Selective Laser Melting of in-situ TiC/Ti5Si3 composites with novel reinforcement architecture and elevated performance

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A B S T R A C T
A novel Selective Laser Melting (SLM) process was applied to prepare bulk-form TiC/Ti5Si3 in-situ composites starting from Ti/SiC powder system. The influence of the applied laser energy density on densification, microstructure, and mechanical performance of SLM-processed composite parts was studied. It showed that the uniformly dispersed TiC reinforcing phase having a unique network distribution and a submicron-scale dendritic morphology was formed as a laser energy density of 0.4 kJ/m was properly settled. The 96.9% dense SLM-processed TiC/Ti5Si3 composites had a high microhardness of 980.3HV0.2, showing more than a 3-fold increase upon that of the unreinforced Ti part. The dry sliding wear tests revealed that the TiC/Ti5Si3 composites possessed a considerably low friction coefficient of 0.2 and a reduced wear rate of 1.42×10−4 mm3/Nm. The scanning electron microscope (SEM) characterization of the worn surface morphology indicated that the high wear resistance was due to the formation of adherent and strain-hardened tribolayer. The densification rate, microhardness, and wear performance generally decreased at a higher laser energy density of 0.8 kJ/m, due to the formation of thermal cracks and the significant coarsening of TiC dendritic reinforcing phase.

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1. Introduction
Titanium silicide Ti5Si3, as a promising structural intermetallic compound, has recently received considerable research interest, due to a unique combination of low density (4.32 g/cm³), high melting temperature (2130 °C), excellent oxidation and corrosion resistance, and adequate creep resistance at high temperatures [1,2]. However, the limited ductility and low fracture toughness of monolithic Ti5Si3, originating from a low symmetry (D8h) in crystal structure, are regarded as the serious drawback for structural applications [3]. Fortunately, the previous work performed by Wang et al [4], Shon et al [5], and Li et al [6] has proved that the synthesis of Ti5Si3 based composites reinforced with the stiffer ceramic phase is a promising alternative method to improve its fracture toughness and/or other mechanical properties. In particular, the in-situ composites are favorable, since the in-situ presented phases are thermally stable and possess compatible interfaces with the matrix, leading to less degradation in elevated-temperature applications [7]. Furthermore, a reasonable selection of the ceramic reinforcement which is feasible for Ti5Si3 is demanding. TiC is compatible with Ti5Si3 and their thermal expansion coefficients (TiC 7.7×10−7/°C and Ti5Si3 9.7×10−7/°C [4]) match well, ensuring that it is a suitable candidate material to be used as the reinforcing phase for Ti5Si3.

Powder metallurgy (PM) methods such as spark plasma sintering [4], hot pressing [6], and self-propagating high-temperature synthesis (SHS) [5,8] are normally applied to process Ti5Si3 based composites. Nevertheless, the considerably high melting point of this kind of material restricts the practical methods to consolidate it into full-density components. Processing problems such as gas entrapment, aggregation of constitution phases, and interfacial microcracks may be produced in PM-processed composites. Recently, considerable research attempts have focused on the application of high power lasers in processing the composite materials having high melting temperatures. A wide range of laser processing methods in terms of laser cladding [9–15], laser surface alloying [16–18], laser melt injection [19–22], laser sintering [23], and laser-triggered SHS and subsequent laser alloying [24,25] have been reported. These laser-based surface engineering methodologies have demonstrated the feasibility of producing thin Metal Matrix Composites (MMCs) layers on the top of a metal or alloy work-piece for improving the mechanical and tribological properties.

On the other hand, the fabrication of MMCs as bulk-form parts affords many advantages in industrial applications and is, accordingly, considered as another important research direction. Selective Laser Melting (SLM), as a newly developed Rapid Manufacturing (RM) technique, enables the quick production of complex-shaped parts...
with full densification directly from powders [26–28]. SLM builds parts in a layer-by-layer fashion by selectively fusing and consolidation of thin layers of loose powder using a high-energy laser beam, without any post-processing requirements. Recent research efforts have demonstrated that SLM, due to its flexibility in feedstock and shapes, has a promising potential for the net-shape production of high-performance composite parts [29–32]. Nevertheless, SLM is performed based on a complete melting/solidification metallurgical mechanism and, accordingly, involves multiple modes of heat, mass, and momentum transfer induced by a localized laser scanning [33]. For the composites synthesized in an in-situ manner, the unpredictability and/or uncontrollability of the formation of in-situ phases and microstructures in SLM route remains a major challenge. As the microstructural development may directly influence the densification behavior and mechanical properties of in-situ composites, it is highly necessary to be able to understand and control it during SLM.

