Microstructural characterization of different metal matrix composite claddings reinforced by TiC through YAG laser cladding

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Abstract
In this work, a YAG laser was used to clad TiC-reinforced metal matrix composite layers on the surface of different types of metals; low carbon steel, high C–Cr bearing tool steel, spheroidal graphite cast iron and commercially pure titanium. The cladding processes were carried out at heat inputs ranging from 175 J mm$^{-1}$ to 700 J mm$^{-1}$ and at a fixed traveling speed of 4 mm s$^{-1}$. The microstructures of the cladding layers were investigated in detail. In all cases, TiC-surface metal matrix composite layers were successfully formed at different laser heat inputs on all the metal surfaces. A few TiC particles seemed as fine dendrites after the laser treatment. The amount of dendritic TiC has a direct relationship with the laser heat input. For low carbon steel, the clad layer showed a martensitic structure, with sound metallurgical bonding to the base metal and without any defects at the highest laser heat input used in this study (700 J mm$^{-1}$). In the case of high C–Cr bearing tool steel, lower laser heat inputs were enough to form a sound clad layer consisting of fine TiC dendrites distributed in a matrix of martensite laths, some retained austenite and acicular carbides. Laser heat input of 175 J mm$^{-1}$ was enough to build a defect-free clad layer on spheroidal graphite cast iron. The matrix comprised of cementite, martensite, and some blocks of retained austenite. Cracking appears at a higher heat input of 500 J mm$^{-1}$ in the spheroidal graphite cast iron. The matrix of the clad layer on pure Ti substrate was $\alpha'$-Ti martensite, which decreased by increasing the laser heat input.

1. Introduction
Carbide particle reinforced metal matrix composite claddings have various tribological uses. This is due to their good resistance to wear and corrosion at low and high surface temperatures (Ravivarman et al. 2018, Qu et al. 2008, Hong et al. 2019). There are several possible metal substrates, including low and medium-carbon steels (Qu et al. 2008), stainless steel (Zhang et al. 2020), tool steel (Jiang and Kovacevic 2007), cast iron (Janicki 2018), titanium (Yamaguchi and Hagino 2017, Zhang et al. 2017), magnesium (Sahoo and Panigrahi 2016) and aluminum (Yang et al. 2019) alloys. The use of metallic substrates renders a good combination of substrate ductility with high wear resistance and hardness of the hard-coating material on the surface. Many ceramics, such as B$_4$C, VC, WC, SiC, Al$_2$O$_3$, and TiC, were employed as reinforcements for improving wear resistance (Huebner et al. 2017, Kong et al. 2019). In recent years, TiC has been extensively used as a coating material because of its outstanding properties, such as high hardness, good thermal stability, good wear resistance, high thermal conductivity and low friction coefficient (Shen et al. 2018, Sahoo and Masanta 2015). Many different surface modification methods are used to build the hard-clad layer on a metallic substrate, including plasma and thermal spraying, traditional arc welding and laser cladding (El-Labban et al. 2016). A high-power laser results in localized melting of the substrate with the addition of reinforcement, mixing the constituents, and solidifying the mixture quickly, forming a significant, thick hard layer. This layer has sound interfacial bonding with the substrate and can effectively protect from severe friction and wear (Jasim 2013, Muvvala et al. 2017, Chen et al. 2019). In the literature, there are multiple reports on the use of lasers for producing TiC reinforced metal matrix
