# MICROSTRUCTURE AND MECHANICAL PROPERTIES OF LENS FABRICATED TiAl STRUCTURES

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## ABSTRACT

It is difficult to produce titanium aluminides (TiAl) components because these materials lack ductility and when used as high temperature structures they fail prematurely due to the inability to self-oxidise. In this study, a 20 by 20 mm cube part was deposited on Ti64 substrate from elemental Ti and Al using LENS and laser power of about 400 W. The microstructural evolution across the sample concluded a lamella rich structure was produced. The EDS concluded an Al:Ti ratio of 1:4 which could mean that $Ti_3Al$ and TiAl were the resulting phases. Vickers's average hardness value of 290 $HV_{0.3}$ was reported. This HV value corresponds to the $Ti_3Al$ and TiAl phases.

Keywords: Aluminium, Hardness, LENS, Microstructure, Titanium and Titanium Aluminides

## 1. INTRODUCTION:

Due to their high temperature properties, titanium aluminides have emerged as strong materials to compete with nickel super alloys as thermal barrier coatings. The industrial need, since their development in 1955, has always been as coatings for high temperature applications and as structures for miniature gas-turbines. Titanium aluminides are interesting in that they are light in weight therefore once manufactured into structures or components they will lead to cost saving serving via fuel consumption while allowing ensuring performance and efficiency are relatively above the norm. The challenge faced by many industries; energy, aerospace and automobile, is that TiAl structures are not easy to manufacture at room temperature given that this material lacks ductility hence cracking is one of the main challenges. In structural engineering, it is documented that any material needed for load-bearing applications must be able to withstand high loads in operation because any crack would lead to failure which is always detrimental to the manufacturing company. This is why high performing materials are to be tested and qualified before being adopted by the aerospace industry.

Titanium aluminides can be fabricated using powder metallurgy processes, but the structural control is still a challenge while cracking presents the biggest challenge to date. It is worth noting that in any metallurgically finished products the microstructural texture of the resulting component will impact on its ability to function during application. The microstructural tailoring by any process is still the biggest challenge in titanium aluminides research. Typically, TiAl are synthesised using the functionally grading material approach. When powder metallurgy tools are used, it is easy to mix the individual powders into a composite via milling before being converted into a structure by means of pressing or moulds or casting. Such processes are known to cause microstructural segregation or produce multi-phase microstructure [1] REFERENCE NEEDED. Moreover, the process of manufacturing TiAl structure by mixing elemental powders of Ti and Al is still not fully developed. The challenge is getting a compositional mixture that would work since Ti and Al, under stable thermodynamic conditions, give rise to the five TiAl phases being produced that Ti3Al, TiAl, TiAl2, TiAl3 and γ-TiAl+α2-Ti3Al. Of the five forming phases, only three are of engineering importance as coating or free-formed structures which are TiAl, γ-TiAl+α2-Ti3Al and TiAl. TiAl and $Ti_3Al$ classified as gamma TiAl alloys are most important only if their microstructure can be refined. They are to be used in structure (boosters, gas turbines etc.) where high temperatures are experienced.

The γ-TiAl are low density, and good oxidation and creep material, and have good high temperature strength retention properties. Other possible structures of all TiAl families include near-gamma phase, duplex, near lamellar and fully lamellar. Wang and Dahms (1993)[2] detailed a comprehensive paper on the reaction mechanisms of producing TiAl from elemental Ti and Al. Their study concluded that only $Ti_3Al$ and TiAl should be present at equilibrium. Lei et al (2001)[3] studied phase orientation relationship in the TiAl-$TiAl_2$ region; this to establish if $TiAl_2$ forms as a stable phase and they concluded that TiAl, $Ti_3Al_5$, $TiAl_2^L$ and $TiAl_2^S$ form as stable phases. They further explained that these other phases are temperature dependent. For example, $Ti_3Al_5$ was found to be present, as a twin to TiAl, in all the heating and annealing temperature profiles and could only be isolated, enhanced and distinguished at 600°C. Doi et al (2002)[3] studied the morphological changes of the $Ti_3Al_5$ phase formed by phase-decomposition of TiAl intermetallics. Kosova et al (2016)[5] reviewed that in the Ti-Al system there is a potential to also form $Ti_2Al_5$ and $Ti_5Al_{11}$ in addition to the four known stable phases. These reviews clearly demonstrate that there is still much that is not known about the chemistry of selectively producing a desirable single phase component. This therefore directly suggests that there is a need to select a process during the developmental stages that will be of good use both to research and industrial development.