Laser surface engineering of SiC/Ti and SiC/Ti–6Al–4V systems have been performed by Choi et al. [34] and Hosson et al. [35,36] by means of a partial melting metallurgical mechanism. The interesting microstructural characterizations of the interfacial reaction between the partially melted SiC particles and the Ti melt during laser processing were presented. In this work, SLM of SiC/Ti powder system was performed to produce bulk-form TiC/Ti5Si3 in-situ composites. A fiber laser with a considerably high energy was used to ensure the complete melting of both the Ti constituent and the high melting point SiC component. Therefore, the metallurgical phenomenon and formation mechanism involved in the present completely melted Ti–C–Si ternary liquid system during SLM were significantly different from the existent previous research reports. The evolutions of phases and microstructures of SLM-processed composites under different laser processing conditions were studied and the mechanical properties in terms of densification level, microhardness, and wear resistance were assessed. The crystallization mechanisms of the in-situ microstructures during laser processing were elucidated.

2. Experimental

2.1. Powder materials

The starting powder materials were 99.9% purity, poly-angular Ti powder with an average particle size of 45 μm and 99.0% purity, irregular-shaped SiC powder with a mean particle size of 13 μm. The original Ti and SiC ingredients were mixed according to a weight ratio of 76.2:23.8 (an equivalent molar ration of 8:3). The two ingredients were milled in a Pulverisette 6 planetary high-energy ball mill (Fritsch GmbH, Germany) at a rotation speed of 300 rpm for 15 h, using a ball-to-powder weight ratio of 10:1.

2.2. SLM processing

The SLM system, as schematically depicted in Fig. 1a, consisted mainly of a YLR-200 Ytterbium fiber laser with a power of ~200 W and a spot size of 70 μm (IPG Laser GmbH, Germany), an automatic powder layering apparatus, an inert argon gas protection system, and a computer system for process control. When specimens were to be prepared, a titanium substrate was fixed on the building platform and leveled. The argon gas with an outlet pressure of 30 mbar was fed into the sealed building chamber and the resultant oxygen content decreased <10 ppm. A thin layer of the powder with a thickness of 50 μm was then deposited on the substrate by the layering system. Afterwards, the laser beam scanned the powder bed surface to form a layer-wise profile according to computer-aided design data of the specimens. The laser power was fixed at 80 W, which was an optimized power by the preliminary experiments. Meanwhile, the scan speeds were settled periodically at 0.1, 0.2, 0.3 and 0.4 m/s by SLM control program, in order to change the processing conditions during one batch of experiment. Four different “linear laser energy densities” (η) of 0.2, 0.267, 0.4, and 0.8 kJ/m, which was defined by the ratio of laser power to scan speed, were used to assess the laser energy input to the powder layer being processed. Laser scanning of each layer was based on a sector scanning manner, as shown in Fig. 1b. The interior borders of each sector, 4 mm × 4 mm in size, were parallel to the part edges. A simple linear raster scanning was performed within each sector, with a scan vector length of 4 mm and a hatching spacing of 50 μm. The cubic specimens with dimensions of
8 mm × 8 mm × 8 mm were prepared, showing a good surface finish quality without any dimensional distortion (Fig. 1c).

2.3. Microstructural characterization

Phase identification was performed by a D8 Advance X-ray diffractometer (XRD) (Bruker AXS GmbH, Germany) with Cu Kα radiation at 40 kV and 40 mA, using a continuous scan mode at 4°/min. Samples for metallographic examinations were prepared according to standard procedures and etched with a solution consisting of HF (2 mL), HNO3 (4 mL), and distilled water (94 mL) for 20 s. Microstructures were characterized using an optical microscope (OM) and a LEO 1550 field emission scanning electron microscope (FE-SEM) (Carl Zeiss NTS GmbH, Germany) in a secondary electron mode at 5 kV. Chemical compositions were determined by an EDAX energy dispersive X-ray spectroscopy (EDX) (EDAX Inc., USA), using a super-ultra thin window (SUTW) sapphire detector.