composites over the surface of different metals for wear resistance applications. Most of the work is focused on the soundness of coating and the mechanical as well as tribological properties. Khalili et al. 2016 produced in situ Fe–TiC coatings on the substrate of carbon steel by laser surface treatment, which exhibits a higher hardness. Cai et al. 2016 deposited two types of ceramics (TiC and Al₂O₃) on the Cr-Mo-V steel through laser cladding, which substantially improved hardness and sliding wear resistance, especially when the ceramic reinforcement was less than 15%; above this, cracks were observed in the coated layer. Xu et al. 2018 employed a fiber laser to form an Inconel-base-metal matrix composite reinforced with TiC. The coating exhibited better corrosion resistance in general corrosive environments such as high temperature, moist ambient conditions, contact with brine, and caustic solution. Chen et al. 2019 examined the consequences of the parameters of the laser cladding process on the coating quality of TiC coatings on Ti6Al4V substrates. Yamaguchi and Hagino 2018 developed a special laser-alloying technique to harden the surface of austenitic stainless steel by forming fine TiC powder. The TiC was formed in situ and increased the hardness to almost six times of the substrate. Zhang et al. 2019 increased the lifetime of 40Cr steel by coating its surface by TiC powder using low energy pulsed laser surface processing. The results showed significant improvement in hardness and resistance to wear. The effects of added TiC contents on the mechanical properties of the laser metal deposition on 718 Inconel alloy were evaluated by Kong et al. 2019. The Vickers hardness and corrosion resistance enhanced with the rise in TiC content. The microstructure of metals and alloys after laser-based additive manufacturing is commonly studied, especially bonding between the deposited layers (Khan et al. 2017). However, a few studies have reported on the consequence of the heat generated by laser on the microstructure of the reinforcement and the matrix of the surface composite. Zhang et al. 2019 deposited a TiC reinforced composite layer on 40Cr steel by a pulsed laser. The increase in the addition of TiC to the cladding resulted in severe segregation of TiC in the coating. In another study, laser additive manufacturing technique was used in another study (Li et al. 2019), to fabricate TiC/AlSi10Mg alloys under high-frequency vibration. The formed alloy was sound and had a homogenous structure. Additionally, the size of the cellular grains was reported to decrease. The current study aims to use the fiber YAG laser system with different powers to produce a hard layer of TiC reinforced metal matrix composite on different metallic surfaces. The microstructures of the formed layers, their interface and the heat-affected zone (HAZ) were studied.

