Evaluation of the microstructure and microhardness of laser-fabricated titanium aluminate coatings

M. Tlotleng[1,2], T. Lengopeng[1] and S. Pityana[1,3]

[1]National Laser Center CSIR
[2]Department of Mechanical Engineering Science, University of Johannesburg
[3]Department of the Chemical and Metallurgical Engineering, Tshwane University of Technology

Titanium aluminide intermetallics are very brittle at room temperature, hence they are challenging to fabricate even by conventional manufacturing techniques such as casting and forging. The production of TiAl from elemental powders using industrial processes is also challenging; in particular regarding the aspects of microstructural tailoring for improved high-temperature performance and oxidation resistance. To circumvent the difficulties, pre-alloyed TiAl powders are used to make industrial components. Electron beam melting was used successfully to produce miniature turbine blades from pre-alloyed TiAl powders. The innovative aspect of this work lies in process development for the fabrication of TiAl materials from elemental powders of titanium and aluminum. The laser metal deposition technique was used to produce TiAl coatings on titanium alloy substrates using a 3 kW laser system. The effects of laser power on the resulting TiAl microstructure were investigated. Scanning electron microscopy (SEM) analysis showed the inclusion of colonies of unmelted TiAl particles and surface pores and cracks. SEM images also revealed that an increase in laser power leads to a microstructural transformation from lamellar to dendritic. The overall hardness of the coating is also a function of laser power. The EDS mapping and hardness measurements confirmed that all the coatings were TiAl phase.

Keywords: hardness, heat inputs, laser power, microstructure, titanium aluminide, X-ray diffraction.

INTRODUCTION

The titanium aluminides are a rather new and attractive class of functionally gradable intermetallic materials that are under development for application as surface coatings and/or as finished engineering structures. These 'smart' materials are lower in strength than nickel superalloys and yet are designed to resist temperatures up to 600–1100°C (Recina *et al*., 2002); hence they are suitable for service in the high-temperature regions of the structures (Kothari *et al*., 2012, 2007; Baudana *et al*., 2016; Balla *et al*., 2016). Titanium aluminides (TiAl) are found primarily in their binary phase form. This phase is made by alloying pure elemental titanium (Ti) and aluminum (Al). However, there has been an interest in the development of ternary phase powders of these aluminides that would serve the same purpose in structural engineering and surface coating. The ternary powders exist merely because of the challenges encountered when trying to convert binary TiAl alloys into tangible structures (Wang and Dahms, 1993). TiAl materials are said to be brittle and lack ductility at room temperature. This is probably due to the mismatch qualities that exist between elemental Ti and Al (Mizuta *et al*., 2008). Ti is regarded as a lightweight, high-strength and high-performing critical metal for various structures, hence it should be suitable for airframes and jet engines (Murr *et al*., 2010).

As opposed to Ti, Al metal has low strength and hence it unlikely to be used in structures that are required to be resistant to deformation and fracture. Typically, Ti alloys are regarded as having high structural efficiency, low density and exceptional corrosion resistive, and excellent materials for applications in operating temperatures up to 600°C (Balla *et al.*, 2010). Al alloys are attractive in applications where specific strength is a major design consideration.

For structural use, any newly developed alloy that meets the requirements of corrosion resistance, ductility and toughness may be selected as long as it adds to the cost benefits in manufacturing. TiAl alloys have unique chemical and mechanical properties, and in general are considered to be superior to the traditional alloys (Romanâov *et al.*, 2006). In the recent times, many TiAl alloys have emerged due to the poor ductility of binary-phase TiAl, and the market that drives innovation. For example, it is believed that TiAl alloys will be used to manufacture aircraft and vehicles with reduced weight (Kim and Hong, 1998), hence reducing emissions while saving costs in fuel consumption. The current drive is directed towards improving ductility at room temperature while promoting self-oxidation at elevated temperatures. The greatest challenge regarding production of these alloys is the identification of an industrial technique that can yield a well-composed, single-phase, homogenously synthesised microstructure that is reproducible (Mizuta *et al.*, 2008). Homogeneity directly affects the strength and performance of the fabricated component. Meanwhile, as well as in structural forms, these high-temperature TiAl alloys are planned for use as thermal barrier coatings (TBCs) in the high-temperature zones of aircraft and automotive engines, and as surface coatings in chemical or nuclear reactors (Sina and Iyengar, 2015).

