33,34,35}. The favorable high-temperature performance is controlled mainly by the inherent solid-solution strengthening effect of the refractory metal elements such as niobium and molybdenum in Ni–Cr matrix. Nevertheless, with the rapid development of modern industry, Inconel 625 parts with higher performance are on increasing demand as the working conditions of components become even rigorous in search for a greater efficiency.

In order to further enhance the service performance of Inconel 625 parts, e.g., the elevated strength and wear/tribological resistance, the incorporation of hard and temperature-resistant ceramic particles within the matrix to produce MMCs is expected to create new technological opportunities for modern industrial applications. MMCs reinforced with discontinuous ceramic reinforcements have received extensive research interest within the last decades due to their sound high-temperature creep behavior, hot corrosion resistance, wear resistance, etc. Conventionally, MMCs parts are produced by powder metallurgy or liquid metal processing methods, in which the insufficient densification response and inhomogeneous microstructures are most likely to occur. Therefore, the application of novel non-traditional processing technology is necessary to produce MMCs with the substantial enhancement of mechanical properties. On the other hand, the limited wetting characteristics between ceramic reinforcing phases and metal matrix are regarded as a significant obstacle in achieving the coherent interfacial structures and sufficiently high mechanical performance. It has been found that the improvement in the mechanical properties of MMCs is influenced by the size and distribution state of the reinforcement. Refining the reinforcing particle size can lead to the simultaneous enhancement of strength and ductility of MMCs \cite{36,37,38,39,40,41,42,43}.

In our previous work\cite{38,39}, the LMD process was applied to produce TiC/Inconel 718 composites using the relatively large micron-sized TiC reinforcing particles with the size distribution of 22.5–45 μm. In order to improve the interfacial wettability and bonding coherence, the in-situ interfacial reaction was tailored between TiC reinforcing particles and Ni-based metal matrix. For mechanical properties, the hardness and wear resistance of LMD-processed TiC/Inconel 718 composites were studied. It was found that with an optimization of LMD processing conditions, the LMD-processed composites demonstrated a considerably low coefficient of friction and resultant low wear rate in sliding tests, due to the combined strengthening of in-situ interfacial layer and multiple reinforcing phases. In the present study, the TiC particles with a considerably reduced size distribution were used to reinforce Inconel 625 alloy. The LMD process was applied to prepare ultrafine TiC particle reinforced Inconel 625 MMCs parts, which demonstrated the significantly different microstructural and mechanical properties as relative to the Inconel based composites reinforced with large-sized ceramic particles. The effects of laser energy input per unit length (LEIPUL) on the microstructure development, densification response, and mechanical performance including wear performance and tensile properties are comprehensively studied. In particular, the tensile performance of LMD-processed Inconel based MMCs has never been studied previously in the existing literature. A relationship between processing conditions, microstructural characteristics, and mechanical performance was proposed for a successful LMD of Inconel based MMCs. The underlying strengthening mechanisms were elucidated to obtain high-performance LMD-processed MMCs parts.

2. Experimental procedures

2.1. Powder preparation

The gas atomized, spherical Inconel 625 powder with the particle size distribution of 45–95 μm (supplier: Sulzer Metco) (Fig. 1a) and the irregular shaped TiC powder with the refined particle size distribution of 5–7 μm (supplier: ABCR GmbH) (Fig. 1b) were used as the starting materials. The nominal chemical compositions of Inconel 625 powder and also 5 wt% TiC powder were 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 8 h. The refined TiC particles were dispersed homogeneously around Inconel 625 particle surface after ball milling (Fig. 1c). Meanwhile, Inconel 625 powder particles did not experience any apparent

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deformation and structural change during the milling process. A sound flowability of the mixed TiC/Inconel 625 composite powder was accordingly maintained, which was particularly important for a successful LMD process.

