Effect of laser scanning speed on surface properties of Ti-Si laser clad intermetallic coatings fabricated on Ti-6Al-4V alloy

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Abstract: A binary Ti-Si (1 : 1) alloy coating was fabricated on Ti-6Al-4V alloy using laser cladding technique where Neodymium yttrium aluminium garnet laser was utilised. Micro-atomised powders of Ti and Si were used for deposition of multi-track clad coatings utilising power of 1.5 kW and a varied speed of 1.2 m/min, 1.6 m/min and 2.0 m/min. Scanning electron microscope coupled with energy dispersive spectrometer was used for microstructural characterisation and elemental analysis while a digital light microscope was used for optical imaging and coating thickness measurements. Phase identification was conducted using X-ray diffraction analyser and transverse microhardness evaluated at 100 gf for 10 s dwell time was achieved with the use of a Vickers hardness machine. The laser fabricated coatings were found to exhibit a decrease in grain size with an increase in the laser scanning speed. The microstructure of the coatings ranged from irregular refined grains, faceted grains and lamellar eutectic network of $Ti_{SS} + Ti_5Si_3$. The hardness of the coatings exhibited Ti, Si, TiS_2 and Ti_5Si_3 phases averaged a 247.4% increase with maximum hardness recorded at 1.2 m/min scanning speed.

Keywords: laser cladding; titanium; Ti_5Si_3 phase; scanning speed; coating thickness; microhardness; microstructure; HAZ; heat affected zone; substrate; grain structure.

Biographical notes: M.F. Phala is an M-Tech Metallurgy candidate from Tshwane University of Technology, South Africa with a standing five years of experience working with materials processing and surface modification technologies. He boasts working together with rated research gurus who amassed plenty of experience in the field of metallurgy and materials.

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1   Introduction

Due to its primarily light-weight, high corrosion resistance and excellent tensile strength, Ti-6Al-4V alloy has been used considerably in aeronautics for jet engine and air frames as well as in power generation industry (Zhu et al., 2004). However, its poor tribological characteristics and low hardness have pinned a limitation on its potency and viability in

most engineering industries (Fellah et al., 2014). The aforementioned shortcomings are linked with the surface properties of Ti-6Al-4V. Therefore, surface modification of materials is considered one of the most efficient methods for improvement of surface properties of Ti-6Al-4V alloy (Cassar et al., 2012; Fu et al., 1998). With pure titanium and titanium alloys, these techniques are commonly used to achieve superior surface properties as well as desired surface finish (Adesina et al., 2016; Kuroda et al., 1998). A host of surface treatment technologies and methods have been tried and tested on titanium alloys to improve its surface properties including corrosion resistance (Hager, Sanders and Sharma, 2008). To improve surface properties of titanium components and substrates, laser cladding is a majorly adopted first grade technique of choice (Weng et al., 2017) because it is a cost-saving and efficient coating technique with extensive applications in many fields (Guan et al., 2013). A unique advantage of laser surface cladding is its ability to be used as component repair technique, especially for complex parts with complex geometries (Jian et al., 2013).

This process utilises laser beam to impinge the surface of a material to modify the surface structure and yield a range of desired metallurgical effects (Gnanamuthu, 1979). Different types of lasers can be used for laser cladding depending on the targeted application. Nd: YAG laser has common use in laser cladding material fabrication owing to its excellent absorptivity and high machine precision (Dinda et al., 2009).

During laser deposition, the control of powder-to-substrate dilution and metallurgical bonding could enhance the strength of bonding between the deposited coating and the substrate (Kwok et al., 2000; Paital et al., 2012). Processing parameters such as laser power, scanning speed, powder feed rate and spot size are critical in laser cladding as varying combinations of these parameters have the potential to give rise to different metallurgical effects on clad characteristics and geometries. Consequently, improper combinations of the said parameters can prove detrimental by yielding residual stresses within the material (Sun and Hao, 2012; Price et al., 2008). Therefore, it is critical to consider optimisation of laser processing parameters, as this is a major aspect requiring strict consideration and proper regulation (Ren et al., 2010).