### 2. Experimental procedure

In this work, four different base metals were used namely; low carbon steel, high C–Cr bearing tool steel, spheroidal graphite cast iron and commercially pure titanium. The chemical compositions of each substrate are listed in table 1.

| Base metal                        | C  | Si   | Mn | P  | S  | Cr | Ni | Mo | O  | Ti | Fe |
|-----------------------------------|----|------|----|----|----|----|----|----|----|----|----|
| Low carbon steel                  | 0.08 | 0.25 | 0.81 | 0.014 | 0.005 | 0.014 | 0.014 | 0.008 | — | 0.008 | Bal. |
| High carbon-chromium-bearing-tool-steel | 0.957 | 0.23 | 0.334 | 0.02 | 0.005 | 1.5 | 0.06 | 0.03 | — | 0.002 | Bal. |
| Spheroidal graphite cast iron     | 3.82 | 2.7  | 0.237 | 0.02 | 0.014 | 0.03 | 0.03 | — | — | — | — | 0.1 | Bal. |
| Commercially pure titanium        | 0.09 | —    | —   | —   | —   | —   | —   | —   | — | — | — | 0.1 | Bal. | 0.43 |

TiC particles having a size range of 3–10 μm (figure 1(a)), were uniformly distributed on the surface of the substrate to produce a coating with a thickness of 1 mm. The surface cladding was performed by a laser machine with 3 kW YAG fiber, traveling with a fixed speed of 4 mm s⁻¹, and laser defocusing distance +65 mm. The experiments were carried out at different laser powers ranging from 700 W to 2800 W, based on the substrate material. By dividing the laser power (Watt) with the traveling speed (mm s⁻¹), the net energy input was represented in Joule/mm (J mm⁻¹). The net heat input was varied from 175 J mm⁻¹ to 700 J mm⁻¹. Argon shielding was adopted maintaining a fixed flow rate of 15 l min⁻¹ during the laser cladding process. The microstructures of the cladding layers and the substrate’s heat-affected areas were investigated using a Leica optical microscope and Scanning Electron Microscope (SEM). The SEM (Philips XL30 ESEM) was facilitated with Oxford Instruments EDX system analyzer (INCA 250). The microstructure samples were prepared following standard procedures of metallography and soaking the samples in an etchant of 3% Nital (nitric acid and ethyl alcohol) for all samples, except titanium. For commercially pure titanium, the etchant used was Kroll’s reagent. The laser clad zones were also observed by an x-ray diffractometer (XRD) (D8 Discover set at 35 kV, 80 mA, Mo-Kα radiation). The XRD analysis helped to identify the retention of initial phases and/or the formation of new phases during the cladding.
3. Results and discussion

3.1. Micro- and macrostructure analysis

The microstructure of low carbon steel substrate metal, as shown in figure 1(b), consisting of ferrite grains. A minute amount of pearlite was observed alongside the grain boundaries due to the low carbon content of the alloy, which reduced the presence of pearlite. The basic microstructure of the high C–Cr bearing tool steel substrate (figure 1(c)), was composed of shiny granular carbide particles precipitated in the matrix of tempered martensite. It was expected that these carbides might be chromium carbides due to the Cr content. The spheroidal graphite cast iron substrate (figure 1(d)), consisting of a typical spheroidized graphite, which was surrounded by a thin perimeter of a semi-circle of ferrite phase, embedded in the pearlite matrix (see figure 1(e)). The pure Ti base metal microstructure was characterized by coarse grains inside which, parallel striations, ascribed to α–platelets with the same orientation may be observed, as presented in figure 1(f).

The cladding zones fabricated on the low carbon steel by different laser heat inputs are shown in figure 2. At high energy input (700 J mm\(^{-1}\)), the cladding zone appeared as two different zones over the base metal. The top layer consisted of dense, long acicular dendritic TiC inside a fine martensite matrix, as shown in the upper portion of figure 2(a). The dramatic change of TiC particles from spherical shape to the shape of the long acicular dendritic structure indicated that it was melted during the laser processing and then due to solidification dendritic structure was formed ascribed to the rapid cooling after the laser surface treatment (Chehrghani et al 2012). All laser processing techniques have a common feature of high energy density and ceramics absorbs higher laser energy compared to metals. Similar observations were reported by other researchers (Mahamood et al 2013, Monfared et al 2013). The lower part of this cladding zone observed as a small slender band between the TiC-rich area and the base substrate. This area was depleted of the TiC particles, and it appeared fused and re-solidified as a martensite structure. It is obvious to note that there were multiple interface zones. One interface zone is between the TiC-rich zone and the fused zone, while the other interface zone is between the

Figure 1. Substrates microstructure of (a) TiC powder, (b) low carbon steel, (c) high C–Cr bearing tool steel, (d) and (e) spheroidal graphite cast iron, and (f) pure titanium base metals. All are SEM images except (d), which is an optical micrograph.
fused zone and the substrate. Both interfaces were clean from any defects like porosity or cracks and showed good adherence to the substrate. Decreasing the energy input to 500 J mm$^{-1}$, as shown in figure 2(b), the TiC observed as fine particles having short dendrites inside the matrix of martensite. The heat produced at lower energy input was insufficient to melt the entire TiC particles before the laser process. Additionally, the fused zone after the TiC-rich zone became very small. When the heat input was reduced to 375 J mm$^{-1}$, most of the TiC appeared with the original spherical shape. However, a small number of short dendrites are observed (see figure 2(c)). The heat generated by energy input less than 375 J mm$^{-1}$ was not enough to form a sound cladding layer. For example, at 250 J mm$^{-1}$, the TiC particles were clustered as a dense block over the low carbon steel substrate, as presented in figure 2(d).