TiAl components are already in use as industrial structures. General Electric, GENx engine uses turbine blades manufactured from TiAl materials while there are turbo chargers rotors that are now commercially produced by the automotive companies to reduce load in vehicles so as to promote efficiency and performance. Unfortunately, such commercial products are manufactured from ternary powder of titanium aluminides as oppose to binary *in-situ* alloying approach. For example, the commercial blades that are used by the GENx engines are cast components made from Ti-47Al-2Cr-2Nb. Mwamba et al (2012)[6] reviewed that in an aluminium rich system only γ-TiAl phase can result as oppose to titanium rich system which lead two phase microstructure consisting of ordered face-centred tetragonal TiAl layers ($L1_0$) and close-packed hexagonal Ti3Al ($DO_{19}$). Using casting, the latter authors were able to study the microstructural, mechanical and oxidation property evolution of the γ-TiAl alloy. During casting, Ti and Al was alloyed with other precious metals and they concluded added precious metals did not completely change the initial structure of TiAl, but formed a new phase, mainly at the grain boundaries, in addition to the original formed or observed TiAl and the $\alpha_2$-$Ti_3Al$ lamellar. In addition, they observed a slight increase in the hardness and significant improvement in oxidation when the precious metals were added to the Ti-Al system. A thresh-hold of 0.2 At.%, was reported post which no significant improvements were achieved. The ternary system powders of Ti-Al are now commercially available. The third and fourth elements added are there to promote self-oxidation at high operating temperature and ductility at room to moderate temperatures.

The on-going research on the aluminides for high temperature applications makes use of several beta/alpha stabilising alloy with addition of elements that promote self-oxidation and ductility in the resulting microstructures. Muhammad and Basuki (2013)[7] used the electric arc furnace to melt specimens of 46% Al, 2% Cr, 2% Mo and Ti [all in At.%] and evaluated the packing mechanism and the resulting microstructure against hot corrosion behaviour at 850°C. Their results suggested that at 900°C corrosion was lowest and this was attributed to the observed thick layer of $TiAl_3$ that formed, also see Talabi et al (2013)[8], while at temperatures above

900˚C the available layer of Al had further diffuse leaving the surface bare. It is known that $Al_2O_3$ layer is most stable and corrosion resistant at higher temperatures when compared to $TiO_2$. It is evident from literature that the resulting TiAl microstructure is controlled by the amount of heat that is generated and how best it is controlled during cooling (Sina and Iyengar, 2015)[9].

Additive manufacturing (AM) systems are playing a significant role in the development studies of producing TiAl structures both at research and industrial level. Cormiere et al (2007)[10] used Arcam electron beam melting process to produced TiAl structures. Murr et al (2010)[11] characterised TiAl alloy components fabricated from pre-alloyed powder using the EBM machine and concluded improved specific yield strength when contrasted with the traditional Ti64 alloy. Shishkovsky et al (2012)[12] used direct laser metal deposition method to study FGMs structures of Ti-Al and concluded that if crack-free structures are to be obtained, the substrates must be pre-heated before. Liu and Dupont (2003)[13] used the laser engineered net-shaping to study the *in-situ* reactive process of Nickel Aluminides and concluded a well solidified structure that formed with the sub-solidus cracks and was porous. Cracking was said to be due to combined effect of the high thermal stresses inherent to LENS processing and brittleness of the intermetallics. Bandyopadhyay et al (2009)[14] reported a successful process when they manufactured porous and functionally graded structures that were intended for load bearing implants using the LENS machine. Hegab (2016)[15] published a review article on the design for AM of composite materials and potential alloys and concluded that FGMs are one of most effective composite materials proposed for AM since they offer the ability to control the composition and optimise the properties of the fully built part. In this paper we report on the microstructure, composition and hardness measurements of the TiAl (90%Ti and 10%Al) structures produced using the Optomec 1 kW LENS machine.