Ti-Si alloys are considered to possess the potential for high temperature applications due to the existence of Ti_5Si_3 transition metal silicide (Yin et al., 2000). This refractory silicide is a high temperature material with exceptional properties that include low density, oxidation resistance (Sauthoff, 1990), high temperature strength and high melting point (Kishida et al., 2010, p.846; Tang et al., 2008; Mitra, 1998; Alhammad et al., 2008). These properties make Ti_5Si_3 a desirable material in the aerospace industry (Kishida et al., 2010; Tang et al., 2008). This study is aimed at investigating the effect of scanning speed on the hardness property improvement and microstructural development and evolution of laser clad Ti-Si binary system alloy fabricated on Ti-6Al-4V titanium alloy. The study will pave way for identification of optimised process parameters for laser cladding of Ti-Si coatings on Ti-6Al-4V alloy, which is highly essential for high temperature applications.

2 Experimental procedure

Ti-6Al-4V alloy ($72 \times 72 \times 5$ mm) was used a substrate materials for the deposition of the coatings via laser cladding. Titanium and Silicon (99.9%) atomised spherical powders were used as clad materials. Due to the high susceptibility of titanium to oxidation, the

Ti-6Al-4V substrates were initially sand blasted to remove prior surface oxides and further cleaned with acetone before the laser cladding process. Titanium and Silicon powder ratio was kept at 1 : 1 using two powder hoppers. Cladding was carried out using a co-axial 3 kW Rofin Sinar DY044, Continuous Wave Nd: YAG laser mobilised by a KUKA articulated robotic arm. Coatings with average clad height of 600 μm were fabricated with power of 1.5 kW and scanning speed of 1.2, 1.6 and 2.0 m/min. The spot diameter of the laser beam was 3 mm and multi-track single layer coating was formed employing a 0.5 clad overlap ratio. To create a non-oxidising atmosphere, argon gas was used as a precursor gas. Laser cladding process was achieved in open air at room temperature. Table 1 represents a summary of cladding parameters. Design of experiment was used to determine the laser parameters where variation in laser scanning speed was extracted for the ongoing study.

Following successful cladding, the coatings were sectioned into 10×10 mm using a wire cutting machine (Charmilles Robofil 240 SL, Meyrin, Switzerland) and mounted using Aka Resin Phenolic SEM Black Conductive resin. Samples were mounted using AMP 500 Automatic Mounting Press (Layree Technologies Co Ltd). Grinding using Struers TegraForce-5 was done at P320, P600, P800, P1000, P1200 and P4000, while polishing was achieved using a MD-Largo and MD-Chem OP-S polishing cloth. A Diapro diamond suspension was used and polishing was repeated at 2 min cycles until a mirror finish was obtained.

An Olympus BX51M Optical microscope with Olympus essential image evaluation software was used for microstructural evaluation and coating height measurements. The mounted samples were swabbed (etching) with Kroll's reagent. SEM/EDS were conducted on the samples at varied magnifications to characterise the resultant microstructure and identified elemental species. X-ray diffraction (XRD) was conducted for phase evaluation using a PANalytical XRD evaluation equipment. Transverse microhardness on the coatings was done across the three areas on interest namely: coating; heat affected zone (HAZ) and; substrate. Hardness was measured at 100 gf bearing load for 10 s using a Zwick/Roel ZHVμ Indentec hardness machine. Representative indents were 100 μm apart.

Table 1   Laser cladding parameters for deposited coatings

Sample no.	Laser power (kW)	Transverse speed (m/min)	Powder ratio	Spot diameter (mm)	Energy density
1	1.5	1.2	1(Ti)	3	50
2	1.5	1.2	1 (Si)	3	50
3	1.5	1.2	1 : 1 (Ti-Si)	3	50
4	1.5	1.6	1 : 1 (Ti-Si)	3	37.5
5	1.5	2.0	1 : 1 (Ti-Si)	3	33.33

## 3   Results and discussions

3.1   Optical microscopy

Figure 1 shows the optical microstructural images of the substrate and the laser deposited coatings.