2.4. Mechanical properties testing

The density of SLM-processed specimens was determined based on the Archimedes principle. The Vickers hardness was measured using a MicroMet 5101 microhardness tester (Buehler GmbH, Germany) at a load of 0.2 kg and an indentation time of 20 s. Dry sliding wear tests were conducted in a HT-500 ball-on-disk tribometer (Lanzhou ZhongKe KaiHua Science and Technology Co., Ltd., China) in air at room temperature. Surfaces of specimens were ground and polished prior to wear tests. A bearing steel GCr15 ball with a diameter of 3 mm and a mean hardness of HRC60 was taken as the counterface material and a test load of 3 N was applied. The friction unit was rotated at a speed of 560 rpm for 10 min and the rotation radius was fixed at 2 mm. The friction coefficients of the specimens were recorded during wear tests. The wear volume (V) was determined gravimetrically using:

\[ V = \frac{M_{\text{loss}}}{\rho} \]  

(1)

where \( M_{\text{loss}} \) was the weight loss of the specimens after wear tests. The wear rate (\( \omega \)) was calculated by:

\[ \omega = \frac{V}{(WL)} \]  

(2)

where \( W \) was the contact load and \( L \) was the sliding distance.

3. Results and discussion

3.1. Phase identification

Typical XRD patterns of the starting powder system and SLM-processed specimens are depicted in Fig. 2. The diffraction peaks for the initial Ti and SiC phases of the powder disappeared completely after SLM (Fig. 2a). Instead, the strong diffraction peaks corresponding to Ti5Si3 (JCPDS Card No. 29-1362) and TiC (JCPDS Card No. 05-0693) were observed. The TiC/Ti5Si3 composites were generally identified in SLM-processed specimens using different laser energy densities (Fig. 2b–e). Thus, it was reasonable to conclude that SLM of the Ti/SiC system led to the in-situ formation of TiC/Ti5Si3 composites free of any residual impurity phases.

When laser beam scans over the powder layer, laser energy is directly absorbed by the powder through both bulk coupling and powder coupling mechanisms [37]. Each localized thermal cycle is developed in a considerably short timescale (<4 ms) [33], heating up the powder particles speedily. The applied fiber laser in this study possesses a sufficiently high energy to elevate the operating temperature above the melting point of SiC component (2730 °C), inducing a complete melting/decomposition of the starting Ti and SiC ingredients without any residuals. With the initial Ti/SiC molar ratio of 8:3 settled, the following reaction then proceeds in the present Ti–Si–C ternary molten system:

\[ 8\text{Ti} + 3\text{SiC} = \text{Ti}_{5}\text{Si}_{3} + 3\text{TiC} \]

(3)

According to Ref. [38], the change in the Gibbs free energies (\( \Delta G \)) of the reaction is \(-911.353 \text{ kJ/mol}\). A large negative \( \Delta G \) favors a spontaneous occurrence of the reaction.

3.2. Compositions and microstructures

Fig. 3 illustrates the influence of laser processing parameters on microstructures of SLM specimens. The quantitative elemental determination by EDX indicated that the dendrite-shaped constituents were typically composed of Ti and C elements with the atomic ratio very close to 1:1. The Ti and Si elements were detected within the underlying matrix and the Ti:Si atomic ratio was ~5:3. A combination of XRD and EDX results thus confirmed the in-situ formation of TiC/Ti5Si3 composites after SLM. Nevertheless, the microstructural features of the formed TiC reinforcing phase exhibited distinct variations with the applied SLM conditions. At the relatively high scan speeds (≥0.3 m/s) and attendant low laser energy densities, the TiC phase generally had a considerably slender dendritic structure and dispersed uniformly throughout the Ti5Si3 matrix (Fig. 3a and c). The length of dendritic trunk and primary dendritic arms reached 8.5–10.0 μm and 0.8–0.9 μm, respectively and the primary arm spacing, 0.15 μm, was well within a submicron scale (Fig. 3b and d). As the laser energy density increased by lowering the scan speed to 0.2 m/s, a continuous, homogeneous network of the significantly refined TiC dendrites was formed (Fig. 3e). The average length of dendritic trunk...
and primary dendritic arms decreased below 4.5 μm and 0.4 μm, respectively and the primary arm spacing was further refined to 0.12 μm (Fig. 3f). However, at a low scan speed of 0.1 m/s and resultant high laser energy density of 0.8 kJ/m, although the composite structure was almost uniform, the dendrites showed a large degree of coarsening (Fig. 3g). The mean length of dendritic
trunk and primary dendritic arms showed 3–5-fold increase, reaching 13.8 μm and 2.2 μm, respectively (Fig. 3h).