Like TBCs, TiAl coatings will have to be sufficiently thick, and have high resistance to thermal shock and low thermal conductivity. More importantly, such coatings are required to be well-consolidate with sufficient internal voids; this would lead to the produced coating having the reduced thermal conductivity values that will be well below that of the bulk materials. It has been proposed that a successful TBC coating must be smooth, well-bonded and should never spontaneously degrade. Spallation generally occurs with surface coatings that serve under high loads and in environments where the temperature changes rapidly. Once this occurs the material degrades, quickly causing fouling and introducing problems like wear, erosion and corrosion to the parent material. It is therefore necessary to thoroughly evaluate potential coating materials through detailed analysis of their chemical and metallurgical compatibility, mechanical properties, component compatibility and process selection. The focus in the past decade has been on the characteristics of the coatings, compatibility and resistance to oxidation and corrosion. Process selection is one aspect that is still outstanding and remains to be fully investigated (National Research Council, 1996).

Industrial processes that have been used to manufacture TiAl alloys have been presented (Balla *et al.*, 2016). It appears TiAl alloys manufactured by ingot processing yield a non-homogeneous microstructure, which means that a secondary heat treatment process step is required to achieve a desirable microstructure. This two-step approach could have led to the birth of the hot isostatic pressing (HIP) process, which is currently used under the powder metallurgy and compaction processing approach for the manufacturing of large sheets of TiAl alloy on the industrial scale (Mills *et al.*, 1989). Cormier *et al.*, (2007) studied freeform fabrication of TiAl from pre-alloyed and blended powders using electron beam melting. Similar research on full characterisation of the composition, crystal structure and hardness of TiAl alloy coupons was presented by Baudana *et al*. (2016). Liu and Dupont (2003) proposed the *in-situ* alloying of nickel and aluminum using laser net shaping to produce NiAl alloys. Methods of additive manufacturing for the synthesis of TiAl coatings on titanium-based structures are under development. These processes still have to overcome the challenge of multi-step processing if a desirable homogenous, well-bonded coating is to be produced. Moreover, some processes require advanced equipment to become cost-ineffective, in which case they become unattractive to industrialists and investors (Gussone *et al.*, 2015; Qu and Wang, 2007; Qu *et al.*, 2010).

While surface coating engineers are still looking to understand and develop a single-step process by which TiAl coatings would be produced, they have not disregarded the fundamentals of a sound and attractive coating. A single-step process that incorporates synthesis of TiAl alloy coatings that adhere well to the substrate, of good thickness and which do not need post-heat processing in order to correct the microstructure, is still needed. Such a process would necessarily have to be capable of being scaled up to industrial scale. Laser cladding is a metal deposition technique that is renowned for surface treatment and coating. The laser beam is able to generate the melt pool, which contains convectional forces that mix the powder plume into a homogenous coating. This process allows the coating developers to control the layer thickness of the produced coatings, owing to the ease with which lasers can be directed and controlled (Tlotleng *et al*., 2014). It is well known that lasers are easy to control and are coherent, making it possible to achieve good quality coatings even on small surfaces where necessary. Most importantly, laser processes are sustained with high process temperatures which are necessary to heat treat and remelt the coating layer at the same time. Due to these advantages, this study was undertaken to evaluate the *in-situ* synthesis of TiAl alloys produced by melting Ti and Al into an alloy using a high-power fibre-optical laser. We evaluated the microstructures using scanning electron microscopy equipped with energy dispersive X-ray analysis (SEM-EDX) and hardness measurements both at the interface and across the coating.