It can be observed that, Figure 1(a) of Ti-6Al-4V substrate exhibits a fine bi-modal orientation of titanium, that is, a mixture of α plates (light blue) and columnar β grains (dark blue). A typical goal in laser cladding on Ti-6Al-4V alloy is to enhance the surface and introduces superior properties that eclipse that surface properties of the underlying alloy. This means formation of new and different microstructures on the surface. In laser cladding, these microstructural features of the alloy material, in this case, Ti-Si, is parameter-dependent. Parameters control determines the quality of the clad and the durability of resultant coatings as well as improvement or deterioration of surface properties.

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Figure 1 Optical microscope images of; a) Ti-6Al-4V alloy, b) alloy coating of 50Ti-50Ti 1.5 kW 1.2 m/min, c) alloy coating of 50Ti-50Si 1.5 kW 1.6 m/min, d) alloy coating of 50Ti-50Si 1.5 kW 2.0 m/min (see online version for colours)
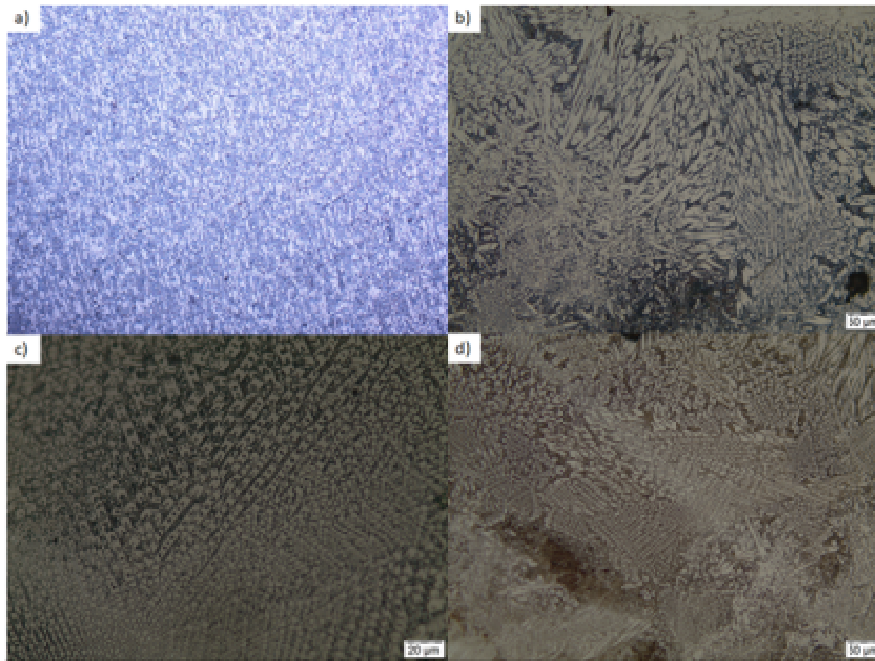
In Figure 1(b) of 50Ti-50Si (1.5 kW and 1.2 m/min), the coatings shows an array of dendritic growth in random direction. The microstructure of the coatings is not well defined but consistent throughout the coating with the presence of eutectics. According to Li et al. (2016) laser scanning speed is considered to possess a considerable amount of influence on the microstructural features.

When the speed was increased to 1.6 m/min, the microstructure was characterised by faceted H-like grains arranged in arrays. It is said that, the microstructural transitions with increasing speed occur due to a decrease in ratio of temperature gradient to the rate of solidification (Huang et al., 2011). The increase in speed also led to a decrease in grain sizes. In addition, at 2.0 m/min, further reduction in grains was evident where colonies of fine grains existed. As seen in comparison of Figure 1(a), (b) and (d), the decrease in grain size of the columnar structures of the coatings was consistent with the increase in scanning speed (Li et al., 2016).

Coating of 50Ti-50Si 1.5kW 1.2 m/min scanning electron microscope imaging at high magnification (Figure 2) revealed presence of large, coarse and irregular elongated grains with a mixture of faceted grains at some regions. These grains were bordered by a field of fine lamellar eutectic phase. The amount of eutectic structures became less visible in the middle of the coating were the coating displayed close-packed large irregular grains as displayed in Figure 3. The large elongated grains structures were identified as Ti_5Si_3 while the lamellar eutectic phase consisted of $Ti_{SS} + Ti_5Si_3$. The eutectic formed a sea-like network of fine lamellar and clubbed grains isolating the larger grains-especially near the bonding line.