Previous work on the conventional PM processing of Ti–Si based in-situ composites reveals that the ceramic reinforcement is typically in a particle morphology [4–6], due to a solid-state sintering (SSS) or a partially melted liquid phase sintering (LPS) mechanism used. Different from these existent results, the TiC reinforcing phase in SLM-processed TiC/Ti5Si3 composites is developed into a unique dendritic structure. Normally, SLM is performed based on a complete melting/solidification manner and the TiC phase is formed through a dissolution/precipitation mechanism by means of the heterogeneous nucleation of TiC nuclei and subsequent grain growth. The local temperature gradient and chemical concentration gradient in the molten pool give rise to surface tension gradient and associated Marangoni convection, producing a significant turbulence in the pool [39]. The TiC precipitates, thus, tend to experience a dendrite growth, due to the instability of the solid–liquid interface caused by the perturbation. According to the Gibbs–Thomson temperature equation, the dendrite tip temperature $T_t$ is expressed as [40]:

$$T_t = T_M + mC_l \frac{RT}{\Delta H_f} \frac{V_s}{V_0}$$  \hspace{1cm} (4)

where $T_M$ is the melting point of the pure component, $m$ the liquidus slope, $C_l$ the liquid solute concentration at the solid–liquid interface, $\Delta H_f$ the latent heat of the material, $V_s$ the growth rate of dendrite tip, and $V_0$ is the kinetic constant. $V_s$ is controlled by laser beam scan speed $V_b$ and can be determined by [41]:

$$V_s = V_b \cos \theta$$  \hspace{1cm} (5)

where $\theta$ is the angle between the vectors $V_s$ and $V_b$. Eq. (5) reveals that $V_s$ is proportional to $V_b$, and, thus, using a higher scan speed leads to a faster development rate of dendrites. Therefore, the length of both dendritic trunk and primary dendritic arms obtained at 0.3 and 0.4 m/s (Fig. 3b and d) shows an increase upon that developed at 0.2 m/s (Fig. 3f). On the other hand, since laser-induced cooling rate can reach a high value of $10^6$ K/s [42,43], the TiC dendrites generally have sufficient time for crystal growth, thereby retaining the ultrafine submicron scale (Fig. 3b, d and f). Based on Eq. (4), however, it reveals that at a considerably low $V_b$ of 0.1 m/s, a significant decrease in the operative $V_s$ tends to markedly elevate $T_t$. A large amount of heat is thus accumulated around the growing dendrite tips, which provides sufficient internal energy and thermodynamic potentials for the coarsening of the finally developed TiC dendrites (Fig. 3h).

3.3. Densification behavior

Fig. 4 depicts the influence of laser energy density on densification rate and cross-sectional microstructure of SLM-processed composite parts. On increasing the applied $\eta$ from 0.2 to 0.267 kJ/m, the densification level increased from 91.6% to 95.7%, which was further elevated to a maximum value of 96.9% at a higher $\eta$ of 0.4 kJ/m. However, as a considerably high $\eta$ of 0.8 kJ/m was used, the densification rate decreased sharply to 87.2%. The corresponding cross-sectional microstructures showed that a number of small-sized interlayer pores with an average size of ~20 μm were formed at a relatively low $\eta$ of 0.2 kJ/m. The considerably dense SLM layers without any apparent pores or cracks were produced as the applied $\eta$ increased $\geq 0.267$ kJ/m. A significant decrease in the obtained density at 0.8 kJ/m was due to the formation of interconnected cracks vertical to SLM layers, with the length in the order of millimeter.