EXPERIMENTAL

Materials

Commercially available pure titanium and aluminium powders supplied by TLS, Technik GmbH & Co. Spezialpulver KG were used. Both powders had a particle size distribution of 45–90 µm and were used as received. Ti-6Al-4V base plates of 70 × 70 × 5 mm^3 were used as substrates. Before deposition the base plates were sandblasted and cleaned with acetone. The sandblasting promotes adhesion of the coating to the substrates during processing.

Methods

A 3 kW IPG continuous fibre laser system was used. This laser is continuously cooled by a model 2KP0063102294953 chiller manufactured and supplied by Riedel Precision, Germany. A 1.5 bar GTV powder feeder system, that uses two hoppers, was used to contain and control the powders during processing. Argon supplied from a bulk compressed gas tank, with high-volume control regulators, was used as both the powder carrier and shielding gas. A copper coaxial three-way nozzle was used to inject the powder plume into the melt pool that was already created on the substrate. Figure 1 shows the equipment and the process set-up.

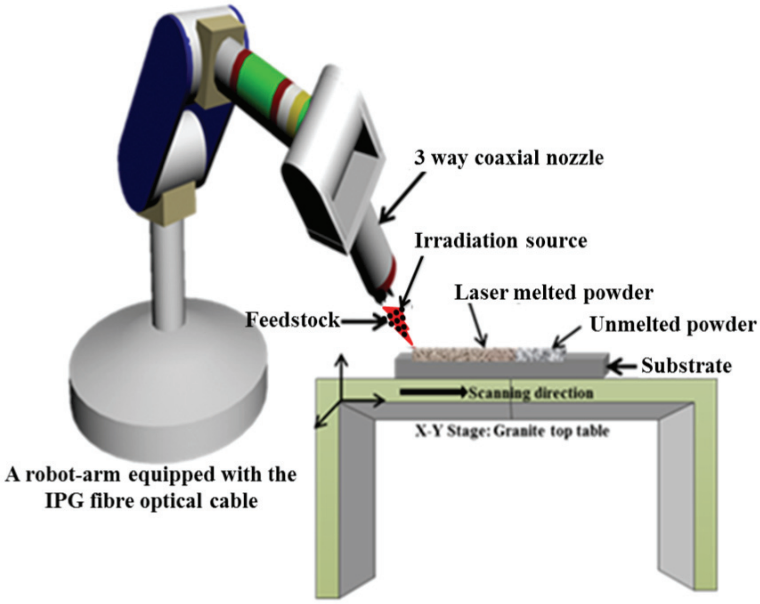

Figure 1. Laser metal deposition set-up.

A Kuka robot arm was used to control both the coaxial nozzle and the laser beam spot. During processing, both powders and the beam were focused to a distance of 12 mm. The laser spot size, carrier gas and the shielding gas were kept constant at 4 mm, 2 l/min and 10 l/min, respectively. The beam interacted with the substrate and the powder plume at an angle of 13°. The Kuka robot arm allowed for the laser beam and powders to be scanned at a constant speed of 2.5 m/min. The set speeds during deposition for the powder feeders were 1.5 r/min for Ti and 1.5 r/min for Al, resulting in a 50/50 TiAl coating. A concentric laser beam-powder spot deposition approach was used. Laser powers of 1.0, 1.3, 1.5 and 2.0 kW were used. The process parameters are summarized in Table I.

Table I. Laser process parameters.

Parameter	Setting
Laser spot	4 mm
Laser power (kW)	1.0, 1.5 and 2.0 kW
Laser scanning speed	2.5 m/min
Carrier gas flow rate	2 l/min
Shielding gas flow rate	10 l/min
Standoff distance	12 mm
Laser-powder spot inclined plane	13°

Sample Preparation

The LMD TiAl coatings were brushed with a wire brush post-processing. The coatings were sectioned, ground and polished to a 0.04 µm (OP-S suspension) surface finish using a Struers TegraForce-5 auto/manual polisher. After polishing, the selected coatings were etched with Keller's reagent for 2–3 minutes and the microstructural features analysed using an Olympus optical microscope equipped with Analysis® software.