Figure 3 displays Close-Packed multi-faceted grains in the middle of the coating of 50Ti-50Si 1.5kW 1.2 m/min

The microstructure displayed combinations of cellular grains, irregular columnar grains oriented at different directions. This observation is evident due to the multi-directional flow of heat during solidification as the upper region of the coating was influenced by convectional heat transfer while the interfacial region was governed by conductive heat transfer.

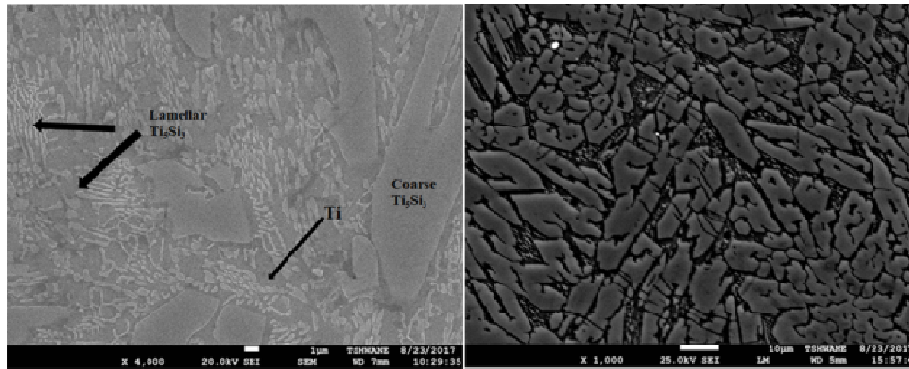**Figure 2**   SEM imaging on alloy coating of 50Ti-50Si 1.5 kW 1.2 m/min

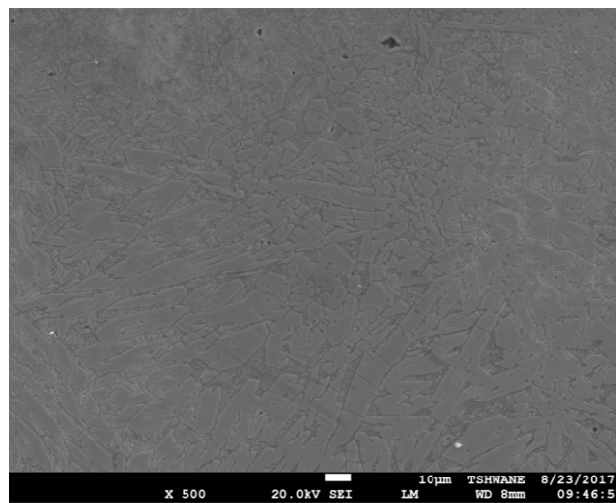**Figure 3**   Close-packed grains in the middle of the coating of 50Ti-50Si 1.5kW 1.2 m/min

Figure 4 shows the EDS micrograph of the interfacial zone between the coating and the substrate. The presence of Si, Al and V indicates an inter-diffusion of elements during cladding.

Table 2 displays the The EDS spectral composition of Spectrum 1 on Alloy Coating of 50Ti-50Si 1.5kW 1.2 m/min.

The presence of V and Al at 2.31 and 3.36 wt. % respectively near the bonding region is consequence of upward diffusion of the elements. The irradiation of the substrate with laser beam resulted in formation of meltpool – a formation of liquid solution of Ti, Al and V. This phenomenon allowed free diffusion of Al and V. However, the diffusion of these elements is limited by the high solidification rate of laser cladding process. It cannot be fairly determined how much of the Ti from Ti-6Al-4V at the scanned region due to the coating being composed partly of titanium. According to Huang (2011), it is this slight diffusion and upward movement of substrate elements that a good metallurgical bond is established.