The success of SLM densification is determined by the wettability of the primarily precipitated TiC phase by the residual Ti–Si liquid in SLM system. According to Hosson et al.’s results [44], for a reactive wetting system of liquid metals on ceramic phase, the wettability is predominated by the formation of ceramic phase during processing. The equilibrium contact angle $\theta_{eq}$ can be approximately written as:

$$\cos \theta_{eq} = (\gamma_{lv} - \gamma_{ls}) / \gamma_{lv}$$  \hspace{1cm} (6)

where $\gamma_{lv}$, $\gamma_{ls}$, and $\gamma_{lv}$ are the interface tensions of liquid metal-vapor, reacted ceramic-vapor, and liquid metal-reacted ceramic, respectively. $\gamma_{lv}$ may vary during reaction as the reaction product may diffuse into the liquid [44].

For Ti–Si liquid system, $\gamma_{ls}$ is dependent with the operating temperature $T$ [45]:

$$\gamma_{ls} = 1656 - 0.375T$$  \hspace{1cm} (7)

An increase in the applied $\eta$ to 0.4 kJ/m tends to elevate the workable $T$, which in turn lowers $\gamma_{ls}$. Therefore, a lower contact angle $\theta$ (Eq. (6)) and, accordingly, an improved liquid–solid wettability is achieved, favoring a sufficient consolidation of the composite system during SLM (Fig. 4). On the other hand, the shrinkage rate $d(\Delta L/L_0)/dt$ during solidification can be estimated by [46]:

$$d(\Delta L/L_0)/dt = \frac{\Delta P W}{2R \mu t}$$  \hspace{1cm} (8)

where $\Delta P$ is the capillary pressure, $R$ the grain radius, $W$ the liquid thickness, and $\mu$ is the liquid viscosity. Based on Ref. [47], $\mu$ can be further defined by:

$$\mu = \frac{16}{15} \sqrt{m/kT} \gamma_{ls}$$  \hspace{1cm} (9)

where $m$ is the atomic mass and $k$ is the Boltzmann constant. As a considerably high $\eta$ of 0.8 kJ/m is applied for SLM, the resultant higher $T$ and lower $\gamma_{ls}$ both result in a decrease in $\mu$ (Eq. (9)), hence increasing the shrinkage rate during solidification (Eq. (8)). A large amount of thermal stresses tend to generate in the solidified parts [48–50], resulting in the formation of thermal cracks and the decrease in densification (Fig. 4).

3.4. Hardness and wear performance

The average microhardness measured on the cross-sections of SLM-processed TiC/Ti5Si3 composite parts, as shown in Fig. 5, exhibited a similar trend as the relative density with respect to the laser energy density (Fig. 4). A maximum microhardness value of 980.3HV0.2 was obtained at 0.4 kJ/m. In this situation, a small-sized indentation with a clear configuration was observed, indicating a
small degree of deformation during indentation and an elevated hardness performance. For comparison, the pure Ti parts without the addition of SiC were prepared under the same SLM processing conditions and the average microhardness was 281.0HV 0.2. The microhardness of in-situ TiC/Ti5Si3 composites prepared by SLM of SiC/Ti system showed more than a 3-fold increase upon that of the unreinforced Ti part.