For the high C–Cr bearing tool steel substrate, the required heat input was lower than that used in the low carbon steel; less than 500 J mm$^{-1}$ was enough to get a sound clad layer, which may be attributed to the relatively lower melting point of the tool steel and its lower thermal conductivity. The SEM micrographs of the clad zone of high C–Cr bearing tool steel, as shown in figure 3, indicate that numerous (dark-colored) fine TiC particles precipitate inside the matrix constituted mainly by laths of martensite and a small amount of retained austenite. The size of the precipitated TiC particles was varied according to the heat input, as clearly shown in figure 3. In the case of heat input of 250 J mm$^{-1}$, the precipitated TiC particles were finer than that of 500 J mm$^{-1}$ (compare figures 3(a) and (c) with figures 3(b) and (d)). Moreover, some of these TiC particles appeared as ultra-fine dendrites (figure 3(e)). In contrast, the relatively slow cooling rate at higher heat input (500 J mm$^{-1}$) provided melted and re-solidified particles more time to become coarser than those formed at lower heat input (250 J mm$^{-1}$). It should be noted that the matrix of the cladding layer in all the investigated heat input conditions largely consists of martensite laths in addition to some acicular carbide. An intricate examination of figure 3 revealed that the lower heat input (250 J mm$^{-1}$) resulted in smaller martensite laths than that formed at higher heat input (500 J mm$^{-1}$). This was probably due to the relatively high cooling rates of lower heat input compared to that of higher heat input. The XRD analysis further confirmed these observations (figure 4). The XRD spectrum of the original base metal showed tempered martensite in addition to the precipitated carbides (Cr$_{23}$C$_6$ and M$_2$C$_3$), as shown in figure 4(a). All the sample cladded by laser heat input of 500 J mm$^{-1}$ (figure 4(b)), the cladded layer comprised of TiC, martensite, retained austenite, and carbides (Cr$_{23}$C$_6$ and M$_2$C$_3$), which conformed to the microstructure analysis. It is obvious to note that despite of the high carbon content of the treated alloy (0.96 wt%) and the faster cooling rate after laser processing, the cladding layer and its interface with the substrate showed excellent bonding with no sign of macro-defects, as observed in figure 5. Figure 6 shows the microstructures at different laser heat inputs in the close vicinity just below the cladding layer (fusion zone without TiC particles and HAZ).

At laser heat input of 250 J mm$^{-1}$, the narrow fusion zone had relatively small laths of martensite microstructure, as shown in figure 6(a). The HAZ of the same condition showed an acicular martensitic
Figure 3. Different magnified SEM images of the laser-treated layer deposited on high C–Cr bearing tool steel substrate by laser heat inputs of (a) and (b) 250 J mm\(^{-1}\) and (c) and (d) 500 J mm\(^{-1}\).

Figure 4. XRD patterns of (a) high C–Cr bearing tool steel substrate and (b) laser-treated layer at a laser heat input of 500 J mm\(^{-1}\).
microstructure and a few acicular carbides, as presented in figure 6(b). At laser heat input of 500 J mm\(^{-1}\), the fusion zone showed large martensite laths inside ferrite grains, clearly visible in figures 6(c), (d). Moreover, the HAZ grain size increased and became larger than that of the base metal, and it showed a ferritic microstructure containing carbide particles. The variation of the microstructure due to the change in the laser heat input was attributed to the relatively low cooling rate of high laser heat input (500 J mm\(^{-1}\)) compared to that of low laser heat input (250 J mm\(^{-1}\)). The low cooling rate lowered the prospect of the formation of a fully martensitic structure. On the other hand, it caused grain coarsening in the fusion zone and HAZ. When the low melting-point spheroidal graphite cast iron was cladded, lower laser heat input was used (from 175 J mm\(^{-1}\) to 500 J mm\(^{-1}\)) to avoid overheating, and the microstructure is shown in figure 7. All the graphite nodules completely disappeared in the cladding layers in all the experimental conditions. This may be due to the high laser heat input density which melted these graphite nodules. During solidification, the fast cooling rate prevented their reformation because they required a very slow cooling rate. The microstructures of the clad zone of the spheroidal graphite cast iron treated by lower laser heat input of 175 J mm\(^{-1}\) are shown at various magnifications in figures 7(a) and (b).
The laser clad zone of dense TiC dendritic morphology distributed homogeneously inside the matrix comprised of fine martensite, cementite, and a few portions of retained austenite. These phases were also confirmed by the XRD patterns (figure 8). The laser cladding process is followed by rapid solidification, resulting in the nucleation of the grains at a temperature even lower than the normal liquidus temperature (Abboud 2012). This phenomenon may have caused the nucleation of more meta-stable phases within a smaller grain boundary. At this rapid cooling rate, carbon might have reacted with the iron to form iron carbide.