Figure 4 EDS imaging on alloy coating of 50Ti-50Si 1.5 kW 1.2 m/min (see online version for colours)
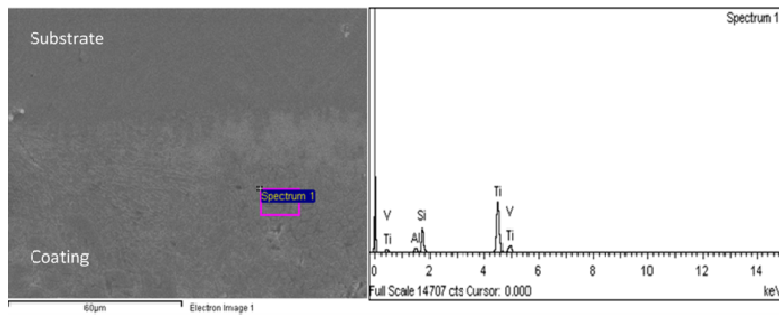

Table 2 The EDS spectral composition of spectrum 1 on alloy coating of 50Ti-50Si 1.5kW 1.2 m/min

| Element	Ti	Si	V	Al
Weight%	76.74	17.6	2.31	3.36
Atomic%	66.80	26.12	1.89	5.19

The EDS spectrum in Figure 4 accounted for high peak of Ti due to Ti_{SS} within which possible phases of Ti-Si binary system are dispersed. The prominent peak of Ti is confirmed by regional compositions revealing 76.74 weight% proving a much more dominant presence of the metal compared to the tetravalent metalloid Si.

3.2 Coating thickness

Figure 5 gives the account of the thickness of coatings deposited at different laser scanning speeds, representing the amount of powder melted within the melt pool. Faster rates result to less powder deposited within the meltpool and this limits the height of the clad layer. At lower scanning speeds, the interaction between the beam and material is prolonged (Li et al., 2014; Izdinska et al., 2010). It is seen that, at the same irradiation

power, lower scanning speeds result in thicker coatings due to the increased amount of powder deposited per minute point across the meltpool.

Figure 5   Laser coating thickness for Ti-Si coatings deposited at 1.5 kW and *x*. Scanning speed (*x* = 1.2, 1.6 and 2.0 m/min)
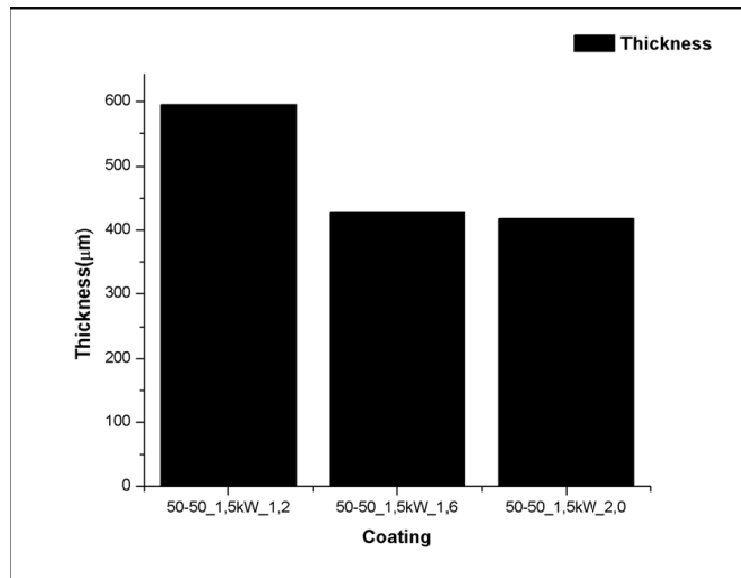
This is consistent with the findings of Riveiro et al. (2014) as well as Sobiyi and Akinlabi (2016) that the scanning speed influences the clad height, which will increase with a decrease in scanning speed and laser power. Thickness decreased from 595.2, 427.5 to 418 μm for speeds of 1.2 m/min, 1.6 m/min and 2.0 m/min respectively. The increase in laser scanning speed was maintained at 33.33% which meant a decrease in irradiation residence. A 33.33% increase in scanning speed from 1.2 m/min to 1.6 m/min resulted in a decrease of coating thickness by 167.7 μm, a 28.18% decrease. A further increase in scanning speed by 33.33% from 1.6 m/min to 2.0 m/min saw a decrease in coating thickness by 9.5 μm; a recorded 2.22% decrease.