The variations of friction coefficients and wear rates of SLM-processed parts are provided in Fig. 6. The TiC/Ti5Si3 composite parts generally had much lower friction coefficients (the average value ~0.5) as relative to the pure Ti part (the average value ~1.3). Also, the local fluctuation of the friction coefficients of the TiC/Ti5Si3 composites was considerably slighter than that of pure Ti part (Fig. 6a). A significantly improved wear performance of the TiC/Ti5Si3 parts was thus proved. On the other hand, the applied $\eta$ played an important role in influencing the wear performance of TiC/Ti5Si3 composites. The mean friction coefficient and wear rate of the TiC/Ti5Si3 part prepared at a high $\eta$ of 0.8 kJ/m increased sharply to 0.45 and $2.65 \times 10^{-4}$ mm$^3$/Nm, respectively (Fig. 6), due to the formation of significantly coarsened dendrites of TiC reinforcing phase (Fig. 3g and h) and the relatively low densification rate caused by thermal cracks (Fig. 4). By comparison, the TiC/Ti5Si3 part prepared using 0.4 kJ/m demonstrated the superior wear performance. A uniform distribution of friction coefficient with a considerably low average value of 0.2 was obtained and the wear rate, $1.42 \times 10^{-4}$ mm$^3$/Nm, was lowest (Fig. 6). This was essentially due to the homogeneous dispersion of ultrafine submicron-scale TiC dendrites (Fig. 3e and f) and the sufficiently high densification level in this instance (Fig. 4).

Characteristic morphologies of the corresponding worn surfaces are revealed in Fig. 7. At a relatively low $\eta$ of 0.2 kJ/m, the worn surface of SLM-processed part primarily showed parallel grooves representing the abrasion wear (Fig. 7a). In this situation, the local severe plowing of the surface during sliding test resulted in a relatively high wear rate (Fig. 6b). As $\eta$ increased to 0.267 kJ/m, a majority of the worn surface was covered with adhesion tribolayer (Fig. 7b). On increasing $\eta$ to 0.4 kJ/m, the worn surface of SLM-processed part became rather smooth. A continuously adherent, strain-hardened tribolayer without any significant fracture was formed on the worn surface (Fig. 7c). Thus, it was reasonable to consider that as the applied laser energy density increased, the mechanism of material removal during sliding changed from the abrasion to the adhesion of tribolayer. Such a transition is expected to stabilize the friction coefficient and reduce the wear rate of SLM-processed parts, as demonstrated in Fig. 6. The worn surface of SLM-processed part at an even higher $\eta$ of 0.8 kJ/m consisted of severely fragmented tribolayer and also entrapped debris (Fig. 7d). The wear rate in this case is nearly 2 times higher than that of the part processed at 0.4 kJ/m (Fig. 6). A close look at the fine-grained (Fig. 3e) and coarse-grained (Fig. 3g) TiC/Ti5Si3 composites at 0.4 and 0.8 kJ/m revealed that the significant grain coarsening of the TiC reinforcing phase at a higher laser energy density might account for the spalling of tribolayer and the resultant increase in wear rate.

4. Conclusions

The present paper reports on a novel Selective Laser Melting (SLM) process for the preparation of bulk-form TiC/Ti5Si3 in-situ composites starting from Ti/SiC powder system. The main conclusions are drawn as follows. These conclusions are applicable and/or transferrable to other laser-based powder processing techniques, e.g. laser cladding or Laser Engineered Net Shaping (LENS).

(1) Laser energy input played a key role in determining the microstructural features of in-situ TiC/Ti5Si3 composites prepared by SLM of Ti/SiC powder system. As a laser energy density of 0.4 kJ/m was properly settled, the uniformly dispersed TiC reinforcing phase having a unique network distribution and a submicron-scale dendritic morphology was formed.
The SLM-processed TiC/Ti₅Si₃ composites with a densification rate of 96.9% had an average microhardness of 980.3HV₀.₂, showing more than a 3-fold increase upon that of the unreinforced Ti part.

A considerably low friction coefficient of 0.2 and a reduced wear rate of 1.42 × 10⁻⁴ mm³/Nm were obtained in sliding test of TiC/Ti₅Si₃ composites, due to the formation of adherent and strain-hardened tribolayer.

The densification, microhardness, and wear resistance decreased at a higher laser energy density of 0.8 kJ/m, due to the formation of thermal cracks and the significant coarsening of TiC dendritic reinforcing phase.

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References


Fig. 7. SEM images showing the morphologies of worn surfaces of SLM-processed parts at different laser energy densities: (a) η = 0.2 kJ/m, showing the occurrence of abrasion and plowing of the surface; (b) η = 0.267 kJ/m, showing the localized formation of the tribolayer; (c) η = 0.4 kJ/m, showing the continuous coating of the tribolayer; (d) η = 0.8 kJ/m, showing the severe fragmentation of the tribolayer and the wear debris.