## 2. EXPERIMENTAL

The Laser Engineering and Net-Shaping (LENS) machine is classed as one of the AM systems. The LENS machine is a fully automated system with 5-axis capabilities. The system consists of a two computer screens; which are used for program manipulation and monitoring, Argon recycling system; that we refer to as the washing machine, a well-controlled process chamber that allows for oxygen to be minimised to about 10ppm so that high purity and desired phases of the printed materials can be retained. A laser beam and powder delivery system which is concentric in set-up is needed for accurate processing. The machine is capable to producing FGMs since it has multi-hopper systems which are individually controlled. The process set-up is illustrated by Figure 1.

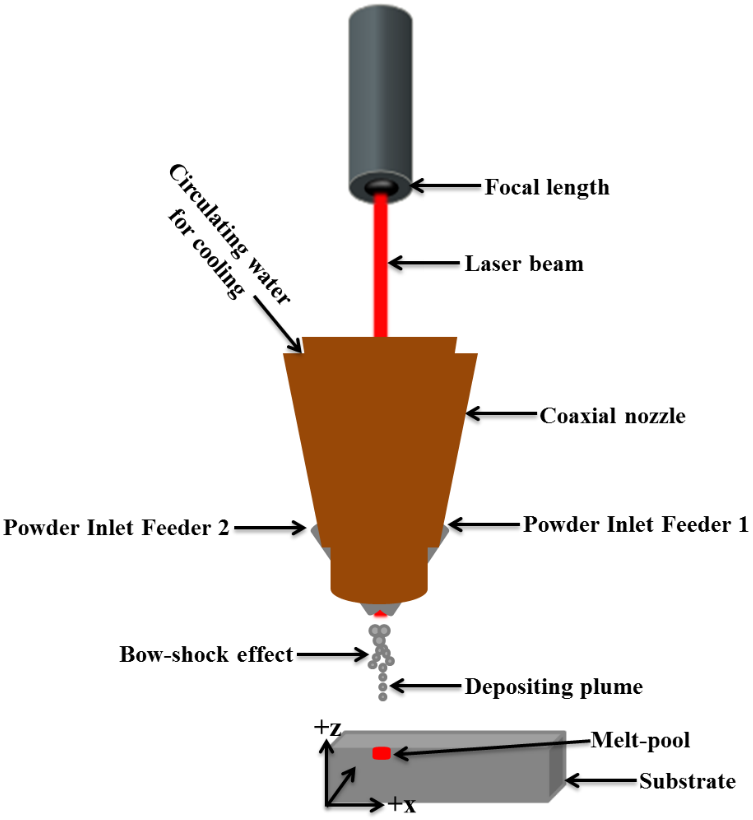


Figure 1: The LENS process set-up illustration.

## 2.1 Materials and Processing

Titanium and aluminium powders of 45-90 µm particle size distribution were used as feedstock materials. Ti-6Al-4V base-plate, of 4.5 mm thickness, was used as the substrate. A coaxial 4-way powder feeding nozzle was used to deliver Ti and Al powder into the melt-pool that was generated on the substrate. The laser head was automatically controlled and manipulated following the CAD file that was loaded onto the Optomec Application Launcher-Work Station Control Version 3.1.6 software. The powders are fed from hopper 1&2 simultaneously. The software will execute the dmi file. During printing the hatch and contour speeds were kept constant at 0.37 m/min and 0.53 m/min, respectively. The speed can be manipulated by adjusting the LENS deposition head speed which is in % terms. The chamber is typically filled with litres of Argon gas which is used for oxygen depletion and in our case it was also used as carrier gas. The produced cubes are of 90%Ti and 10%Al compositions.