3.3   *Coating XRD characterisation*

Figure 6 represents the XRD spectral plots of Ti-Si coatings deposited at varying scanning speeds representing thus, different rates of solidification. This is due to the fact that that increase in scanning speeds favour rapid solidification of laser clad coatings. The plots correspond with the data in microhardness analysis.

It is seen that, at 1.5 kW power and scanning speed of 2.0 m/min, the prominent peak of Ti, which is visible at 1.2 and 1.6 m/min diminishes. This diminishing behaviour is accompanied by development of small peaks of Ti-Si silicide phases. This is revealed by Figure 3(d) of coating optical microscopy to be accompanied by refinement in grains structure. The reduced peak of Ti_{SS} led to an increase in pronounced peaks of Ti_5Si_3 intermetallic. The increased presence of Ti_5Si_3 and $TiSi_2$ intermetallics at 2.0 m/min scanning speed is consistent with the increase in hardness of the coating. These phases

are known for their high hardness, high melting points and strengthening effects (Pei et al., 2002). Therefore, even dispersion of these hard phases is responsible for the improved hardness of the coatings.

It is also worth noting that, the peak intensity of secondary peaks at 1.6 m/min has increased compared to peaks at 1.2 m/min. The prominent Ti peak is identified at $2\theta = 44.4°$ in all coatings with no observed positional change.

The presence of Si metalloid is identified with a most prominent peak observed at $2\theta = 57.2°$ with (011) lattice dimensions. TiS_2 at 1.2 kW is observed at $2\theta = 49.95°$ where it had coordinates of (133). The increase in k and l coordinates was a result of the phase being identified at higher 2θ. As scanning speed was increase, the peaks formed at $2\theta = 45°–49.95°$ disappeared.

Figure 6 XRD curves of Ti-Si alloy deposited via laser cladding process (see online version for colours)
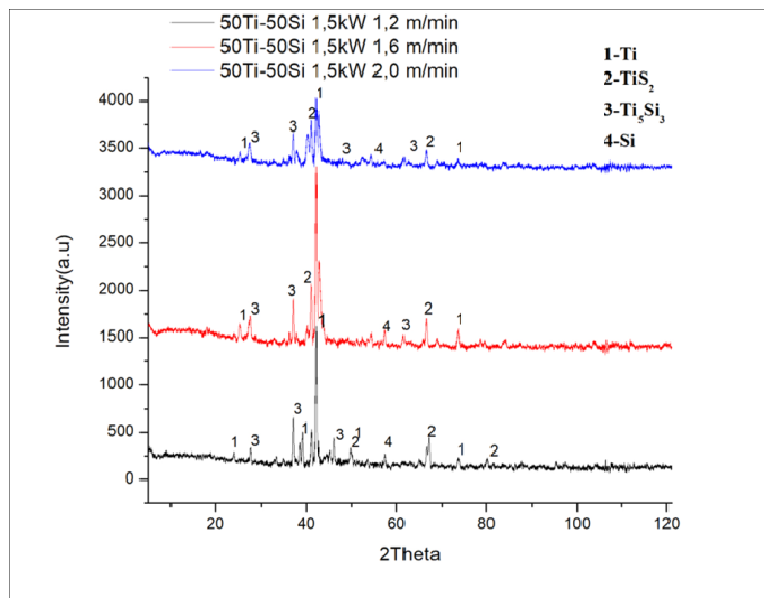

3.4 Microhardness evaluation

Figure 7 represent the microhardness profile of Ti-6Al-4V and Ti, Si and Ti-Si based coatings deposited via laser cladding of Ti-6Al-4V alloy surfaces.

It is observed that the hardness of Si coating is averaged at 784.6 $HV_{0.1}$ while Ti is averaged at 338.2 $HV_{0.1}$ and Ti-Si coatings averaged at 889 $HV_{0.1}$. A combination of the two materials of high and low surface hardness resulted in coating exhibiting 247.4% increase in hardness across the coating compared to the Ti-6Al-4V substrate that averaged 359.4 $HV_{0.1}$. This increase was attributed to formation of hard refractory silicides such as Ti_5Si_3. This necessitates the presence of Ti_{SS} to accommodate for the room temperature brittleness of Ti_5Si_3 (Majumdar et al., 2007). The increase in hardness from 100% Si coating to 50Ti-50Si also is a validating point for the presence of hard phases formed across the coating since the addition of low hardness Ti would logically,

out of metallurgical reasoning, be expected to result in a decrease in the hardness of Si. As such, the increase proves the existence of hard intermetallics dispersed in Ti_{SS}.

