



ELSEVIER

Journal of Materials Processing Technology 185 (2007) 38–45

**Processing  
Technology**[www.elsevier.com/locate/jmatprotec](http://www.elsevier.com/locate/jmatprotec)

# Incorporation of 3 $\mu\text{m}$ $\text{SiC}_p$ into Titanium surfaces using a 2.8 kW laser beam of 186 and 373 $\text{MJ m}^{-2}$ energy densities in a nitrogen environment

S. Mridha<sup>a,\*</sup>, T.N. Baker<sup>b</sup><sup>a</sup> Department of Manufacturing and Materials Engineering, International Islamic University Malaysia, Jalan Gombak, 53100 Kuala Lumpur, Malaysia<sup>b</sup> Department of Mechanical Engineering, University of Strathclyde, Glasgow G1, UK

## Abstract

The formation of composite layers using a 2.8 kW laser beam of 186 and 373  $\text{MJ m}^{-2}$  energy densities, on commercial purity titanium surfaces preplaced with 3  $\mu\text{m}$  size, 1–4 vol.%  $\text{SiC}_p$  powder in a 100% nitrogen environment, produced gold colour tracks. The tracks gave reflective surfaces after glazing at an energy density of 373  $\text{MJ m}^{-2}$  and dull or a mixture of dull and shiny surfaces at 186  $\text{MJ m}^{-2}$  energy density. Surface cracks were visible in tracks containing 1 and 2 vol.%  $\text{SiC}_p$ , but none were observed in the 4 vol.%  $\text{SiC}_p$  tracks glazed at both energy densities. In the track cross sections, vertical cracks were seen in the 373  $\text{MJ m}^{-2}$  tracks but it was absent in 186  $\text{MJ m}^{-2}$  tracks. The  $\text{SiC}_p$  particles completely dissolved in all the tracks processed in this investigation producing a complex and inhomogeneous microstructure of dendrites and needle particles. At the half way of the melt depth from the surface, the dendrites were larger and densely populated, especially after glazing at 373  $\text{MJ m}^{-2}$ . The hardness measurement of the MMC layer recorded a wide range of hardness values which gave loops in the hardness profiles. Hardness values ranging from 700 to 1000 Hv were observed up to a melt depth of 1 mm in many tracks and the maximum surface hardness of 2250 Hv was measured in the track containing 1 vol.%  $\text{SiC}_p$  and glazed at 373  $\text{MJ m}^{-2}$ . The surface hardness developed 5.6–15 times the base hardness (150 Hv) depending on the dendrite population. The 3  $\mu\text{m}$  size  $\text{SiC}_p$  produced MMC layers 1.5–2 times greater than those previously observed with 6  $\mu\text{m}$   $\text{SiC}_p$ . The large surface area for an equivalent volume fraction of the three micron carbide particles is considered to have a high laser coupling action and hence absorbed more heat energy to produce deeper melt depth compared to those produced using the 6  $\mu\text{m}$   $\text{SiC}_p$ .

© 2006 Published by Elsevier B.V.

**Keywords:** Laser processing; Titanium; MMC layer; Dendrite; Hardness; Melt pool

## 1. Introduction

The incorporation of ceramic particles on laser melted titanium alloy surfaces to modify the poor wear and oxidation resistance to superior surface properties has attracted increasing interest in recent years. Ayers and co-workers [1–3] injected 30–50 vol.% of 100  $\mu\text{m}$  size TiC into laser treated titanium alloys and observed a hardness level of about 450 Hv, the hardness being measured between the embedded particles. This was an increase of over 100% of the hardness of the base alloy. Abboud and West [4] injected 150  $\mu\text{m}$   $\text{SiC}_p$  particles into a commercial purity titanium (CPTi) surface and found a partial dissolution of the  $\text{SiC}_p$ , which led to enrichment of the matrix with silicon and carbon during solidification. Dendrites of TiC were

found at the  $\text{SiC}_p$ -matrix interface while the matrix consisted of  $\alpha/\alpha' + \text{Ti}_5\text{Si}_3$  eutectic, and the hardness increased from 210 to about 600 Hv.

Similar work using preplaced 3 and 6  $\mu\text{m}$  size  $\text{SiC}_p$  particles, ranging in nominal volume fraction from 5 to 20%, onto CPTi produced globular, thread-like, columnar and dendritic morphologies with some agglomerated  $\text{SiC}_p$  [5,7]. The phases identified were TiC,  $\text{Ti}_5\text{Si}_3$  and Ti, but very little  $\text{SiC}_p$  was observed in the melt pool [5,6]. The hardness with 6  $\mu\text{m}$   $\text{SiC}_p$  was in the range of 1000–1400 Hv for the 100  $\mu\text{m}$  deep surface layer, which consisted of a mixture of globular and agglomerated  $\text{SiC}_p$  particles, followed by a plateau of hardness between 450 and 550 Hv in the thread-like structures, extending to a depth of about 500  $\mu\text{m}$  [5]. The large increase in hardness is considered to be due in part to the formation of TiC during the glazing process, which was conducted in a protective environment of argon or helium. The 3  $\mu\text{m}$  carbide particle tracks with thread-like structures produced a maximum hardness development of 450 Hv [7].

\* Corresponding author. Tel.: +60 3 61964452; fax: +60 3 61964477.  
E-mail address: [shahjahan@iiu.edu.my](mailto:shahjahan@iiu.edu.my) (S. Mridha).

Table 1  
Crack intensity and surface conditions of the tracks

% SiC <sub>p</sub> actual	Energy density (MJ m <sup>-2</sup> )	Crack intensity, no./mm track length	Vertical crack	Surface smoothness
1	373	0.15	p	S, Sh
1	186	0.40	a	S,O
2	373	0.15	p	S, Sh
2	186	0.30	a	D
4	373	0	p	D, Sh
4	186	0	a	D

p: present, a: absent, S: smooth, Sh: shiny, O: oxidised, D: dull, R: rough, V: very.