Figure 7(c) shows the structure of the cladding zone by increasing the laser heat input to 375 J mm\(^{-1}\), which consisted mainly of iron carbides (Fe\(_3\)C) and ferrite, in addition to some martensite. The relatively higher laser heat input of 375 J mm\(^{-1}\) resulted in lowering the cooling rate during solidification, which enabled localized carbon originating from the dissolved graphite to diffuse into larger areas, reacted with iron and formed more iron carbide. Due to the high carbon content, the martensite phase was always observed. In contrast, the high laser heat density can burn and evaporate some of the cast iron graphite nodules (Xu et al 2018), leaving pores in the substrate side HAZ, as observed in figure 7(c). At laser heat input of 500 J mm\(^{-1}\), the cladding zone consisted of a mix of almost spherical TiC particles with short dendrites, precipitated within the eutectic carbides, inter-lamellar retained austenite, and martensite (figures 7(d) and (e)). This higher laser heat input, 500 J mm\(^{-1}\), affected a wider area in the substrate below the cladding zone, as shown in figure 7(d). A zoomed SEM image in this area, as shown in figure 7(e) revealed its microstructure, dominated by dendrite-like martensite, plate-like eutectic carbides, as well as some ledeburite, which were formed between the inter-dendritic structures. It is exciting to record to note that the microstructure observed here was similar, as observed in white cast iron. Transannular cracks were also observed in this zone (see figure 7(f)). It is most likely that coarse carbides were
formed during solidification resulted in connecting brittle linkage inside the fused vicinity. Additionally, the formation of thermal stresses due to higher laser heat input of 500 J mm\(^{-1}\) may have also contributed to the formation of cracks.

Figure 9 shows micrographs for the laser-treated layer on the pure Ti surface at 425 J mm\(^{-1}\) of laser heat input. Macroscopically, the cladded layer was directly bonded with the base metal and there were macro-defects or cracks, as shown in figure 9(a). In the cladded zone, many long acicular dendrites appeared, as shown in figure 9(b). Zoomed SEM micrographs accompanied by EDX analysis, shown in figure 10 revealed that these dendrites were TiC, which appeared in dendritic morphology in addition to its original spherical shape. As in previous base metals, the presence of TiC dendrites gave evidence that the melted TiC powder particles were re-solidified during the laser processing. The matrix mainly consisted of \(\alpha'\)-Ti martensite structure, as observed in the micrograph shown in figure 10(c). The fast cooling rate immediately when the laser switched-off during the cladding process transforms the \(\beta\)-Ti structure into \(\alpha'\)-Ti martensite. Other investigators also reported such a transformation in different applications (Adib et al 2008, Fernandez-Vicente et al 2012).