## 2.2 Sample Preparation and Characterisation

After the deposition process, the samples were sectioned along the transverse direction across the clad layer for phase and microstructure analyses. Before mounting and polishing, the specimens' height was measured to be 13.33 mm after 36 layers of deposition which took about 22 minutes to execute. The deposition rate was calculated at 0.6 mm/m. After sectioning, the samples were ground and polished to a 0.04 micron (OP-S suspension) surface finish using a Struers TegrsForce-5 auto/manual polisher. Post polishing the samples were etched with Keller's reagent for 2-3 minutes and then analysed for microstructures using Olympus light optical microscope which connected to Analysis® software.

The prepared samples were characterised for microstructure and elemental analyses using Joel JSM-6010PLUS/LA scanning electron microscope (SEM) that was equipped with energy dispersive X-ray spectroscopy (EDS). The SEM-EDS system uses the Intouch Scope software for analyses. The SEM system is equipped with a tv camera that allows for sample stage height to be visualised and controlled. The phase compositions of the coatings were determined by Panalytical XPert Pro PW 3040/60 X-ray diffraction with Cu Kα monochromater radiation source. The phases were identified using material PDF files.

## 3. RESULTS

The reported microstructures were taken at the top, middle and bottom of the LENS produced TiAl cubes.

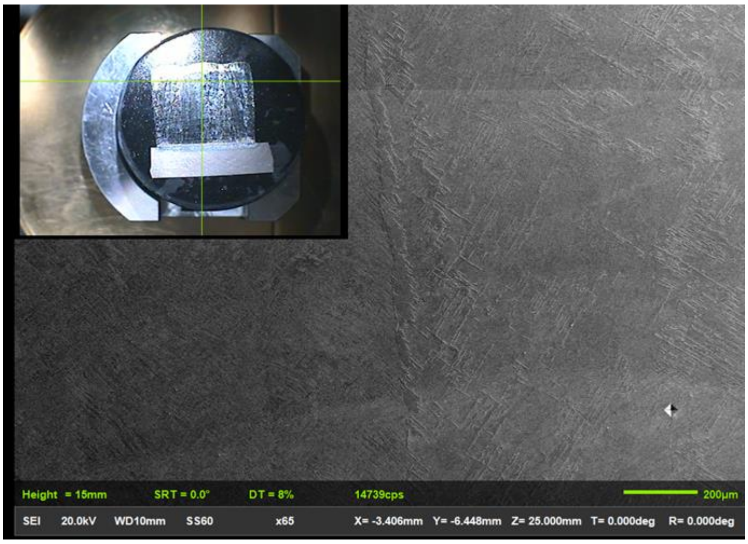
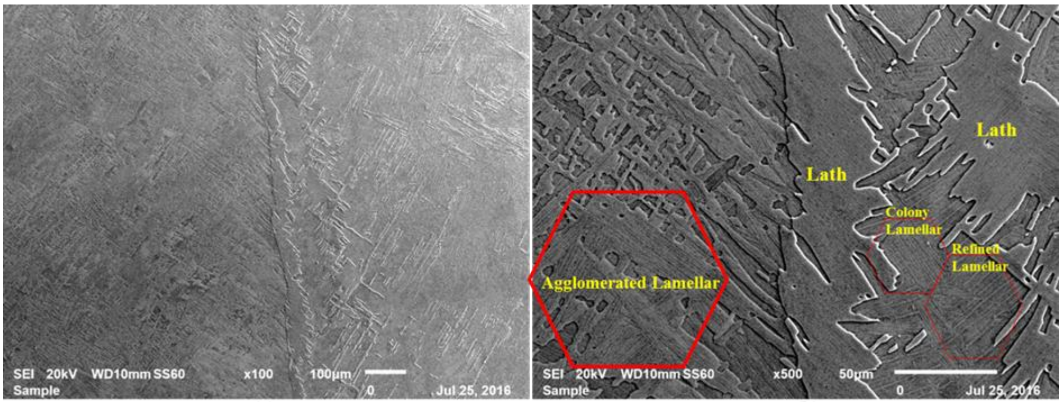


Figure 2: The as produced LENS sample showing top position of imaging.