2.2. Laser processing

The LMD system consisted of a Trumpf Nd:YAG laser system with a maximum output power of 3 kW, a powder feeding system, a 5-axis CNC machine, and a standard optics equipped with a coaxial nozzle. The TiC/Inconel 625 composite parts were built by depositing the melted powder onto the prepared C45 carbon steel substrate in a powder feeding rate of 2.4 g/min, using argon as the powder carrier gas as well as the shielding gas to prevent the melt pool from oxidation. The multiple tracks were cladded for each layer and multiple layers were deposited on the substrate to produce the desired 3D parts with a height of 30 mm. For the LMD process, the main parameters including laser spot diameter, laser power, and laser scan speed played a significant role in determining microstructures and resultant mechanical properties of as-fabricated parts. Through a series of preliminary experiments, the laser spot diameter was focused at 1 mm, the laser powers \( P \) were preset periodically at 500, 800, 500, and 800 W, and the scan speeds \( v \) were varied at 900, 900, 300, and 300 mm/min, respectively. Four different “laser energy input per unit length” (LEIPUL) of 33, 53, 100, and 160 kJ/m, which was defined by [38]:

\[
\text{LEIPUL} = \frac{P}{v}
\]

was used to investigate the influence of LMD processing parameters on layer-by-layer deposition process and attendant microstructural and mechanical properties of LMD-processed parts.

2.3. Microstructure characterization and mechanical properties tests

Phase identification of specimens was performed by a Bruker D8 Advance X-ray diffractometer (XRD) with Cu K\( \alpha \) radiation at 40 kV and 40 mA, using a continuous scan mode. The fabricated composite parts

![Fig. 1. Typical morphologies of the starting Inconel 625 powder (a), TiC reinforcing particles (b), and the homogeneously mixed TiC/Inconel 625 composite powder.](image)

![Table 1: Chemical compositions of Inconel 625 alloy powder (in weight fraction, wt%).](table)
were cut by a spark-erosion wire cutting machine to obtain cross-sections. The samples were then inlaid and polished for metallographic observation in line with the standard procedures. The densification behaviors were revealed by using optical microscopy (OM). Microstructures were characterized using a Hitachi S-4800 scanning electron microscopy (SEM) with the samples being etched by a solution containing of HCl (10 ml) and H2O2 (3 ml) for 10 s. EDAX energy dispersive X-ray spectroscopy (EDX) was applied to determine the chemical compositions, using a super-ultra thin window sapphire detector. The characteristics of crystal structures of the LMD-processed samples were further studied by an EDAX high-speed and high-sensitivity Hitari electron backscatter diffraction (EBSD) camera. An EDAX OIM data collection and analysis software was applied to acquire crystallographic data. For specimen preparation for EBSD, the LMD-processed samples were ground with SiC paper and polished with diamond suspension down to 1 μm, followed by 1 h polishing using a Bühler gamma micropolish machine with vibration function. A FEI Tecnai G2 20 S-TWIN transmission electron microscopy (TEM) was used to investigate the interior microstructures and strengthening mechanisms of the samples after tensile tests.

The density (ρ) of the LMD-processed TiC/Inconel 625 composite parts was determined based on the Archimedes’ principle. The dry sliding wear tests on the polished cross-sections of the LMD-processed parts were conducted in a HT-500 ball-on-disc tribometer at room temperature. A 03 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 fixed at 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. The LMD-processed parts were cut using a spark-erosion wire cutting machine to prepare standard specimens for tensile tests, according to the standard ISO 6892-1:2009. The tensile direction was parallel to the laser deposited layers. The uniaxial tensile tests were performed at room temperature with a universal testing machine integrated with extensometer (Zwick Universalprüfmaschine 1488) at a strain rate of \( 2 \times 10^{-3}/s \). The ultimate tensile strength and elongation were further determined from the stress–strain curves.

### 3. Results and discussion

#### 3.1. Constitutional phases

The typical XRD patterns of LMD-processed TiC/Inconel 625 composite parts within a wide range of \( 2\theta = 30–110^\circ \) are depicted in Fig. 2. At all given LEIPUL, the diffraction peaks corresponding to Ni–Cr \( \gamma \) matrix phase and TiC reinforcing phase were clearly identified. In order to exactly investigate the influence of TiC reinforcement on diffraction peaks for Ni–Cr \( \gamma \) phase in the composites, XRD characterization in the vicinity of the 1st \( (2\theta = 43.774^\circ) \) and 2nd \( (2\theta = 50.992^\circ) \) strong peaks for Ni–Cr \( \gamma \) phase was performed, respectively, as depicted in Fig. 3. The quantitative determination of \( 2\theta \) location and intensity of the detected Ni–Cr \( \gamma \) phase are listed in Table 2. The \( 2\theta \) locations of diffraction peaks for Ni–Cr \( \gamma \) phase at various LEIPUL generally shifted to lower \( 2\theta \) angles. According to the Bragg’s law [39]:

\[
2d \sin \theta = n\lambda \quad (n = 1, 2, 3\ldots)
\]