Characterisation

The microstructural and elemental analyses of the prepared coatings were carried out using scanning electron microscopy (SEM) (Joel JSM-6010PLUS/LA) equipped with energy dispersive X-ray spectroscopy (EDS). The SEM-EDS system used the Intouch Scope software for analysis, and was equipped with a video camera that allowed for the sample stage height to be visualised and controlled. The phase compositions of the coatings were determined by X-ray diffraction (Panalytical XPert Pro PW 3040/60) with a Cu Kα monochromator radiation source. The phases were identified using material PDF files.

Microhardness Tests

The mechanical microhardness values of the coatings were measured using a Matsuzawa Seiko Vickers microhardness tester model MHT-1. An indenting load of 300 g and dwell time of 10 seconds were used for each hardness indentation action. The hardness measurements were taken across and along the length of the coatings at 100 µm spacing. The HV values across were taken at the interface so as to calculate bonding, while the values measured along the coating were used to indicate the coating hardness. The average of three micro-tracks was taken and reported as the average hardness of the coatings.

RESULTS

Microstructural and Composition Evaluation

The TiAl coatings produced show the effects of laser power on the resulting microstructure. Figure 2 shows the stereomicroscope images of the prepared coatings.

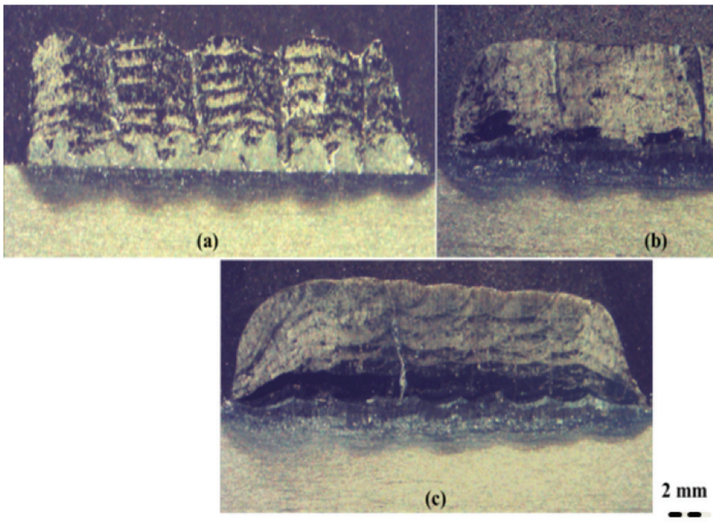

Figure 2. Stereomicroscope images of the as-produced TiAl coatings: (a) 1.0, (b) 1.5 and (c) 2.0 kW.

Figure 2 illustrates the effects of laser power on the resulting microstructure of the TiAl coatings. It appears that the melting, smoothness, cracking, heat affected zone (HAZ) and adhesion became more pronounced with increasing laser power. The SEM images of these coatings are illustrated in Figure 3. These images, taken using the secondary electron image mode, clearly defined the observed unmelted particles of Ti/Al (Wang and Dahms, 1993). These particles disappeared with increasing laser power, which is indicative that melting is achieved by increasing the power density. This melting is corroborated by the observed HAZ (Figure 2), which increased with increasing laser power.

The high-resolution SEM images of the produced coatings are shown in Figure 4.