Recent work by the authors [8] using preplaced nominal 5, 10, or 20 vol.% 6 μm SiC<sub>p</sub> powder on CPTi surfaces and laser melting in a nitrogen environment, produced metal-matrix-composite (MMC) tracks consisting of dendrites at the top surface followed by thread-like particles. The melt zone was free from any SiC<sub>p</sub> agglomeration/segregation and the thickness was 2–4 times more than those tracks produced in a helium environment under similar processing conditions [5,7]. The surface layer had hardness between 4.5 and 9 times the base metal (150 Hv), and was backed by a deep underlying layer with a plateau of hardness of about 2.8–4 times the base hardness (b.h.). It was postulated [8] that the thermal reactions involved in the formation of nitrides or carbonitrides, which in effect released a large amount of heat, were responsible for producing a deep melt and led to the complete dissolution of the 6 μm size SiC<sub>p</sub> particles, by increasing the temperature of the melt. The formation of dendrites was due partly to the protective nitrogen environment.

The influence of smaller size of 3 μm SiC<sub>p</sub> in producing MMC layers on CPTi surfaces by laser processing in nitrogen environment has presently been investigated in terms of microstructure, hardness and melt depth. The paper describes the development of microstructure and hardness in the composite layer produced in the 100% nitrogen environment in comparison with those reported for 6 μm SiC<sub>p</sub> particle [8].

## 2. Experimental method

Commercial purity titanium (CPTi) sheet of 3 mm thickness was used in this investigation. The SiC<sub>p</sub> particles of 3 μm average size were used to prepare a thin ceramic coating on the metal surfaces. The experiments used nominal 5, 10 and 20 vol.% SiC<sub>p</sub> coatings, estimated on a hemispherical melt pool of 0.44 mm depth established from earlier work [8]. However, after laser processing, the actual melt depth and width were measured to obtain a more realistic percentage of incorporated SiC<sub>p</sub>. This was approximately one-fifth of the nominal value, i.e. the actual SiC<sub>p</sub> contents were approximately 1, 2 and 4 vol.%, respectively. The SiC<sub>p</sub> powder was preplaced on the metal surface using a low temperature burn off varnish (GE7036) as an adhesive. Glazing was carried out at Culham Laboratory, Atomic Energy Authority, Abingdon, using a 5 kW CO<sub>2</sub> laser. The specimens surface melting were carried out under a 2.8 kW stationary laser beam at a 30 mm defocused distance (DFD) with traverse velocities of 5 and 10 mm s<sup>-1</sup> in a 100% nitrogen environment at a gas flow rate of 50 l min<sup>-1</sup>. Calculated energy densities of the applied laser beam with 30 mm DFD were 373 and 186 MJ m<sup>-2</sup> for 5 and 10 mm s<sup>-1</sup> velocities, respectively.

The MMC surface was investigated on each of the tracks of about 20 mm length. The track cross sections were prepared by mounting the samples in bakelite and polishing by standard metallographic techniques and the polished samples were chemically etched in a solution of 10 cm<sup>3</sup> of hydrofluoric acid, 30 cm<sup>3</sup> of nitric acid and 50 cm<sup>3</sup> of water for a period of 5 s. Optical microscope and SEM were used for detailed metallographic studies. Microhardness measurements were carried out using a 100 gf load on polished and lightly

etched track sections starting from the surface and proceeding towards the matrix through the melt zone. The vernier attachment in the microscope of the hardness tester was used for depth measurements on etched cross sections.

## 3. Results

### 3.1. Surface condition

The MMC tracks were examined at a 50× magnification to assess the surface cracking and surface smoothness, and results are presented in Table 1 as crack intensity (no. of surface cracks per mm track length) and surface shininess/roughness. The vertical cracks, which were seen in some tracks passing from surface to matrix through the melt cross section, are also presented in Table 1, as present (p) or absent (a). Results in Table 1 show that tracks glazed with an energy density of 373 MJ m<sup>-2</sup> (5 mm s<sup>-1</sup> velocity and 30 mm DFD) in pure nitrogen environment produced shiny surfaces and those of 186 MJ m<sup>-2</sup> energy density had dull, or a mixture of dull and shiny, surfaces. The shiny surfaces had a gold colour which is indicative of nitride formation. When laser glazed at 373 MJ m<sup>-2</sup> the surface colour became more golden. The variation of colour intensity with laser power density was also found previously with 6 μm SiC<sub>p</sub> tracks produced in a nitrogen environment [8]. The surfaces of all tracks were reasonably smooth.

The tracks containing 1 and 2 vol.% SiC<sub>p</sub> have surface cracks but of low intensity. However, there is no surface crack in the 4 vol.% SiC<sub>p</sub> tracks (Table 1). Vertical cracks are present in all tracks glazed at 373 MJ m<sup>-2</sup> but none in those 186 MJ m<sup>-2</sup> tracks, see Table 1. It is to be noted that the vertical cracks are related to the cracks along the glazing direction and the surface cracks are across the track width (transverse cracks). No surface and vertical cracks were seen previously in tracks laser processed with 6 μm SiC<sub>p</sub> particles [8].