It was seen that an increase in laser scanning speed resulted in an increase in coating hardness. This trend was also reported by Mahamood and Akinlabi (2016). However, at speed of 1.2 m/min, the average hardness across the coating was $975.3HV_{0.1}$. This could have been due to regions across the indentation line possessing hard intermetallics resulting in high regional hardness than the rest of the coating. The trend of an increase in hardness with an increase in laser scanning speed was maintained at speed of 1.6 m/min and 2.0 m/min where a 33.33% increase in speed resulted in a further increase of 7.86% in average hardness across the coating. The microstructural refinement with an increase in laser scanning speed as observed in Figure 1 also contributed to the increasing trend in hardness. Furthermore, according to Huang et al. (2011), finer microstructure formation introduces the vantage of more grain boundaries thus increasing the ability to resist lattice distortions due to indentation.

Figure 7   Microhardness profile for Ti-Si coatings deposited at 1.5 kW and varying scanning speeds (see online version for colours)
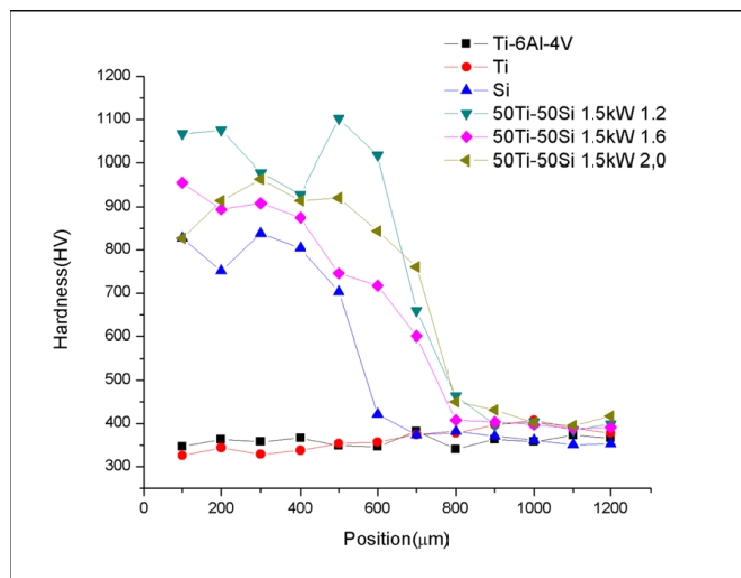
4   Conclusion

Ti-Si binary system laser fabricated coatings were successfully produced on Ti-6Al-4V α+β titanium using Nd: YAG kuka robot-controlled laser system and the microstructure and hardness of the coatings were studied.

Based on the nature of the results and properties exhibited by the coatings, it was concluded that:

- The laser scanning speed is a determining factor of the nature of the microstructure produced by the coatings.

- An increase in laser scanning speed led to microstructural refinement and thus an increase in coating hardness as a result of an observed decrease in grain sizes.

- XRD analysis of the coatings identified presence of Ti_5Si_3 desired phase and $TiSi_2$ phase. The phases were observed for coating deposited at 50Ti-50Si 1.5 kW 1.2 m/min and disappeared when the laser scanning speed was increased.

- An increase in laser scanning speed was also observed to result in a decrease in the prominent peak of Ti citing an increase presence of Ti-Si intermetallics.

- The deposited coating of Ti, Si, and Ti-Si binary system coating exhibited an improvement in microhardness compared to the Ti-6Al-4V alloy. The average increase across the coatings was in the order 338.2, 784.6, 975.3, 813.9 and 877.9 $HV_{0.1}$ for Ti, Si, 50Ti-50Si 1.2 m/min, 50Ti-50Si 1.6 m/min and 50Ti-50Si 2.0 m/min respectively. In average, the Ti-Si coating achieved a rocketing 247.4% increase in coating hardness compared to the substrate material Ti-6Al-4V.

Acknowledgements

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