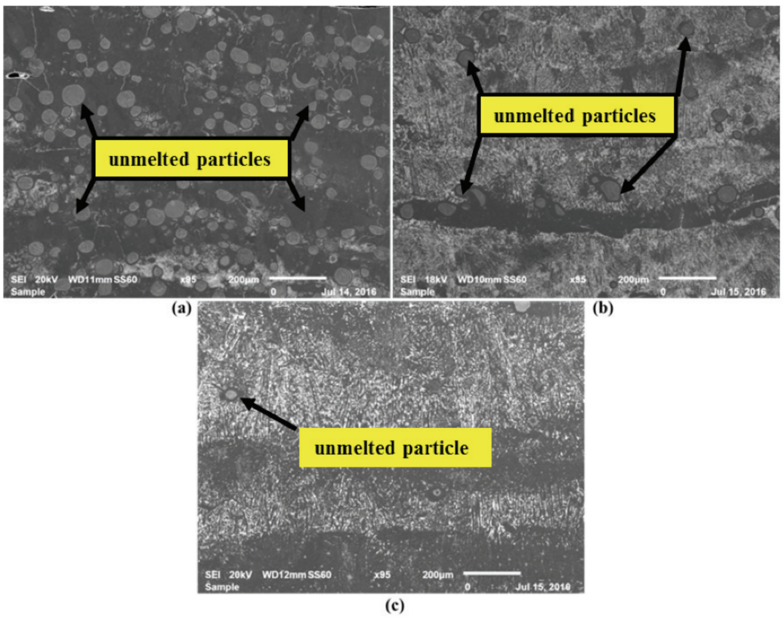

Figure 3. SEM images of the TiAl: (a) 1.0, (b) 1.5 and (c) 2.0 kW.

Figure 4a indicates that the unmelted particle is of titanium origin, given the needles that formed in the core centre. The resulting microstructure of the coating could not be fully resolved, but it was possible to conclude that the white regions are aluminium-rich and the dark-greyish phase was of TiAl origin.

The white-coloured, Al-rich phase seems to be characterised by fine pores that formed into surface cracks. Meanwhile, the resulting microstructures seem not to be fully melted, but it is possible to identify the primary dendrites. In Figure 4b an aluminium rich-coating is seen. Likewise, this coating has fine pores and cracks on the bright white phase. The overall microstructures of the coating was resolved and was seen to be rich in primary lamellae that were formed and had coarsened; this probably being due to excess heating in the melt pool. The authors have a working theory that TiAl will form TiAl rods/woven-like microstructures at low-temperature sites which will, with sustained heating, transform into refined lamellar structures which then coarsen with continued, controlled heating and eventually form primary α-TiAl dendrites. The rods are formed due to heat loss and will be observed mainly at the topmost and the peripheral surface of the cladding. Figure 4c illustrates the forming α-TiAl dendrites. The primary trunks of the titanium dendritic structures are observed at 2.0 kW. The forming dendrites, like the lamellar structures observed at 1.5 kW, seem to grow in the vertical direction. EDS mapping across the overall coatings indicated that the Ti:Al ratio is 1.4, 1.6 and 1.8 for 1.0 kW, 1.5 kW and 2.0 kW coatings, respectively. The conclusion follows that the coatings might at least be $TiAl_2$-rich.