### 3.2. Microstructure

The vertical sections of the tracks gave melt structures free from any SiC<sub>p</sub>. The SiC<sub>p</sub> particles dissolved completely in the melt under the laser processing conditions. The solidified microstructures consisted of dendrites in general and the dendrites were seen densely populated in the high temperature zone of the melt pool. The tracks processed with an energy density of 373 MJ m<sup>-2</sup> produced larger dendrites covering 80% of the melt pool (Fig. 1a) and vertical cracks were seen crossing through this densely populated dendrite zone. The tracks processed with

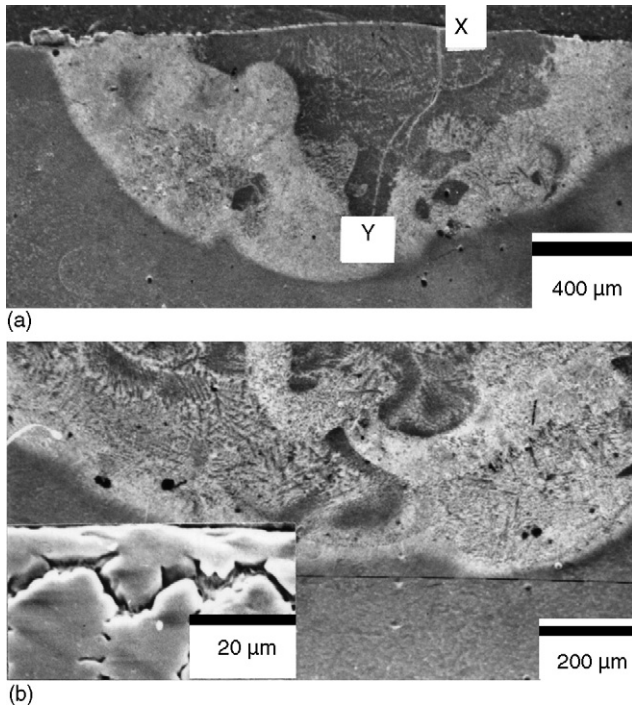


Fig. 1. SEM micrographs showing high density dendrites at high temperature region, (a) a vertical crack XY passing through dense dendritic zone of the  $373 \text{ MJ m}^{-2}$  track and (b) high population of dendrites in the  $186 \text{ MJ m}^{-2}$  track showing no cracks; a thin surface layer (inset): 1 vol.%  $\text{SiC}_p$ , 100%N.

$186 \text{ MJ m}^{-2}$  ( $10 \text{ mm s}^{-1}$  velocity) had less populated dendrites (Fig. 1b) and vertical cracks were not seen in any of these tracks. A very thin layer formed at the top surface of all tracks (inset in Fig. 1b).

The  $186 \text{ MJ m}^{-2}$  tracks produced conventional flow patterns in the melt zone, and large dendrites were found accumulated along the Maragonian flow lines (Fig. 2a). This type of dendrite concentration along the fluid flow lines was also observed previously in the laser nitrided melt pool [9]. However, the other regions away from the conventional flow lines consisted of small dendrites, needles and basket-weave type structures (Fig. 2b). The micrograph in Fig. 3 shows needle and platelet type particles below the dense dendrites, and it may be that the needle particles are platelets.

The  $373 \text{ MJ m}^{-2}$  tracks produced larger dendrites of  $100\text{--}150 \mu\text{m}$  long and  $20 \mu\text{m}$  wide compared to those observed in the melt processed at  $186 \text{ MJ m}^{-2}$ . These larger dendrites covered the entire region and produced an almost continuous solid zone by joining of the dendrites, as shown in Fig. 4. The dendrites at the edge region were quite thin compared to those in the middle zone. The tracks containing 4 vol.%  $\text{SiC}_p$  contained a denser population of larger dendrites than those with 1 and 2 vol.%  $\text{SiC}_p$ . Considering the Gaussian energy distribution of the laser beam reported previously [10], the heat intensity is a maximum at the centre of the beam which created a higher temperature zone in the melt. This might explain why thicker dendrites grew in certain regions of the melt and thinner one at the edge. It is to be noted that the MMC tracks previously produced using  $6 \mu\text{m}$  size  $\text{SiC}_p$  powder had melt structures consisting of dendrites at

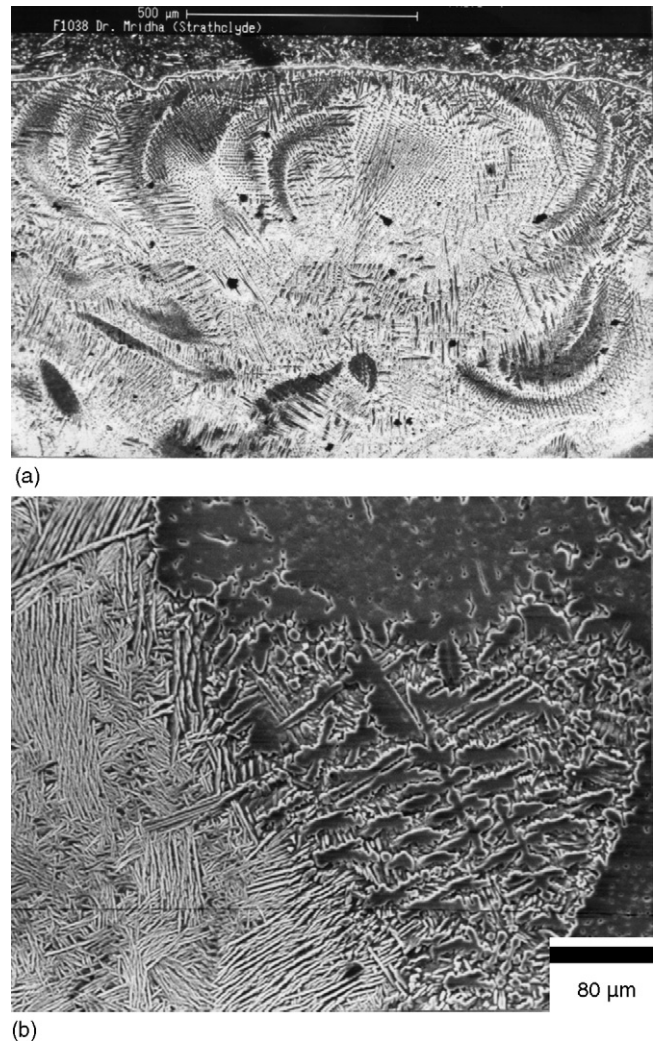


Fig. 2. SEM micrographs showing (a) Maragonian flow lines with high concentration of dendrites: 2 vol.%  $\text{SiC}_p$ ,  $186 \text{ MJ m}^{-2}$  and (b) a mixture of different sized dendrites and basket-wave structures: 1 vol.%  $\text{SiC}_p$ ,  $373 \text{ MJ m}^{-2}$ .