The displacement of \( 2\theta \) to lower Bragg angles indicated an increase in the distance between adjacent lattice planes \( (d) \), which was believed to be caused by the distortion of lattice due to the incorporation of Ti and C atoms derived from TiC particles into the Ni–Cr \( \gamma \) lattice. Furthermore, a close comparison of the intensities of Ni–Cr \( \gamma \) phase at different LEIPUL revealed that as the applied LEIPUL increased from 33 to 100 kJ/m, the diffraction peaks for Ni–Cr \( \gamma \) phase became considerably broadened (Fig. 3) and the intensity showed a significant decrease (Table 2). These phenomena implied that a proper increase in LEIPUL favored the formation of refined crystals and microstructures of Ni–Cr \( \gamma \) phase in LMD-processed composites. However, a further enhancement of LEIPUL resulted in the increase of the intensity of Ni–Cr \( \gamma \) diffraction peaks without the apparent broadening of peaks (Fig. 3 and Table 2), which suggested the coarsening of crystals in this instance with a significantly elevated laser energy input.

#### 3.2. Densification behavior

The characteristic microstructures between neighboring layers on the cross-sections of LMD-processed TiC/Inconel 625 composite parts
under various processing conditions are revealed in Fig. 4. The layerwise microstructural features were generally produced, due to the layer-by-layer incremental manufacturing nature of the LMD process. Fig. 5b depicts the effect of LEIPUL on the average thickness of the deposited layers. On increasing the applied LEIPUL from 33 to 100 kJ/m, the average thickness of the deposited layers increased sharply from 510 to 990 μm; a further enhancement of LEIPUL led to a slight increase of the average layer thickness to 1100 μm (Fig. 5b). It was accordingly concluded that an increase in laser energy input favored the formation of thicker deposited layers during the LMD process. The change of the densification rates of the corresponding parts is depicted in Fig. 5a. At a relatively low LEIPUL of 33 kJ/m, larger-sized residual pores were observed between the adjacent deposited layers (Fig. 4a). The irregularly shaped molten pools with the disorderly arranged solidification front were obtained, leaving the insufficient densification response of 96.8% theoretical density (TD) (Fig. 5a). On increasing LEIPUL to 53 kJ/m, the configuration of the molten pools became clearer and regular, showing coherent bonding between the neighboring tracks and deposited layers (Fig. 4b). In this situation, only a small amount of micro-pores were remained in the deposited layers and the obtained densification level was enhanced to 97.4% TD (Fig. 5a). As LEIPUL increased to 100 kJ/m, the sufficiently strong metallurgical bonding was achieved between the neighboring layers and adjacent scan tracks (Fig. 4c). There were no apparent residual pores formed on the cross-section of LMD-processed part and the obtained densification rate was as high as 99.0% (Fig. 5a). On further enhancing LEIPUL to 160 kJ/m, the relative density of LMD-processed part slightly increased to 99.5% (Fig. 5a), showing the formation of the metallurgically bonded layers free of any micro-pores and micro-cracks (Fig. 4d).

During the LMD process, a moving molten pool was created by high-energy laser beam, in which the mixed TiC/Inconel 625 composite powder is injected inside. The sound bonding property obtained in LMD-processed tracks and layers is guaranteed by the effective diluting of the previously solidified materials and the favorable wetting of the freshly deposited molten materials with neighboring layers and adjacent scanning tracks. Therefore, a sound metallurgical quality of LMD-processed parts can only be obtained under the coordination of processing parameters, such as laser power, laser scan speed, powder feed rate, etc. In the present study, the integrated parameter LEIPUL proves to be useful in controlling inter-track/inter-layer metallurgical bonding and resultant densification activity of LMD-processed parts. At a relatively low LEIPUL, the operative temperature in the molten pool is low, due to the insufficient thermalization of the absorbed energy from laser beam. A limited amount of liquid is generated in the molten pool and then solidifies immediately as laser beam moves away, thereby restricting the complete spreading of the molten materials on the previously deposited tracks and layers. It accordingly results in the formation of relatively thin deposited layers with the residual pores in the bonding areas of adjacent tracks and layers (Figs. 4a and 5).

Table 2
XRD data showing displacement and intensity variations of identified peaks of Ni–Cr γ phase in LMD-processed TiC/Inconel 625 composite parts.