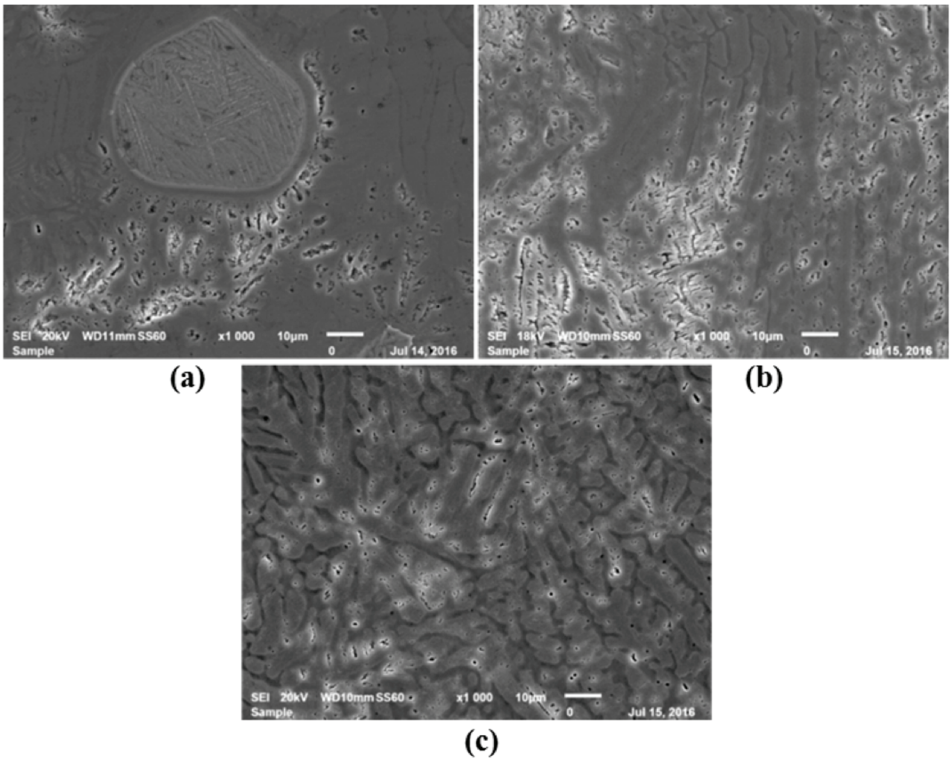

Figure 4. SEM micrographs of the TiAl coatings: (a) 1.0, (b) 1.5 and (c) 2.0 kW.

Hardness Measurements

The hardness measurements of the coatings were taken at the interface and across (traverse) the coating.

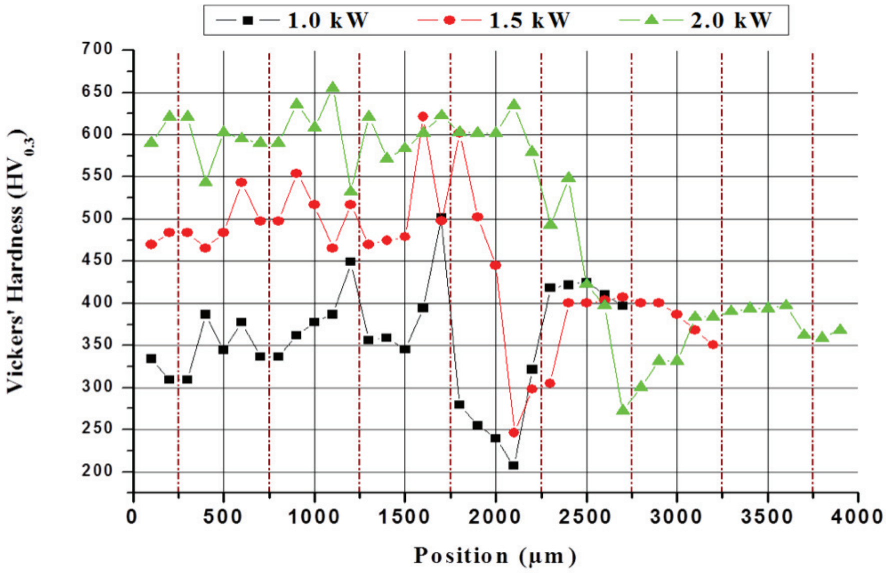

Figure 5. Vickers microhardness of the coatings.

Figure 5 presents the microhardness values of the produced coatings. It appears the coating became harder with increasing laser power. The average hardness values of the coatings were calculated as 357 $HV_{0.3}$, 451 $HV_{0.3}$ and 506 $HV_{0.3}$ for the 1.0 kW, 1.5 kW and 2.0 kW coatings, respectively. The hardness values taken across the interface were recorded. The overall results indicated that the coating-substrate bonding also increased with increasing laser power. The average hardness at the interfaces was calculated as 345 $HV_0.3$ (1.0 kW coating), 350 $HV_{0.3}$ (1.5 kW) and 364 $HV_{0.3}$ (2.0 kW). The hardness values and the EDS results were used for the phase match. The EDS spot was taken inside the hardness indents; this was an attempt to correlate the obtained HV to composition and then infer the phase. These results are reported in Figure 6.