the top surface followed by thread-like particle structures [8]. The thread-like structures were not seen in any of the  $3 \mu\text{m}$   $\text{SiC}_p$  tracks. Recently using a combination of analytical SEM, TEM and X-ray diffractometry the thread-like structure has been identified as a  $\text{Ti}_5\text{Si}_3/\alpha' \text{Ti}$  eutectic, which formed a network enclosing  $\alpha' \text{Ti}$  [11,12].  $\text{Ti}_5\text{Si}_3$  has a hexagonal structure with  $a = 7.444$  and  $c = 5.143 \text{ \AA}$ . Abboud and West [13] investigated the laser alloying of titanium aluminide with large  $\text{SiC}$  ( $150 \mu\text{m}$ ) particles and they found a similar phase, which they identified as  $\text{Ti}_5(\text{SiAl})_3$ . The dendrites have been analysed as  $\text{TiC}$  [11,14,15].

### 3.3. Melt depth

Table 2 shows the MMC layer thicknesses for all the tracks processed in this investigation. The melt depth of the  $373 \text{ MJ m}^{-2}$  tracks ranges from  $1400$  to  $1440 \mu\text{m}$ , which is greater than those produced with  $186 \text{ MJ m}^{-2}$  ( $960\text{--}1100 \mu\text{m}$ ). There is no remarkable variation in melt depth with changing  $\text{SiC}_p$  addition, a factor which was reported to be very significant in tracks produced when helium rather than nitrogen was

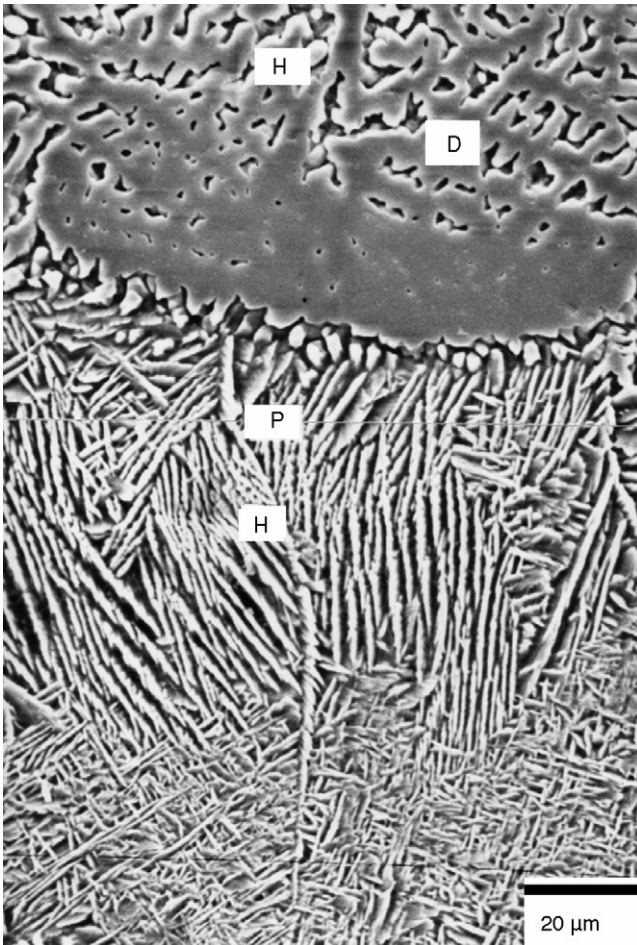


Fig. 3. SEM micrograph showing small needles (10–15 μm) below larger needles (30–35 μm) following a concentrated dendritic structure (D). Platelet particles (P) in the needle structures and the variation of hardness indentations (H) in different microstructures is also visible: 1 vol.% SiC<sub>p</sub>, 186 MJ m<sup>-2</sup>.

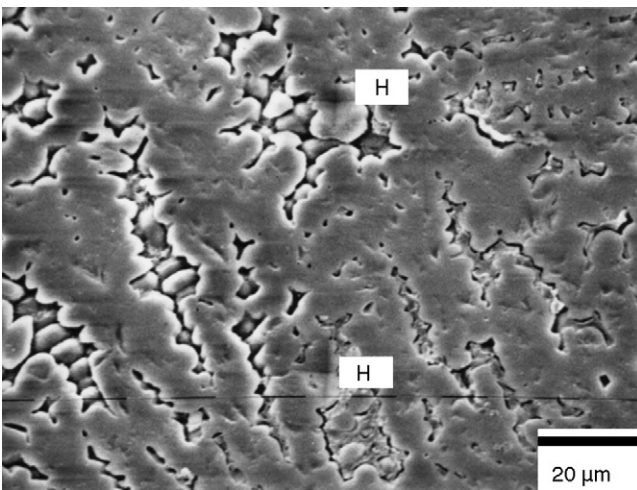


Fig. 4. Micrograph showing large dendrites which joined to form a compact dendritic structures. The dendrite produced small hardness indentations (H): 1 vol.% SiC<sub>p</sub>, 186 MJ m<sup>-2</sup>.

Table 2  
Melt depths of the MMC tracks

Energy density (MJ m <sup>-2</sup> )	Melt depth in micron		
	1 vol.% SiC <sub>p</sub> track	2 vol.% SiC <sub>p</sub> track	4 vol.% SiC <sub>p</sub> track
373	1440	1420	1400
186	960	1060	1100

used [7]. The MMC layer thickness produced under a nitrogen environment is 2–3 times greater than that for the helium environment, reported in literature [7].