(a) (b)

Figure 3: SEM images of the LENS coating (top).

Figure 2 show the as produced microstructure of the TiAl cube that was fabricated with the LENS machine. The microstructure concludes that no obvious cracks or pores were observed at the high magnification as indicated in Figure 3(a) and 3(b). The overall structure was lamellar rich (refined, clusters or colony and agglomerated). The agglomerated lamellar structures formed TiAl laths. This is well identified on the presented microstructure in Figure 2(b).

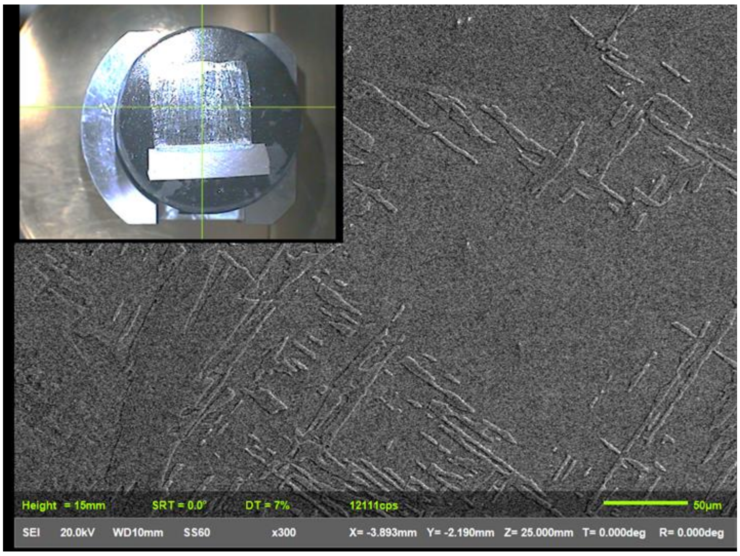
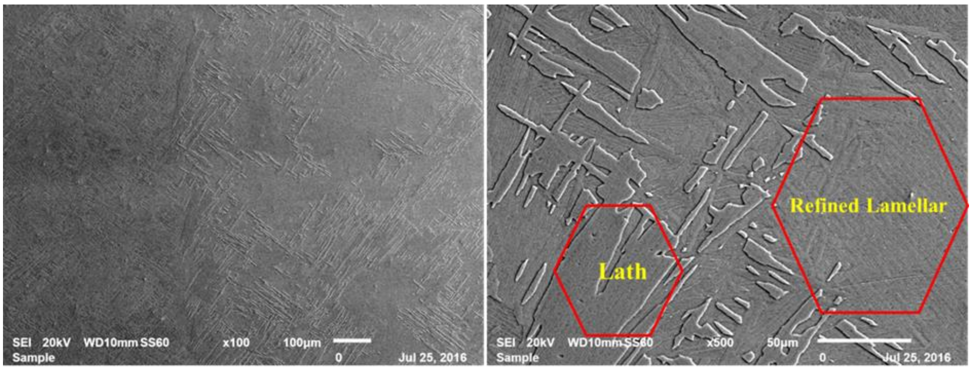


Figure 4: The as produced LENS sample showing middle position of imaging.



(a) (b)

Figure 5: SEM images of the LENS coating (middle).

The middle part of the TiAl is lamellar rich and has lath structures that formed. The refined lamellar structures seem to dominating the right side of the image while the lath structures are easily spotted on the left side. These details are highlighted in Figure 5(b).