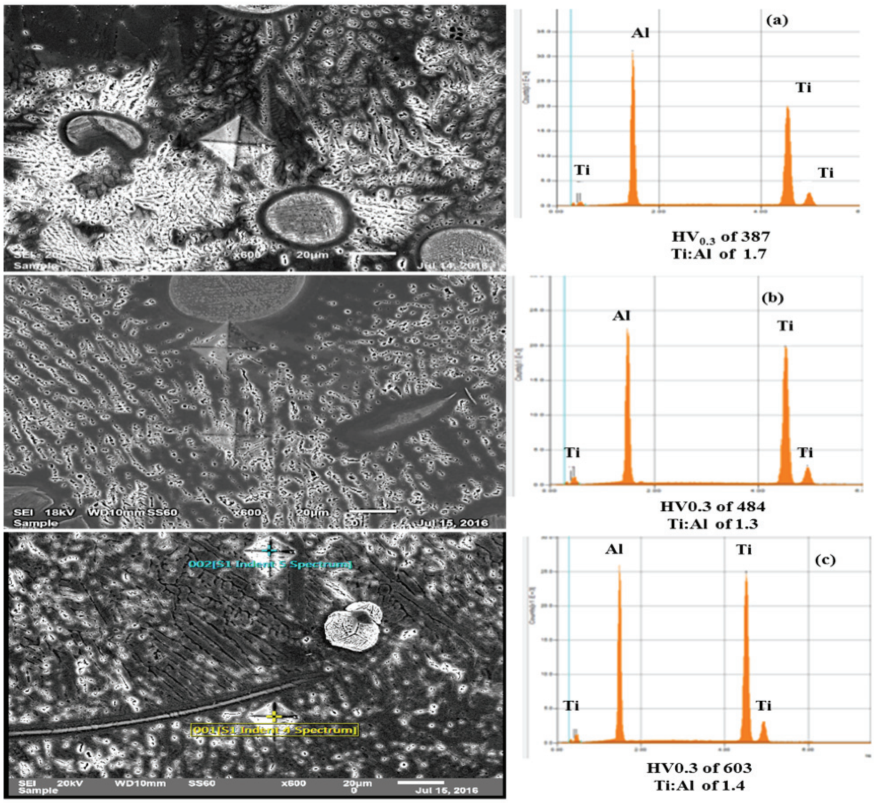

Figure 6. Hardness phase-matched using EDS analysis.

Typically, a harder surface is identified by a small pyramid that forms post indenting (Tlotleng *et al*,. 2014). All the indents show no crack formation on the edges, which is indicative of good strength. According to the hardness calculation, the hardness of the indented phases was 387 $HV_{0.3}$, 484 $HV_{0.3}$, and 603 $HV_{0.3}$ for laser powers of 1.0 kW, 1.5 kW and 2.0 kW, respectively. The dark phase was less hard than the white phase. The white phase indents seem to have similar Ti:Al ratios of about 1.4. This phase can be represented as $Ti_1Al_{1.4}$ while the dark phase would be $Ti_1Al_{1.8}$. Generally, the average Vickers hardness values for TiAl is 300 HV (Mwamba *et al*., 2012) or 350±5 HV and 285±5 HV for TiAl and Ti_3Al (Sun *et al*., 2011), respectively. The average hardness value for TiAl is reported to be 418 HV (Murr *et al*., 2010). Guo *et al*. (2007), using laser cladding process, observed a wavy hardness profile. The hardness increased with the aluminum content of the TiAl coatings.

DISCUSSION

Wang and Dahms (1993) showed that the reaction between elemental Ti and Al is by diffusion, where aluminium migrates into the core of the Ti particles to form different TiAl phases. At best, five reaction rates exist. Of the five, only two phases are thermodynamically stable products and the rest are known to be intermediates in the reaction sequence which form faster or more slowly. The reaction sequence is: Ti-Ti_3Al-TiAl-$TiAl_2$-$TiAl_3$. Only TiAl and Ti_3Al are thermodynamically stable. However, some researchers have shown that in an aluminium-rich environment, $TiAl_3$ can form as a stable phase while Ti_3Al_5 forms as a twin to TiAl during high-temperature transformation of Ti and Al into TiAl. The proposed theory suggests that during high-temperature processing of Ti and Al the resulting complex will first form a rod-woven microstructure due to lack of heating, which with continued laser processing, layer by layer, the sustained heating will transform rods into lamellae, which become coarsened with high heat inputs and finally transform into α-titanium dendrites. The formation of dendrites during the synthesis of TiAl from Ti and Al has been reported previously, while the formation and transformation of titanium dendrites at low to high temperature has been described by Gu *et al*.