### 3.4. Hardness development

The hardness profiles of the MMC tracks are presented in Figs. 5–7. During hardness measurement, the indentations on the melt sections produced erratic values enclosing a range of hardnesses within the melt pool (see Figs. 5–7). These types of erratic hardness values were also reported previously in the nitrated melt zone containing flow loops; the variation of dendrite population due to the Maragonian forces was considered to be responsible for the erratic hardness values [9]. Similar microstructural variations in the present investigation, as observed in the melt structures in Figs. 1–3, are thought to be associated with the formation of these types of hardness loops.

The hardness profiles in Fig. 5 shows that the 1 vol.% SiC<sub>p</sub> track glazed at 373 MJ m<sup>-2</sup> had a maximum surface hardness

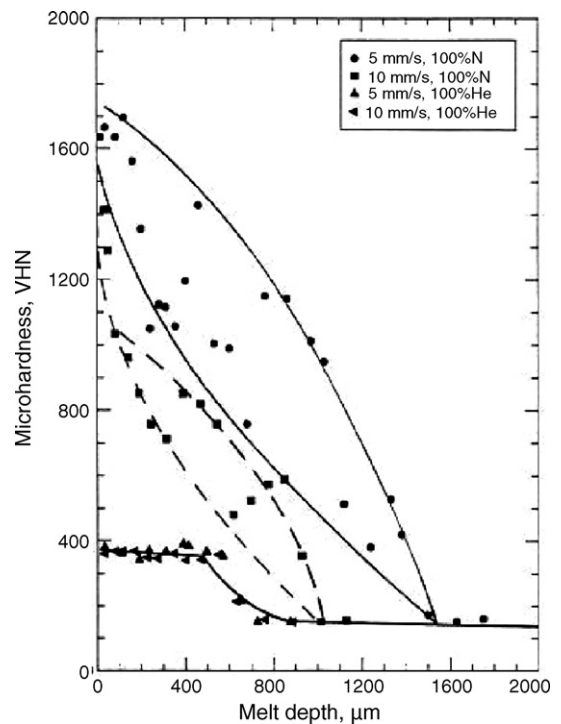


Fig. 5. The hardness profiles showing hardness loops. The hardness decreases with decreasing energy density (10 mm s<sup>-1</sup> velocity), but it is several times higher than that in helium environment also shown in this graph from literature [6]: 1 vol.% SiC<sub>p</sub>.

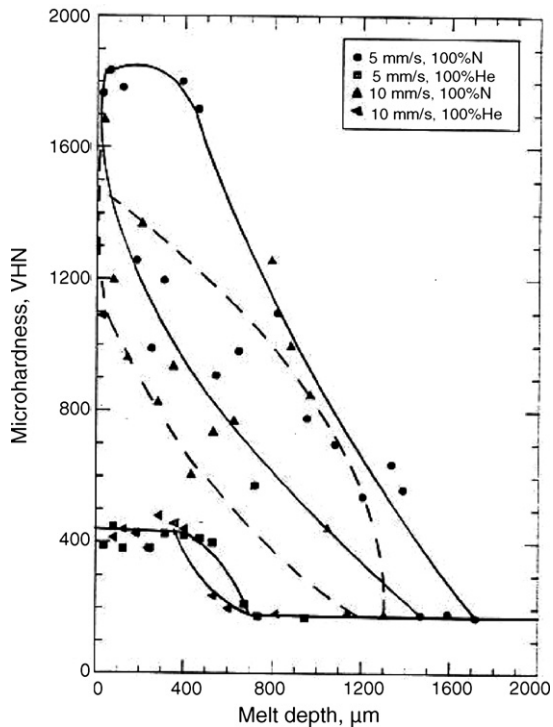


Fig. 6. The hardness profiles of the 2 vol.% SiC<sub>p</sub> tracks give higher hardnesses compared to the 1 vol.% SiC<sub>p</sub> tracks. The hardness under helium environment from previous work [6] is several times lower than this hardness.

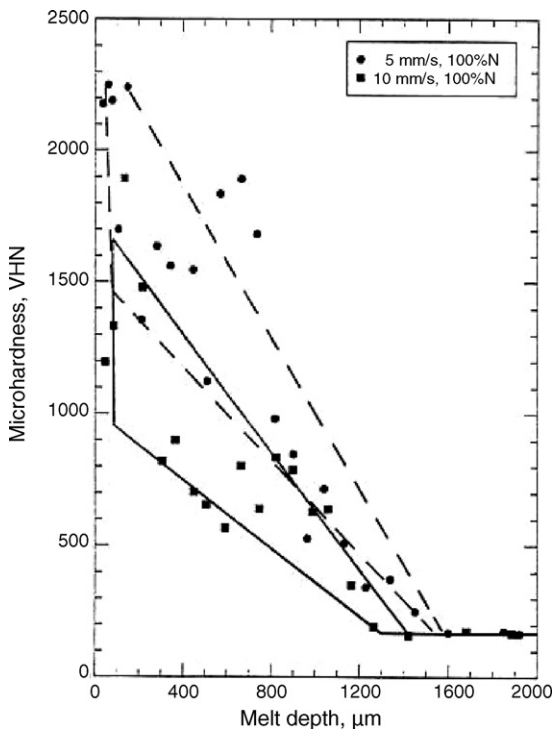


Fig. 7. The hardness profiles of the 4 vol.% SiC<sub>p</sub> tracks give very high hardnesses compared to the 1 and 2 vol.% SiC<sub>p</sub> tracks.

of 1700 Hv, and the melt zone up to 1000  $\mu\text{m}$  deep had hardnesses over 1000 Hv. The 186  $\text{MJ m}^{-2}$  tracks produced a maximum hardness of 1400 Hv and maintained a hardness of over 800 Hv to a melt depth of 500  $\mu\text{m}$ . The surface hardness values are 11.3 and 9.3 times, and at deep melt depths, the hardnesses are 6.6 and 5.3 times, greater than those of the base hardness of CPTi (150 Hv) for 373 and 186  $\text{MJ m}^{-2}$  laser processed tracks, respectively. The reduced hardness values with 186  $\text{MJ m}^{-2}$  tracks are believed to be related to a decreased dendrite population. The dendrites in the 373  $\text{MJ m}^{-2}$  tracks are larger, densely populated and covering over 80% of the melt zone, and they are considered to be responsible for higher hardnesses.