<table>
<thead>
<tr>
<th>Samples and processing parameters</th>
<th>1st strong peak</th>
<th>2nd strong peak</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2θ (deg)</td>
<td>Intensity (CPS)</td>
</tr>
<tr>
<td>Standard</td>
<td>43.774</td>
<td>-</td>
</tr>
<tr>
<td>P = 500 W, v = 900 mm/min, LEIPUL = 33 kJ/m</td>
<td>43.58</td>
<td>4644.3</td>
</tr>
<tr>
<td>P = 500 W, v = 300 mm/min, LEIPUL = 100 kJ/m</td>
<td>43.56</td>
<td>2174.7</td>
</tr>
<tr>
<td>P = 800 W, v = 300 mm/min, LEIPUL = 160 kJ/m</td>
<td>43.52</td>
<td>2339.6</td>
</tr>
</tbody>
</table>

Fig. 4. OM images showing the molten pool configuration and densification behavior of the deposited layers in LMD-processed TiC/Inconel 625 composite parts using different LMD processing parameters: (a) P = 500 W, v = 900 mm/min, LEIPUL = 33 kJ/m; (b) P = 800 W, v = 900 mm/min, LEIPUL = 53 kJ/m; (c) P = 500 W, v = 300 mm/min, LEIPUL = 100 kJ/m; and (d) P = 800 W, v = 300 mm/min, LEIPUL = 160 kJ/m.
sufficiently high LEIPUL was applied for LMD, the significantly elevated working temperature in the molten pool yields a large amount of liquid formation by means of the complete melting of matrix metal. Either chemical concentration gradient or temperature gradient at the solid–liquid interface may generate surface tension gradient and resultant Marangoni flow, thereby inducing capillary forces for liquid flow in conjunction with the reinforcing particles [40,41]. The formation of the strong stirring behavior in the molten pool induced by Marangoni flow provides the significant driving forces for the complete spreading of the molten materials on the previously processed track and layer, thereby increasing the wetting characteristics of liquid–solid system and resultant densification response of LMD-processed composite parts (Figs. 4c, d, and 5a). Therefore, it is reasonable to conclude that the operating temperature during the LMD process and the attendant liquid spreading and wetting behavior in the molten pool play a key role in determining the configuration of molten pool and densification activity of LMD-processed composite parts.

3.3. Microstructures and compositions

Fig. 6 illustrates the influence of LEIPUL on microstructural evolutions of LMD-processed TiC/Inconel 625 composites observed at a relatively low magnification. High-magnification characterization was also carried out on specific positions to reveal microstructures of the composites, as shown in Fig. 7. At a lower LEIPUL of 33 kJ/m, the insufficiently developed columnar dendrites were present in LMD-processed composites, showing a considerably heterogeneous microstructure (Fig. 6a). High-magnification observation revealed that the incorporated reinforcing particles having an original irregular shape experienced a severe agglomeration during solidification (Fig. 7a). On increasing LEIPUL to 53 kJ/m, the disorderly arranged columnar dendrites in a fragmentized feature were obtained in LMD-processed part.
(Fig. 6b). The reinforcing particles were smoothened on their surfaces and, meanwhile, became dispersed in a microscopic scale (Fig. 7b). As LEIPUL was further elevated to 100 kJ/m, the crystallized columnar dendrites became uniformly distributed and the primary dendrites could be clearly distinguished (Fig. 6c). The surface of TiC reinforcing particles became apparently smoothened and the attendant dispersion state became much more homogeneous throughout the matrix (Fig. 7c). Furthermore, it was interesting to note that the average diameter of TiC reinforcing particles was significantly refined in the scale of several hundred nanometers (Fig. 7c), which indicated the partial dissolution of the starting micro-sized TiC particles (5–7 μm) during the LMD process. On further enhancing LEIPUL to 160 kJ/m, the severely coarsened columnar dendrites were present in LMD-processed structures (Fig. 6d), even though the TiC reinforcing particles were still refined and homogeneously distributed in the matrix (Fig. 7d). Table 3 depicts the EDX analysis results of the chemical compositions in the inter-dendrite matrix in LMD-processed TiC/Inconel 625 composites at various LEIPUL. The contents of matrix elements (Ni and Cr) and main alloying elements (Mo, Nb, and Fe) of Inconel 625 did not exhibit any significant change with the variations of LEIPUL. However, as LEIPUL increased from 33 above 100 kJ/m, the identified content of C element dissolved in the matrix showed at least double increase. It was noted that a large fraction of incorporated TiC particles became melted on surfaces at a relatively high LEIPUL larger than 100 kJ/m (Fig. 7c and d) and the released Ti and C atoms tended to dissolve in the matrix to form the supersaturated structures.