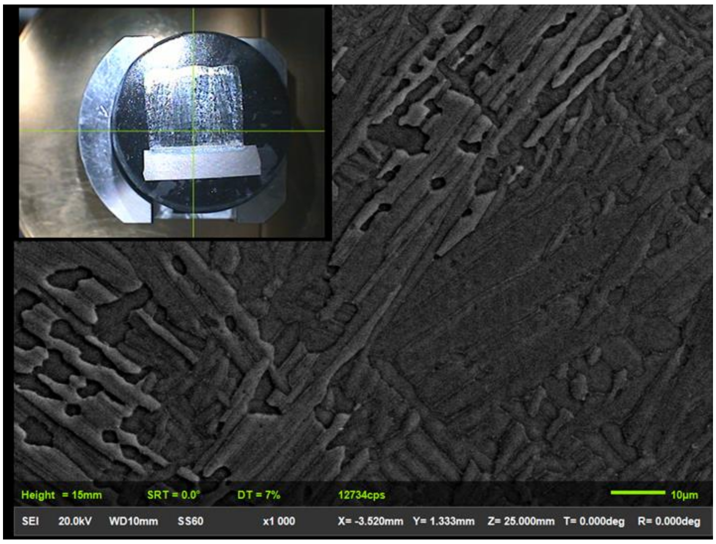


Figure 6: The as produced LENS sample showing bottom position of imaging

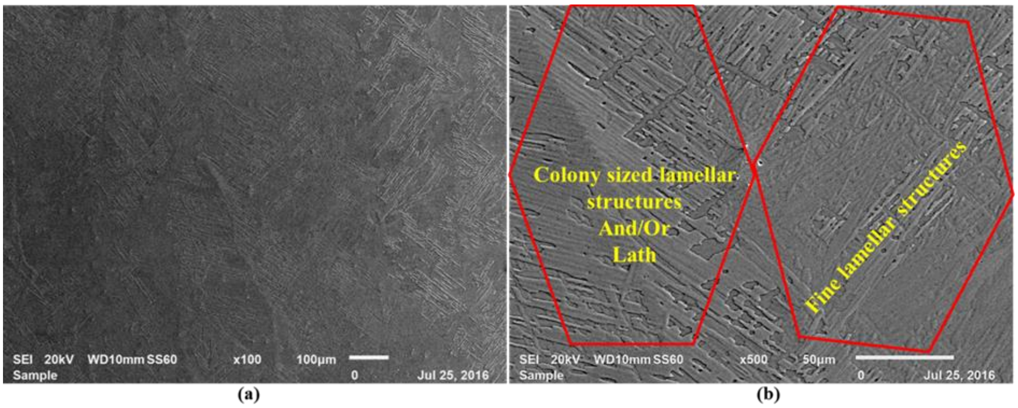


Figure 7: SEM images of the LENS coating (bottom).

The bottom part of the TiAl is lamellar rich and has lath structures that formed probably due to the unrefined lamellar that formed colonies of TiAl. The refined lamellar structures seem to dominating the right side of the image while the lath structures are easily spotted on the left side. These details are highlighted in Figure 7(b).

## 3.1 Composition

To understand the similarity and differences in the microstructures presented in Figures 2-7, the mapping on the different portions of the coating (bottom, middle and top) was conducted. The results are given in Figure 8.