The 2 vol.% SiC<sub>p</sub> track glazed at 373  $\text{MJ m}^{-2}$  had a hardness of 1840 Hv ( $12 \times \text{b.h.}$ ), over 500  $\mu\text{m}$  throughout the melt zone (Fig. 6). A region of hardness over 1000 Hv ( $6.6 \times \text{b.h.}$ ) covering about a 800  $\mu\text{m}$  thick layer of the melt, is also seen clearly in Fig. 6. When glazed with 186  $\text{MJ m}^{-2}$ , the surface hardness of the 2 vol.% SiC<sub>p</sub> track decreased to 1680 Hv ( $11 \times \text{b.h.}$ ), but a layer of over 1000  $\mu\text{m}$  thickness had hardness of 950 Hv ( $6.3 \times \text{b.h.}$ ) (Fig. 6). These values are greater than those obtained from the 1 vol.% SiC<sub>p</sub> track (Fig. 5) which indicates that increasing the SiC<sub>p</sub> addition influenced the hardness developed in the track.

The 4 vol.% SiC<sub>p</sub> tracks show the highest hardness of 2250 Hv ( $15 \times \text{b.h.}$ ) after glazing at 373  $\text{MJ m}^{-2}$  (Fig. 7). A high hardness region about 1800 Hv was found to retain up to a thickness in the melt pool of 700  $\mu\text{m}$ . The 186  $\text{MJ m}^{-2}$  track had a hardness over 1500 Hv ( $10 \times \text{b.h.}$ ) to a melt depth of 300  $\mu\text{m}$ . However, both the tracks show a hardness region of over 800 Hv ( $5.3 \times \text{b.h.}$ ) to a melt depth of 1000  $\mu\text{m}$ . For comparison purpose, the hardness profiles of the 3  $\mu\text{m}$  SiC<sub>p</sub> tracks produced in helium environment and taken from earlier work [7] are incorporated in Figs. 5 and 6. The tracks produced in helium environment show maximum surface hardness ranging from only 380 to 450 Hv under all glazing conditions.

## 4. Discussion

### 4.1. Surface smoothness and cracking

The results in Table 1 show that tracks laser glazed in a pure nitrogen environment had fairly smooth surfaces. The shiny surfaces had a golden colour and the dull surfaces were actually a pale and spotty gold colour. The thin continuous layer at the surface in Fig. 1b produced only after glazing in 100% nitrogen environment and identified as TiN [11], is responsible for producing the gold colour because TiN is gold colour. So the shiny gold surface is indicative of TiN formation. The 4 vol.% SiC<sub>p</sub> tracks processed at a low energy density had a thinner and discontinuous surface layer, which may be the cause of dull surface appearance.

The presence of a few surface cracks in the 1 and 2 vol.% SiC<sub>p</sub> tracks and none in the 4 vol.% SiC<sub>p</sub> tracks (Table 1) may be associated with the formation of a continuous and very hard TiN layer at the top surface only of the 1 and 2 vol.% SiC<sub>p</sub> tracks. The discontinuous TiN layer on the 4 vol.% SiC<sub>p</sub> track surfaces

probably reduced the formation or propagation of any surface cracks.

Vertical cracks, elongated along the glazing direction were seen in the cross sections of the 373 MJ m<sup>-2</sup> tracks are believed to be associated with the melt microstructure. The tracks containing densely populated dendrites of larger sizes and covering the entire melt zone (Fig. 4) formed cracks, as these large dendrites gave relatively high hardness values compared to other regions of lower dendrite concentration. The tracks glazed at 186 MJ m<sup>-2</sup> formed no vertical cracks. The microstructures in Fig. 2 gives thinly populated smaller size dendrites compared to those of 373 MJ m<sup>-2</sup> tracks, Fig. 4. The dendrite population and size are found to be related to laser processing parameters. The population of larger dendrites increased at a higher energy density glazing, especially in the high temperature region of the melt pool. This finding is in agreement with the previous suggestion that a track glazed with a low energy density absorbs less nitrogen, resulting in fewer dendrites [8]. This correspondence is reflected in overall lower hardness values in the 186 MJ m<sup>-2</sup> tracks in Figs. 5–7. The main hardening component in the composite layer is due to dendrites. The microstructures having a higher population of larger dendrites, therefore gave a high hardness.

#### 4.2. Microstructure

The melt structure of the tracks consisted of a mixture of dendrites and needle-like particles (Figs. 2–4). The microstructures in all the melt zones were non-homogeneous; the variation of dendrite concentration from region to region is due to the Maragonian forces developed in the liquid melt. The melt zone of the 4 vol.% SiC<sub>p</sub> track contained more dendrites than in the 1 and 2 vol.% SiC<sub>p</sub> tracks processed at 373 MJ m<sup>-2</sup>. In a helium environment these dendrites were identified as TiC [4,5,14], but in a nitrogen environment, the dendrites are a mixture of carbides and nitrides, as characterized using Selected Area Electron Diffraction and Energy Dispersive Analysis in a transmission electron microscope [11]. The concentrations of carbon/carbide and nitrogen in the liquid melt will play a role in the formation of the dendritic and needle-like structures, and in turn, depend on the dissolution of SiC<sub>p</sub>.