It is found that the applied LEIPUL plays a crucial role in determining distribution and morphology of TiC reinforcement in LMD-processed composites. Using an insufficient LEIPUL ranging from 33 to 53 kJ/m results in the formation of the severe aggregation of TiC reinforcing particles having a poly-angular morphology (Fig. 7a and b). A reasonable increase in LEIPUL to 100 kJ/m has the capacity to homogenize the distribution state of reinforcing particles with a significant smoothening of particle surfaces (Fig. 7c). However, care should be taken to control LEIPUL, since the columnar dendrites of the matrix have a high tendency to become coarsened as the excessive LEIPUL of 160 kJ/m is applied (Fig. 7d). It has been disclosed that the rearrangement and attendant distribution of the TiC reinforcing particles are highly dependent on the thermal capillary force induced by the Marangoni convection within the molten pool. Based on the theory established by Arafune and Hirata [42], the dimensionless Marangoni number ($M_d$) can be used to define and evaluate the intensity of Marangoni flow:

$$M_d = \frac{\Delta \sigma L}{\mu_d V_k} \quad (3)$$

where $\Delta \sigma$ is the surface tension difference of Marangoni flow (N/m), $L$ the length of free surface, $\mu_d$ the dynamic viscosity (Pa s), and $V_k$ is the kinematic viscosity (m$^2$/s). Eq. (3) reveals that $M_d$ is inversely proportional to $\mu_d$, which is temperature dependant and, normally, a higher working temperature leads to a lower $\mu_d$ [43]. At a relatively high LEIPUL, the operative temperature within the molten pool elevates apparently, lowering the viscosity $\mu_d$ and accordingly intensifying the Marangoni flow. The liquid capillary forces within

<table>
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<th>Position</th>
<th>Element (at%)</th>
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<tbody>
<tr>
<td>Ni 62.50</td>
<td>Cr 21.00</td>
</tr>
<tr>
<td>Ni 55.21</td>
<td>Cr 24.21</td>
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<tr>
<td>Ni 50.04</td>
<td>Cr 20.02</td>
</tr>
<tr>
<td>Ni 57.95</td>
<td>Cr 20.89</td>
</tr>
</tbody>
</table>

**Fig. 7.** High-magnification FE-SEM images showing composite structures of LMD-processed TiC/Inconel 625 parts using (a) $P=500$ W, $v=900$ mm/min, LEIPUL=33 kJ/m; (b) $P=800$ W, $v=900$ mm/min, LEIPUL=53 kJ/m; (c) $P=500$ W, $v=300$ mm/min, LEIPUL=100 kJ/m; and (d) $P=800$ W, $v=300$ mm/min, LEIPUL=160 kJ/m.
the pool resulted from the significant Marangoni flow can effectively accelerate the arrangement rate of reinforcing particles, favoring the homogeneous distribution of the reinforcement throughout the inter-dendrite matrix of the finally solidified composites (Fig. 7c and d). Therefore, the distribution state of TiC reinforcement is mainly dependent on the interaction between the reinforcing particles and the dynamic Marangoni flow. On the other hand, a larger volume of melt is obtained within the molten pool as a result of the elevated operative temperature at a higher LEIPUL, thereby improving the rheological properties of the melt in conjunction with the reinforcing particles, due to the elevated wettability of the solid particles by the surrounding melt. Therefore, an increase in LEIPUL favors the significant smoothening and refinement of the initially irregular TiC reinforcing particles (Fig. 7c and d). In general, the columnar dendrites of the matrix are developed by means of the heterogeneous nucleation of nuclei and subsequent dendrite growth during LMD. The internal energy and thermodynamic potentials caused by heat accumulation are responsible for the microstructure development in LMD-processed composites. A high ratio of G/R is regarded as the driving force for the dendrite nucleation and growth, where G is temperature gradient and R is solidification speed [44]. At a limited LEIPUL caused by a lower laser power or a higher scan speed, the operative temperature produced in the molten pool is relatively low, due to the limited laser energy input or the comparatively short laser irradiation time. As a result, a lower G/R ratio is obtained, which further inhibits the sufficient development of columnar dendrites of matrix metal, producing the heterogeneous microstructure of LMD-processed composites (Fig. 6a). While at an excessive LEIPUL of 160 kJ/m, a large amount of heat is accumulated around dendrite tips because of the markedly increased thermalization of laser energy. It accordingly provides the sufficient internal energy and thermodynamic potential for driving the development and coarsening of the columnar dendrites of the matrix (Fig. 6d). As the reasonable LEIPUL of 100 kJ/m is set for LMD, the homogeneous and refined columnar dendrites are well developed (Fig. 6c) and, in this situation, a favorable mode of particle–interface interaction is realized in the molten pool, i.e., the trapping of the reinforcing particles by the growing dendrite [45]. The reinforcing particles having smoothened and refined morphologies are well incorporated in the growing dendrite trunks and side branches, leading to a homogeneous dispersion of the reinforcing particles in the finally solidified LMD-processed parts (Figs. 6c and 7c). For a comparison, as the low LEIPUL of 33 kJ/m is applied, the relatively coarsened, irregular shaped reinforcing particles are pushed ahead by the growing dendritic interface and have a tendency to travel along with the dendrites, resulting in the serious collision of particles and attendant segregation after solidification. Furthermore, the growth direction of the dendrites is significantly disordered or, more seriously, the dendrites cannot develop and grow efficiently, caused by the impediment of the poly-angular and aggregated reinforcing particles (Figs. 6a and 7a).