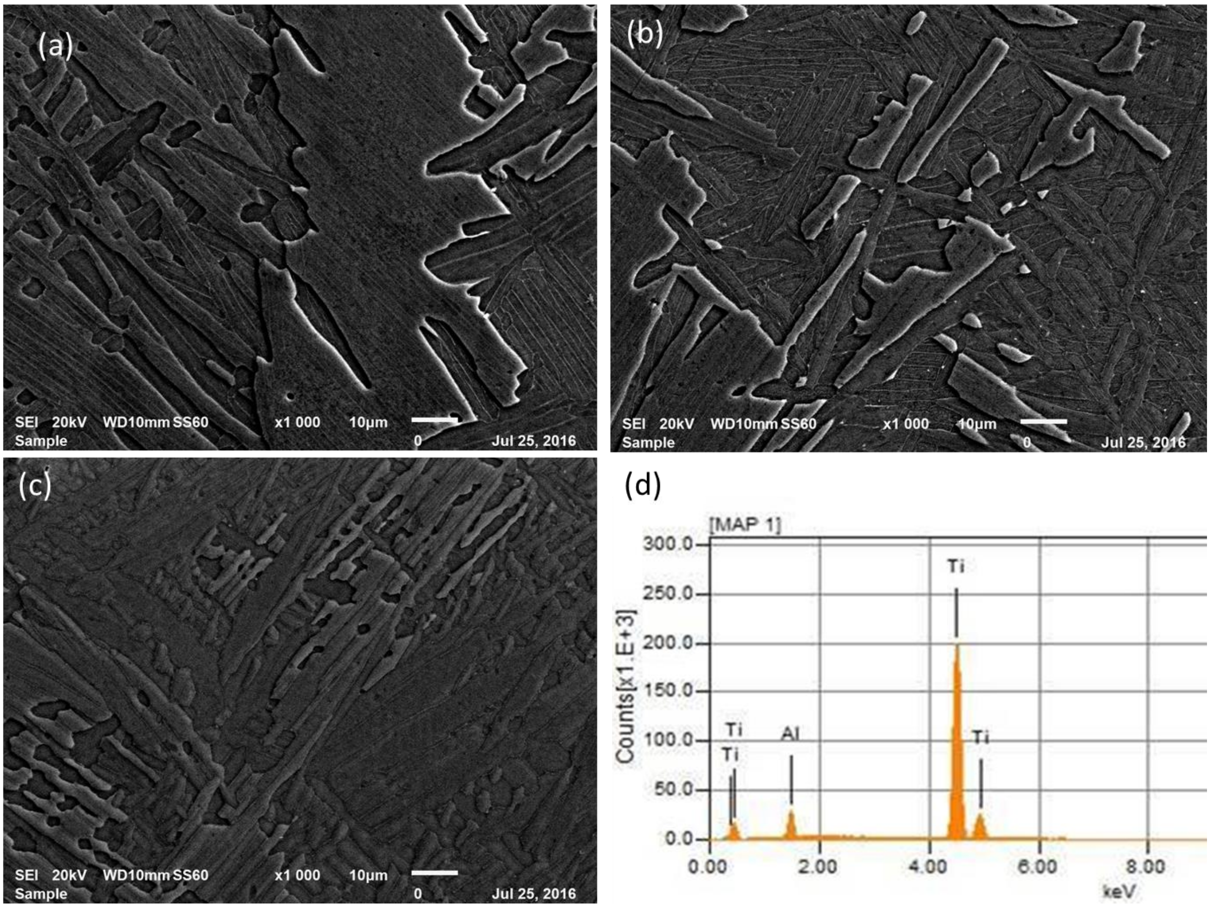


Figure 8: The highly resolved SEM images used for composition analyses (a) top, (b) middle and (c) bottom.

Obviously, the details captured in Figures 2-7 are now fully resolved in Figure 8. At this magnification it is easy to distinguish that the lath structures are dominant at the top of the built while the middle part comprises of both lath, refined and coarse lamellar TiAl structures. The bottom part can be said to be rich in average spacing lamellar, coarse lamellar or thin laths lamellar structures. The EDS mapping concluded that compositional wise the Al:Ti ratio of the regions were as reported in Table 1.

**Table 1: EDS mapping of the produced LENS TiAl cube**

| ***Region*** | ***Ti*** | ***Al*** | ***Al:Ti*** |
|---|---|---|---|
| **Top** | **89.37** | **10.63** | **1:8.4** |
| **Middle** | **89.40** | **10.60** | **1:8.4** |
| **Bottom** | **88.02** | **11.98** | **1:7.3** |

Table 1 summarises that the composition of the coating was homogenous from bottom to the top and was of the $Ti_xAl_y$ phase not pure γ-TiAl phase.

## 3.2 Hardness

The hardness measurements of the coating, taken in the vertical direction, are reported in Figure 9.