By increasing the processing laser heat input to 500 J mm\(^{-1}\), the depth of the melted and cladded zone was increased due to the additional heat input generated from the higher laser processing power. In addition, the length of the TiC dendrites was remarkably decreased, as observed in figures 11(a), (b). Similarly, the clad zone and the HAZ generated at a heat input of 425 J mm\(^{-1}\), was free from any voids or cracks and had a good

![Figure 8. XRD patterns of a laser-treated layer on spheroidal graphite cast iron by processing laser heat input of 175 J mm\(^{-1}\).](image)

![Figure 9. Different magnified optical images of the laser-treated layer deposited on pure titanium substrate by laser heat input of 425 J mm\(^{-1}\) where (a) general view of the different zones and (b) enlarged view of the dendritic structure inside the clad zone.](image)

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Figure 10. Different magnified SEM images and EDX analyses of the laser-treated layer produced on pure titanium substrate by laser heat input of 425 J mm$^{-1}$ where (a) general view of dendritic TiC structure, (b) enlarged view of TiC dendrites and (c) zoomed view of TiC dendrite arms.

Figure 11. Different magnified SEM images of the laser-treated layer deposited on pure titanium substrate by laser heat input of 500 J mm$^{-1}$ where (a) general view of the clad zone structure, (b) enlarged view of TiC dendrites, (c) the boundary between the clad zone and the HAZ and (d) the HAZ α’-Ti martensite structure.
interfacial bonding with the base metal, as observed in figure 11(c). The HAZ of this condition mainly consisted of $\alpha'$-Ti martensite (see figures 11(c), (d)). When the processing laser heat input was further increased to 700 J mm$^{-1}$, the depth of the clad zone was increased. Figure 12 represents the microstructure of the cladded zone processed at 700 J mm$^{-1}$ of heat input. The density of the TiC particles inside the clad zone was relatively low. The heat generated by 700 J mm$^{-1}$ of heat input was sufficient to melt both the base metal and the added TiC powder. This amount of heat increased the dilution process between the TiC layer and the substrate, which allowed the TiC particles to be distributed over a large area. In this condition, most of the TiC particles were observed as short dendrites. The relatively slow cooling rate during solidification restricted the formation of long dendrites.

4. Conclusions

In this work, four different base metals; low carbon steel, high C–Cr bearing tool steel, spheroidal graphite cast iron and commercially pure titanium were cladded by TiC particles using YAG Fiber laser at a laser-power of 3 kW. The heat input was varied ranging from 700 W to 2800 W (175 J mm$^{-1}$ to 700 J mm$^{-1}$), maintaining 4 mm s$^{-1}$ of traveling speed. The TiC powders were uniformly distributed on the substrate’s surface to form thick film with a thickness of 1 mm. The microstructures of the cladded specimens were investigated in detail. The obtained results are summarized as follows:

1. TiC-reinforced surface metal matrix composite layers were successfully formed on all the processed surfaces through different laser heat inputs.

2. Some of the TiC particles appeared as fine dendrites regardless of the processed metal, and its amount increased with the laser heat input.

3. The matrix of the low carbon steel clad layer was mainly martensitic structure, and it showed good interfacial bonding with the substrate.

4. The clad layer formed on the surface of high C–Cr tool steel consisted of fine dendrites of TiC, sporadically presented in the matrix of martensite laths, acicular carbides (Cr$_{23}$C$_6$ and M$_7$C$_3$), and some retained austenite. The size of these TiC dendrites varied according to the laser heat input. The size of the martensite is also affected by the laser heat input. Lower laser heat input resulted in smaller martensite laths than that formed at a higher laser heat input.

5. The lower laser heat input of 175 J mm$^{-1}$ was optimum to build a defect-free composite coating on the surface of the spheroidal graphite cast-iron. Cracking appeared in the fusion zone at 500 J mm$^{-1}$ processing heat input. The cladding layer microstructure comprised of fine TiC acicular dendrites distributed homogeneously in a matrix of cementite, martensite, and some patches of retained austenite. All original graphite nodules were totally disappeared and were unable to form again while re-solidification took place.

6. A surface composite layer of TiC dendrites distributed in a matrix of $\alpha'$-Ti martensite was deposited on the surface of pure Ti.
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