3.4. Wear performance – COF, wear rate and worn surface morphology

The influence of the applied LEIPUL on wear properties (e.g., COF and wear rate) of LMD-processed TiC/Inconel 625 parts is depicted in Table 4. The characteristic morphologies of the corresponding worn surfaces are shown in Fig. 8. For a comparison, the Inconel 625 alloy without any reinforcing was processed using the same LMD parameters and the COFs measured were typically in the range of 0.50–0.75. The LMD-processed TiC/Inconel 625 composites had an apparently decreased COF (generally less than 0.45) due to the incorporation of the TiC reinforcement in the matrix. Nevertheless, the applied LEIPUL played a key role in affecting the wear performance of LMD-processed TiC/Inconel 625 parts. At a relatively low LEIPUL of 33 kJ/m, the average COF and attendant wear rate of the LMD-processed part were comparatively high, reaching 0.44 and 2.3 × 10⁻⁴ mm³/N m, respectively (Table 4). The worn surface was considerably rough and consisted of parallel plowing grooves with larger-sized particle-structured fragments (Fig. 8a), implying the prevailing of severe abrasion wear mechanism in this instance. The insufficient densification response of the LMD-processed part due to the formation of residual micro-pores (Figs. 4a and 5a) and the presence of the disordered dendritic microstructure (Fig. 6a) with the heterogeneous dispersion of poly-angular TiC reinforcing particles (Fig. 7a) were responsible for the limited wear performance. On increasing the LEIPUL properly to 100 kJ/m, the average COF and wear rate decreased sharply to 0.30 and 1.3 × 10⁻⁴ mm³/N m (Table 4), respectively, demonstrating 33% and 43% decrease compared to the part processed at 33 kJ/m. The worn surface of LMD-processed part was considerably smooth. The continuously adherent and strain-hardened tribolayer, free of any significant fracturing or local plowing, was present in the worn surface (Fig. 8b). In this situation, the formation of the ordered columnar dendrites of matrix metal (Fig. 6c) combined with the homogeneously distributed TiC reinforcing particles (Fig. 7c) contributed to the enhancement of wear performance. At an even high LEIPUL of 160 kJ/m, however, the average COF increased slightly to 0.36, increasing the resultant wear rate to 1.7 × 10⁻⁴ mm³/N m (Table 4). Although the worn surface was still remained dense, the tribolayer on the worn surface was fragmented and the entrapped debris was produced, generating a rough worn surface (Fig. 8c). The decreased wear performance in this instance was mainly ascribed to the formation of significantly coarsened columnar dendrites of the metal matrix at the excessive laser energy input (Figs. 6d and 7d).