(2007). The microstructures reported here clearly indicate that with the increase in laser power, which is directly correlated to the heat input, there is a microstructure transformation from a mixture of dendrites and lamellae at 1.0 kW to lamellar at 1.5 kW and finally to a dendrite-rich microstructure at 2.0 kW. Moreover, the increase in laser power led to a harder microstructure (Figure 5).

The transformation of the microstructure with increasing laser power is acceptable. In this work, identical compositions of TiAl were used to study the effects of laser power on the resulting microstructure, composition and hardness. Using the powder density formula it can be concluded that laser power is directly proportional to the overall energy transferred to the workpiece and therefore to the sustained overall heat within the forming melt pool. According to the TiAl phase diagram, changing the process temperature while keeping the composition constant will result in different phases being formed. Different phases always have different hardness values. The phase match between the EDS results and the hardness indicated that laser powers of 1.5 and 2.0 kW will produce coating with similar phases but different hardnesses. The phase might be correlated to the TiAl microstructure since Ti_3Al has a hardness below 300 HV. The EDS spot analyses correlated with the HV values presented in Figure 6. This is in itself acceptable, since most metallic surfaces can be hardened by post-heat treatment processing. Moreover, lasers are widely used for welding and to increase hardness or toughness (Aigbodion *et al*., 2016; Anandan *et al*., 2012).

CONCLUSION

The effects of laser power on the resulting microstructure, phase composition and hardness of the TiAl coating produced by *in-situ* alloying of Ti and Al were studied. The following conclusions can be drawn.

- Increasing laser power led to different microstructures
- The microstructure will evolve from mixed through lamellar to α-dendritic with increasing laser power
- The increase in hardness with increasing laser power is due to the difference in the degree of melting and the related microstructure
- The correlation between hardness and the EDS phase map can be used to infer the resulting phase of the as-produced coating. However, this is the subject of ongoing investigations and must be fully corroborated through XRD analyses on a large number of samples
- Overall, the results presented here demonstrated that Ti and Al can be melted into a TiAl phase composition. If a tailored microstructure with good hardness properties is to be attained the laser power must be above 1.0 kW and about 1.5 kW.

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Monnamme Tlotleng

Senior Researcher
National Laser Centre, Council for Scientific and Industrial Research.

Monnamme Tlotleng, PhD is a Senior Researcher at the CSIR National Laser Centre, Pretoria South Africa. He is a research associate at the University of Johannesburg in the Department of Mechanical Engineering Science. Monnamme has some scientific publications and is currently looking to establish his research in laser materials processing. His main focus is in laser materials processing looking into titanium and aluminium and their functional graded structures. He is currently doing work with titanium aluminides where he is looking to use 3D printing LENS machine to manufacture titanium aluminides structures at the NLC CSIR, South Africa.

Thabo Lengopeng

Lab Technologist
National Laser Centre, Council for Scientific and Industrial Research.

Thabo lengopeng is a Laboratory Technologist at the National Laser Center, CSIR. He is current working on his M-Tech Metallurgical Engineering at the Tshwane University of Technology. Thabo has some publication on the laser cladding of high carbon steel refurbishment, which is also his main focus towards his Masters studies. He is currently working on various projects within the National Laser Center.

Sisa Pityana

Principal Scientist
National Laser Centre, Council for Scientific and Industrial Research.

Sisa Pityana, PhD is a Principal Scientist at the CSIR National Laser Centre, Pretoria South Africa. He is a Professor at the Tshwane University of Technology in the Department of Chemical and Metallurgical Engineering. Sisa has numerous publications and has contributed greatly to the human capital development in South Africa and in Africa. His main focus is in laser materials processing looking into titanium, aluminum and other materials such as metal matrixes and functional graded. He is currently doing work with titanium aluminides and has recently established a world class laser shock pinning laboratory at the NLC CSIR, South Africa.