Compared with the microstructure of the surfaces developed with 6 μm SiC<sub>p</sub> particles, discussed in the literature [8], the dendrites of the present tracks produced in the surfaces laser melted using 3 μm SiC<sub>p</sub> particles are larger and densely populated. The growth of these dendrites with 3 μm particles may be related to the high temperature of the melt that resulted from more absorption of laser radiation with larger volume of equivalent amount of smaller SiC<sub>p</sub> particles. More nitrogen will dissolve in the melt at a higher temperature, and the exothermic reaction for nitride formation will further contribute to raise the temperature. The high temperature of the melt containing greater nitrogen concentration will contribute to the growth of the dendrites on solidification. However, the convective flow of the melt is believed to have contributed largely to accumulate dendrites at the high temperature region and flow lines of the melt (Figs. 1b and 2a). This is in conjunction with the observation

of nitride precipitation along the flow lines of the laser nitrided tracks [9].

The lower population of dendrites in the microstructure observed after laser treating using 186 MJ m<sup>-2</sup>, may also be related to the temperature of the melt. At this laser energy density, the melt had a small superheat, and near to the edges of the melt region, the intensity of the laser beam is lower because of a Gaussian distribution of laser heat energy [10]. So at a lower temperature, the solubility of nitrogen will be reduced resulting in smaller and less populated dendrites, and with fewer dendrites at the edges of the melt. This observation suggests that the temperature of the melt controls the dendrite size and population. The ‘basket-weave’ type structure in Figs. 2b and 3 resembles those produced in a nitrided track glazed in a dilute environment [9,16].

#### 4.3. Melt depth

The formation of a deep MMC layer, ~1000 μm thick, using a nitrogen atmosphere (Table 2) without any undissolved SiC<sub>p</sub> particles (Figs. 1–4) is presumed to be related to the reactive nature of nitrogen gas with titanium which has previously been suggested to increase the melt temperature [8]. This increased melt temperature was probably sufficiently high to dissolve SiC<sub>p</sub>. The formation of nitride releases three times more heat than for the corresponding TiC formation by dissociation of SiC<sub>p</sub> [8]. Under a nitrogen environment the melt temperature is therefore expected to increase and produce a deeper melt compared to processing in an inert environment. It is also believed that the larger surface area of the smaller carbide particles absorbed more energy from the laser beam and contributed to greater melt thicknesses in the 3 μm SiC<sub>p</sub> tracks compared to those processed with 6 μm SiC<sub>p</sub> [8].

#### 4.4. Hardness

Laser processing using a 100% nitrogen atmosphere produced MMC layers with high hardnesses ranging from 800 to 2250 Hv (Figs. 5–7). A thread-like microstructure observed in the tracks processed using 6 μm SiC<sub>p</sub> particles in a helium environment reported to produce a hardness of 450 Hv [5]. A maximum hardness of 1400 Hv was found in the dendritic region of these tracks, with a corresponding plateau of hardness about 2.8–4 times the base hardness [5,7]. The high hardness values of the present tracks are related to the dendritic microstructure and the population density of larger dendrites as illustrated in Figs. 1 and 4. Another important factor for the development of high hardness is the composition of the dendrite. The dendrites were identified as a mixture of carbides and nitrides [11]. The variation in hardness in Figs. 5–7 is considered to be related to the size, concentration and composition of dendrites. TiN has a hardness of 2500 Hv. If the nitrogen or nitride content in dendrite increases, the hardness value is likely to increase.

The tracks processed with a high heat input of 373 MJ m<sup>-2</sup> produced an increased population of larger dendrites (100–150 μm), see Figs. 3 and 4 and they contributed more with

higher hardnesses (Figs. 5–7). Nitrogen dissolution in the high energy density melting is expected to increase, and the solidification of the high temperature melt will produce larger dendrites of higher nitrogen concentration. This may be one of the reasons why the 373 MJ m<sup>-2</sup> tracks gave high hardnesses compared to those of 186 MJ m<sup>-2</sup> tracks, the latter produced thinly populated smaller dendrites. Because of high heat generation in the melt using 3 μm ceramic particles and the nitrogen reactive environment, many tracks produced in this investigation produced deep melt depths of 1000 μm of high hardness ranging from 700 to 1000 Hv (Figs. 5–7). The variation of hardness in Figs. 5–7 is associated with dendrite size and population density, and the Maragonian forces (Fig. 2) are considered to be responsible for creating a microstructure with different dendrite agglomerations in different regions.

Previous work using 45–150 μm size SiC<sub>p</sub> particles and a particle injection technique, reported an hardness increase from only 1.1 to 4 times the base hardness by increasing the SiC<sub>p</sub> volume fraction from 25 to 60% [3,4,17]. By comparison, the incorporation of 1–4 vol.% of 3 μm size SiC<sub>p</sub> produced hardnesses up to 15 times the base hardness and had a melt zone thickness of 960–1440 μm. The high hardness was retained to a greater depth in a crack-free microstructure and is likely to improve the load bearing capacity and wear resistance, as reported in the literature [18,19].

The high hardnesses over a deep MMC layer are expected to improve the surface properties, especially the wear resistance, which was reported to increase by a factor of 100 in tracks glazed in a helium environment, using 6 μm SiC<sub>p</sub> particles, which produced a hard layer (1000–1400 Hv) with a thickness of 100 μm [7]. However, the most attractive microstructure is one which contains no vertical, transverse or surface cracks, has a high surface hardness to confer good wear resistance, and a layer from the surface of at least 1 mm, with hardness in excess of 600 Hv. With the present experiments, these conditions were approached by the following conditions: laser glazing in a pure nitrogen environment with 4 vol.% SiC<sub>p</sub> using 373 MJ m<sup>-2</sup>. This summary assumes that the surfaces are not dressed. If machining of the surfaces removed a small layer containing cracks, then several other sets of processing conditions may equally be suitable to produce attractive properties.