3.5. Tensile properties and strengthening mechanisms

The tensile strength and elongation of LMD-processed TiC/Inconel 625 composite parts using the optimal LEIPUL of 72–100 kJ/m were measured. One batch of tensile experiments conducted on each LMD processing condition contained three specimens and the obtained tensile strength and elongation were the calculated average values. Compared to LMD-processed Inconel 625 part without any reinforcing particles (i.e., the tensile strength of 840 MPa, the yield strength of 531 MPa, and the elongation of 16.0%) [46]), the LMD-processed TiC/Inconel 625 composite parts in this study had an apparently improved tensile properties of both tensile strength and elongation, with the elevated tensile strength of 1077.3 MPa, yield strength of 659.3 MPa, and elongation of 20.7%. The typical morphologies of the fracture surface of the corresponding LMD-processed TiC/Inconel 625 composite part is shown in Fig. 9. A low-magnification micrograph, which revealed the general overview of fracture surface, showed a ductile type of fracture of LMD-processed composite part during tensile tests (Fig. 9a). A high-magnification characterization showed that a large amount of deep and uniform ductile dimples were present on the fracture surface, having the considerably refined dimple size of 3 μm in average (Fig. 9b).

<table>
<thead>
<tr>
<th>LEIPUL (kJ/m)</th>
<th>Average COF</th>
<th>Wear rate (mm³/N m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>33</td>
<td>0.44</td>
<td>2.3 × 10⁻⁴</td>
</tr>
<tr>
<td>53</td>
<td>0.41</td>
<td>2.0 × 10⁻⁴</td>
</tr>
<tr>
<td>100</td>
<td>0.30</td>
<td>1.3 × 10⁻⁴</td>
</tr>
<tr>
<td>160</td>
<td>0.36</td>
<td>1.7 × 10⁻⁴</td>
</tr>
</tbody>
</table>
The ductile fracture mode and attendant increase in tensile strength and elongation of the LMD-processed TiC/Inconel 625 composite parts are attributed to the following.

The significant grain refinement effect of the matrix, which is revealed in Fig. 10 from EBSD micrograph: The significant grain refinement effect of LMD-processed composites at a proper LEIPUL, which was previously disclosed by FE-SEM characterization (Figs. 6c and 7c), was further approved by EBSD results (Fig. 10). The quantitative measurement of the average grain sizes was performed within an area of 3 mm$^2$ in LMD-processed structures. For LMD-processed Inconel 625 without reinforcement, 3283 grains were counted in this area, with the average grain size of 34.1 μm. For Inconel 625 based composites with the addition of 5 wt% TiC reinforcement using the same LMD conditions, 5175 grains were counted in this area and the average grain size decreased markedly to 27.2 μm. Furthermore, the crystallographic features of grain development of Ni–Cr $\gamma$ matrix became more regular and uniform in LMD-processed TiC/Inconel 625 composites (Fig. 10). The elevated degree of grain refinement of composites was induced by (i) laser rapid melting/solidification process and (ii) the pinning effect of the homogeneously dispersed ultrafine TiC reinforcing particles upon the crystalline growth of the metal matrix during LMD (Fig. 7c), which further contributed to the improvement in strength and ductility properties of

Fig. 8. FE-SEM images showing typical morphologies of worn surfaces of LMD-processed TiC/Inconel 625 composite parts using various LEIPUL: (a) LEIPUL = 33 kJ/m; (b) LEIPUL = 100 kJ/m; and (c) LEIPUL = 160 kJ/m.

Fig. 9. FE-SEM characterization of the fracture surface of LMD-processed TiC/Inconel 625 composites at LEIPUL of 100 kJ/m: (a) Low-magnification image showing a ductile type of fracture. (b) High-magnification micrograph showing the formation of refined ductile dimples.
LMD-processed TiC/Inconel 625 composite parts. It reveals that the presence of the TiC reinforcing particles influences both crystalline nucleation and crystalline growth of the matrix during solidification. The reinforcing particles in the molten pool tend to induce a dragging/pinning effect on the movement of grain and phase boundaries [47]. Therefore, for the melt in the solidification system, the impetus for grain growth is weakened and, accordingly, the nucleation rate is enhanced, favoring the formation of refined crystalline structure.

The above study shows that the fine-grained microstructure of laser-processed Inconel based parts contributes to the presence of excellent properties. As Ni-based superalloys are typically applied at elevated temperatures, the assumed effects of grain refinement at elevated temperatures are of particular interest. In Wang et al.’s work on LMD processing of Ni-based superalloy Rene’41 [48,49], a significantly fine directionally solidified cellular or cellular-dendrite structure having little features of secondary dendrite arms was obtained. The secondary dendrite arm was greatly suppressed because of the very high temperature gradient and rapid solidification rate. Extremely fine γ’ precipitates (80–110 nm) were present in the matrix. High-temperature tensile tests showed the high-temperature strength and thermo-plasticity of LMD-processed Rene’41 at 800 °C exceeded considerably as relative to the minimum specifications for commercially Rene’41, revealing that the effects of grain refinement of Ni-based superalloys are also effective at elevated working temperatures.