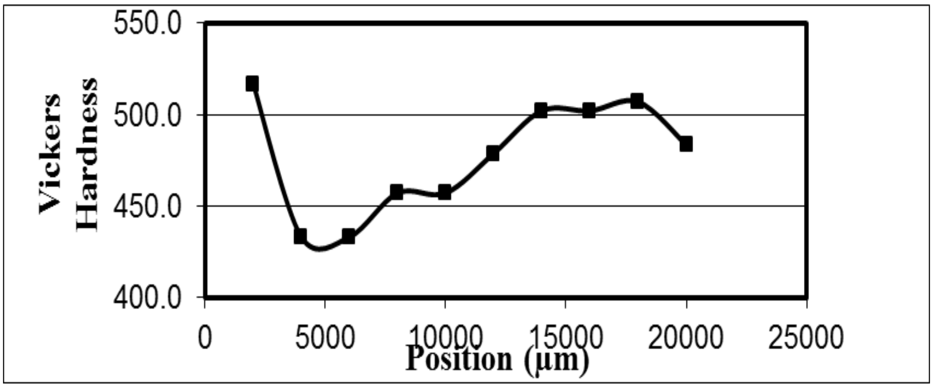


Figure 9: Vickers' micro-hardness plots.

Figure 9 presents the recorded micro-hardness ($HV_{0.3}$) of the LENS produced structure. It seems the hardness ranges between 450 and 530 $HV_{0.3}$. The average hardness per region is reported in Table 2.

Table 2: Vickers' micro-hardness values taken vertical and at the interface across the LENS structure.

| *Region* | *$HV_{0.3}$* |
|---|---|
| **Top** | **460** |
| **Middle** | **468** |
| **Bottom** | **504** |
| **Interface** | **363** |

Table 2 reports on the average hardness of the structure per region and at the interface across the structure. The hardness value of the interface is lowest followed by the middle and top and then bottom region. The hardness of the top and middle are the same and similar to the hardness of the bottom of the structure. The EDS spot analyse taken on every indent concluded similarity in composition per region.

## 4. DISCUSSIONS

The LENS produced Ti-Al structure is reported in this paper. The microstructures of the produced coating were evaluated by means of regions. Three regions were identified into top, middle and bottom. The microstructural evaluation revealed that the produced cube was lamellar rich and in some instances the non-refined lamellar agglomerated to form a lath structure. Even so, the resulting microstructure could be said to be lamellar rich. The fine lamellar seems to form in air cooling contrast to coarse lamellar which forms in slow cooling conditions. The LENS analyses is always conducted under controlled environment this would explain the observed lamellar which transformed from fine to coarse and finally into lath TiAl structures. The similar microstructures that were observed were in agreement or supported by the overall mapping results and the hardness measurements. The EDS-mapping concluded that the cube structure was mainly of the TixAly nature which had similar hardness across.

Ma et al (2014)[16] reported that thin lamellar had maximum hardness when compared to the thick lamellar and inter-dendritic γ-phase. The average Vickers hardness values for TiAl is 300 HV (Mwamba et al, 2012) [6] or 350±5 HV and 285±5 HV for TiAl and $Ti_3Al$ (Sun et al, 2011)[17], respectively. Murr et al (2010)[11] reported the average hardness value of 418.1 HV for TiAl materials produced with EBM. Guo et al (2007) [18], using laser cladding process, observed a wavy hardness profile. The hardness increased with the aluminium content in the formed TiAl coatings. Their 80Ti/20Al coating was least hard, by extrapolation, with the average hardness of about 630 HV at the top and 460 HV in the middle. Looking at this reviewed data and considering the EDS spot done on the hardness profiles or the pyramid indents there is clear correlation between same phase and the indent hardness and the composition. Overall, then the hardness measurements like the microstructure indicate that the LENS produced cube of TiAl by in-situ alloying approach produced a homogenous microstructure.

## 5. CONCLUSION

The LENS system was evaluated if it could produce defect free sample built from Ti and Al by in-situ alloying approach. The powders were delivered into a melt pool simultaneously to make a TiAl with a composition of 90%Ti and 10%Al. The microstructure revealed a homogenous LENS produced TiAl structure that had similar composition and hardness. In the future we wish to manufacture functionally grading material of the TiAl by means of in-situ alloying and compare them to structures produced from pre-alloyed powders using additive manufacturing systems.

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