## 5. Conclusions

1. The tracks processed in a nitrogen environment at an energy density of 373 MJ m<sup>-2</sup> had smooth and shiny surfaces, while those glazed using an energy density of 186 MJ m<sup>-2</sup> had dull surfaces. The tracks had gold colour surfaces with a varying intensity, depending on the energy density of the laser beam. The SiC<sub>p</sub> particles were completely dissolved in the melt.
2. The solidified MMC layer consisted of dendrites and needle particles. The dendrites had different sizes and population densities, and they were found to agglomerate mostly in the high temperature melt zone. An increased population of larger dendrites was seen covering 80% of the melt depth when laser glazed with 373 MJ m<sup>-2</sup>. A higher melt

temperature, enhanced by the heat of solution due to the reactive nature of nitrogen with titanium is considered to be responsible for producing large and densely populated dendrites.

3. The melt thickness of all tracks containing different volume fractions of 3 μm SiC<sub>p</sub> particles were 1.5–2 times greater than that previously reported for 6 μm tracks produced under identical conditions. The larger surface area of 3 μm SiC<sub>p</sub> particles that absorbed a higher fraction of the laser radiation, and the energy released due to the formation of TiN are believed to have raised melt temperature leading to deeper melt zones.
4. The hardness development in the MMC layer was up to 15 times that of the base hardness. A melt depth over 1 mm in thickness and over 5–7 times the base hardness could be achieved by glazing at 373 MJ m<sup>-2</sup>. The formation of large dendrites of higher population and increased nitrogen content is suggested to have produced high hardness tracks, especially when processed at 373 MJ m<sup>-2</sup>.
5. All tracks containing 1 and 2 vol.% SiC<sub>p</sub> showed surface cracking, but none were observed in the 4 vol.% SiC<sub>p</sub> tracks. Vertical cracks were present only in tracks processed using 373 MJ m<sup>-2</sup>, including the 4 vol.% SiC<sub>p</sub> addition, but not in the tracks processed using 186 MJ m<sup>-2</sup> energy density. The high population of the large dendrites in the melt is believed to have created this crack.
6. The development of crack-free surfaces with hardnesses in excess of 1000 Hv, together with a 1 mm depth of at least 600 Hv, could be achieved by the sets of conditions explored in the present study of laser nitriding preplaced SiC<sub>p</sub> powders on CP titanium.

## References

- [1] J.D. Ayers, R.N. Bolster, *Wear* 93 (1984) 193.
- [2] J.D. Ayers, R.T. Schaefer, W.P. Robey, A laser processing technique for improving the wear resistance of metals, *J. Metals* 33 (1981) 19.
- [3] J.D. Ayers, Particulate composite surfaces by laser processing, in: K. Mukherjee, J. Majumder (Eds.), *Lasers in Metallurgy*, Metal Society of AIME, Warrendale, Pennsylvania, 1981, p. 115.
- [4] J.H. Abboud, D.R.F. West, *J. Met. Sci. Lett.* 10 (1991) 1149.
- [5] S. Mridha, H.S. Ubhi, P. Holdway, T.N. Baker, A.W. Bowen, Metal-ceramic composite layer formation on titanium surfaces through laser treatment, in: F.H. Froes, I. Caplan (Eds.), *Proceedings of the Titanium'92*, TMS, Warrendale, Pennsylvania, The Minerals, Metals and Materials Society, USA, 1993 pp. 2641–2649.
- [6] M.S. Selamat, L.M. Watson, T.N. Baker, XRD and XPS studies on surface MMC layer of SiC reinforced Ti-6Al-4V alloy, *J. Mater. Process. Technol.* 142 (2003) 725–737.
- [7] T.N. Baker, H. Xin, C. Hu, S. Mridha, Design of surface in-situ metal ceramic composite formation through laser treatment, *Mater. Sci. Technol.* 10 (1994) 536–544.
- [8] S. Mridha, T.N. Baker, Metal matrix composite layers formed by laser processing of commercial purity Ti-SiC<sub>p</sub> in nitrogen environment, *Mater. Sci. Technol.* 12 (1996) 595–602.
- [9] S. Mridha, T.N. Baker, Crack-free hard surfaces produced by laser nitriding of commercial purity titanium, *Mater. Sci. Eng.* A188 (1994) 229–239.
- [10] S. Mridha, T.N. Baker, Surface cracking in laser nitrided titanium of commercial purity and possible ways of reducing these defects, *J. Process. Adv. Mater.* 4 (2) (1994) 85–94.

- [11] C. Hu, M.S.B. Salemat, H.S. Ubhi, T.N. Baker, Characterisation of surface MMC layers developed in Ti-6Al-4V alloy using a combination of SiCp and dilute nitrogen, *Mater. Sci. Technol.* 14 (1998) 1045.
- [12] C. Hu, H. Xin, T.N. Baker, Surface MMCs on a Ti-6Al-4V alloy produced by combining laser nitriding and powder alloying, *Proc. ICCM 11* (3) (1997) 80.
- [13] J.H. Abboud, D.R.F. West, Titanium aluminide composites produced by laser melting, *Mater. Sci. Technol.* 10 (1994) 60.
- [14] A.B. Khoosterman, B.J. Kool, J.Th.M. De Hosson, Electron microscopy of reaction layers between SiC and Ti-6Al-4V after laser embedding, *Acta Mater.* 46 (1998) 6205.
- [15] Y.P. Pei, V. Ocelik, J.Th.M. De Hosson, SiCp/Ti6Al4V functionally graded materials produced by laser melt injection, *Acta Mater.* 50 (2002) 2035–2051.
- [16] M.S. Selamat, T.N. Baker, L.M. Watson, Study of the surface formed by the laser processing of Ti-6Al-4V alloy in a dilute nitrogen environment, *J. Mater. Process. Technol.* 113 (2001) 509–515.
- [17] K.P. Cooper, J.D. Ayers, Laser melt-particle injection processing, *J. Surf. Eng.* 1 (1985) 263.
- [18] M.M. Khrushchov, *Conference Proceedings of the Lubrication and Wear*, Inst. of Mech, Engrs., London, 1957, p. 655.
- [19] R.N. Bolster, I.L. Singer, Surface hardness and abrasive wear resistance of ion-implanted steels, *TRANS ASLE* 24 (1981) 526.