The efficient prohibition of ultrafine reinforcing particles on the mobility of dislocations, which is disclosed in Fig. 11 by TEM characterization: The interior high-resolution microstructure of LMD-processed composites was further studied by the TEM method in Fig. 11, in order to have a better understanding of the effect of ultra-fine reinforcing particles on strengthening effect and tensile properties enhancement of LMD-processed TiC/Inconel 625 composite parts. Typically, the TEM micrograph consisted of the following microstructural features, i.e., the refined and smoothed reinforcing particles, the significantly refined crystals of the matrix with the crystalline size in a nanometer scale, and the dislocations which were generated across the deformed metal matrix and were pinned by the reinforcing particles. The chemical compositions of the reinforcing particle measured by EDX were as follows: C 54.004 at%, Ti 12.165 at%, Cr 1.270 at%, Ni 0.770 at%, Cu 2.670 at%, Nb 23.862 at%, and Mo 5.255 at%. The reinforcing particles, identified as titanium carbide dissolved with Cr, Nb, and Mo metallic elements, had a smooth surface and a considerably refined size of ~ 200 nm, exhibiting a clean and compatible interface between the reinforcing particles and the matrix. TEM micrograph also revealed that the ultra-fine reinforcing particles could act as the pinning points in the crystals that opposed the motion of dislocations, thereby reducing the dislocation mobility due to the incorporation of reinforcing particles. Dislocations were further pinned due to stress field interactions with other dislocations and reinforcing particles, creating physical barriers from the reinforcing phase along grain/phase boundaries. The strengthening effect of LMD-processed TiC/Inconel 625 composite parts was accordingly realized, since the homogeneous incorporation of ultrafine reinforcing particles throughout the matrix was an efficient method to prevent dislocation motion and propagation, or make it energetically unfavorable for the dislocation to move across grain/phase boundaries.

4. Conclusions

(1) The densification behavior of TiC/Inconel 625 composite parts fabricated by LMD was influenced by the applied LEIPUL. Using an insufficient LEIPUL of 33 kJ/m lowered the relative density of LMD-processed composite parts, due to the appearance of residual large-sized pores in inter-layer areas in the finally solidified parts. An increase in LEIPUL from 100 to 160 kJ/m yielded the near fully dense composite parts after LMD.

(2) On increasing the applied LEIPUL above 100 kJ/m, the incorporated TiC reinforcing particles became significantly refined and smoothed through the elevated melting of particle surfaces. An increase in LEIPUL homogenized the dispersion state of ultra-fine TiC reinforcing particles, due to the efficient action of Marangoni convection within the molten pool. The columnar dendrites of Ni–Cr γ matrix underwent a successive change from an insufficiently developed, disordered microstructure to a refined, ordered microstructure with the increase of LEIPUL. However, the columnar dendrites of Ni–Cr γ matrix exhibited an apparent coarsening at an excessive LEIPUL of 160 kJ/m because of the elevated thermalization of the input laser energy.

(3) A proper increase in the applied LEIPUL to 100 kJ/m led to a considerably low average COF of 0.30 and reduced wear rate of 1.3 x 10^-4 mm³/N m for LMD-processed composites, due to the formation of adherent and strain-hardened tribolayer on the worn surface during sliding wear tests. The formation of
the refined column dendrites of Ni–Cr γ matrix combined with the homogeneously distributed ultra-fine reinforcing particles contributed to the enhancement of wear performance. The significant coarsening of columnar dendrites of the matrix at an excessive LEIPUL of 160 kJ/m lowered the wear/tribological property.

(4) The optimally prepared TiC/Inconel 625 composite parts using a LEIPUL of 72–100 kJ/m demonstrated a ductile fracture mode with the sufficiently high tensile strength of 1077.3 MPa, yield strength of 659.3 MPa, and elongation of 20.7%. The superior tensile properties of LMD-processed parts were attributed to the significant grain refinement effect of the matrix during laser process and the efficient prohibition of ultrafine reinforcing particles on the mobility of dislocations.

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