MICROSTRUCTURAL EVOLUTION OF THE LENS MANUFACTURED TIAL STRUCTURE

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ABSTRACT

The advent of additive manufacturing presented the new-era where complex structures can be prototype and rapidly manufactured from a computer aided device file. Robust industries such as the aerospace and medicinal require 3D printed complex components which can be reproduced and are without defects. The laser engineered net-shaping (LENS) system is an additive manufacturing machines and can be used for industrial research and prototyping. In this paper, the Optomec LENS; a 1 kW maximum power output system, was used to fabricate a cubic structure of titanium aluminides from elemental titanium (Ti) and aluminium (Al) by *in-situ* alloying. This is a novel approach since the traditional methods used in the production of TiAl structures focus primarily on powder metallurgy and electron beam melting (EBM) process where pre-alloyed powders instead are used. These pre-alloyed powders are superior since they already have oxidation promoting and ductility stabilisers element. The resulting microstructure of the *in-situ* alloyed Ti and Al powders were evaluated, bottom-up, on the produced structure. The SEM images concluded a lamellar rich microstructure at the top, middle and the bottom. The HAZ showed a hexagonal structure. The EDS map of the different regions concluded Ti:Al ratio of 3:1 which could mean the overall composition was Ti_3Al.

Keywords: Aluminium, Scanning Electron Microscope, LENS, Microstructure, Titanium and Titanium Aluminides.

1. INTRODUCTION

There is a potential to use titanium aluminide (TiAl) alloys as replacements for Ni-based supper-alloys in structural components required by the aerospace and automobile industries [1] [2]. The reaction between titanium and aluminium lead to three stable aluminides compounds, namely ɣ-TiAl, α_2-$TiAl_3$ and $TiAl_2$ [4]. The gamma (γ)-TiAl alloy in particular, is interesting because it is lightweight and good high-temperature strength material. γ-TiAl is characterised of low density, high melting point and acceptable oxidation resistance at both room, and moderately to elevated temperatures. Its draw back however, is its low ductility, and difficulties associated with shaping the alloys when using traditional metal working methods, such as machining, cold working and casting [5].
Titanium aluminides can be produced as functionally graded materials (FGMs) to serve in severe changing temperature and creep intensive environments. FGMs were initially developed by Japanese researchers in 1980's [6]. This breed of materials is proposed to increase structural integrity, particularly at interfaces were failure occurs, while they reduce the sudden changes in local stress load that occurs when materials undergo complex environmental changes. There are two classifications of FGMs and these are: continuous structure and step-wise structure as presented in Figure 1.

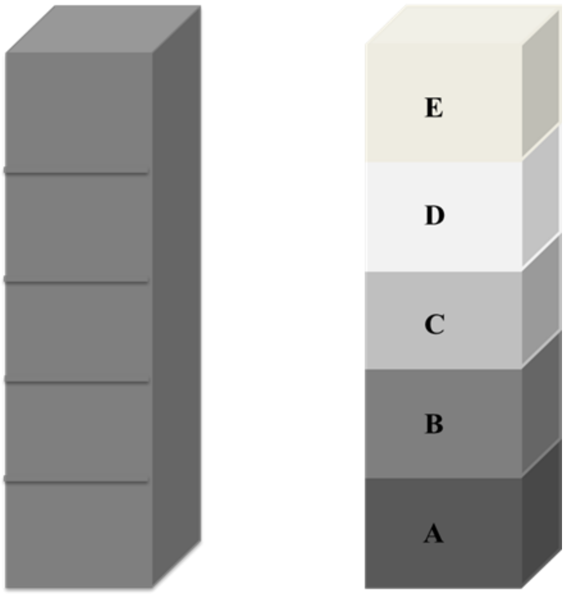

Figure 1: Functional grade materials of a continuous structure (left) and step-wise building (right) [5].

Homogenously built FGM structures are achieved using a single phase metallic material and are normally used for wide range of engineering applications. Compositionally graded structures are needed for applications where composition, physical structural change; chemical and thermal properties are needed during use. Such a structure is the one presented in Figure 1 on the right. The outcomes of FGMs are obviously directed towards microstructural tailoring targeted towards an engieering application [5].

FGM materials can be fabricated using several different techniques. It is important to note that FGMs can be produced as surface coatings and as readily manufactured structures; this therefore allows for a process selection. Mahamood et al [5] described the different techniques that are available to produce FGMs. Surface coatings of FGMs can be called thin FGM while a finished structure can be classified as bulk FGM. Surface coating techniques like cold spraying, cladding, thermal spraying, physical and chemical vapour deposition, etc. can be used to surface coat FGMs while bulk FGMs require metallurgical processes like casting and forging, and expensive processes that allow for fabircation of solid

free form structures. A review by Sobczak and Drenchev [9] captured and articulated the different techniques and the resulting microstructures of FGMs and their possible applications. They further described techniques called solidification of graded slurries and graded structure coatings and plates. These two coatings were outstanding in that grading led to different microstructures being achieved per layer.
The idea of layer by layer complex components production has been recently realised by additive manufacturing processes. Suryakumar and Somashekara [10] stated that additive manufacturing as the state of the art to be used for the production of FGMs where products or components can be directly printed from a click of a button making use of the CAD models. Bandyopadhyay and Krishna [8] used the laser engineered net-shaping technique, also an additive manufacturing system, to manufacture porous and functionally graded structures for load bearing implants. The laser additive manufacturing technique (3D printing) presents a viable alternative with regards to producing near net shaped components[11].

In this paper we used the Optomec LENS machine to synthesise titanium aluminide coupons by in-situ alloying elemental Ti and Al into miniatures cubes. Studies reporting on the *in-situ* alloying approach of TiAl are limited; to the best of our knowledge, but scientific papers reporting on the produced structures of TiAl from pre-alloyed powder using AM processes are numerous.
In this study we report only on the microstructures and phases of the LENS-produced TiAl coupons (See Figure 2). Since multiple phases as produced when melting Ti and Al into TiAl,there need to develop and establish a process that facilitates the tailoring of the TiAl microstructure using the LENS. Such a process will enable the fabrication of structures that can be used in industries.

Figure 2: LENS fabricated TiAl coupons by method of in-situ alloying.

2. EXPERIMENTAL SET-UP

The Laser Engineering and Net-Shaping (LENS) machine is classed as one of the AM system. 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; an Argon gas 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 the desired high-purity phases of the printed materials can be retained. A laser beam and powder delivery system that 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 3.

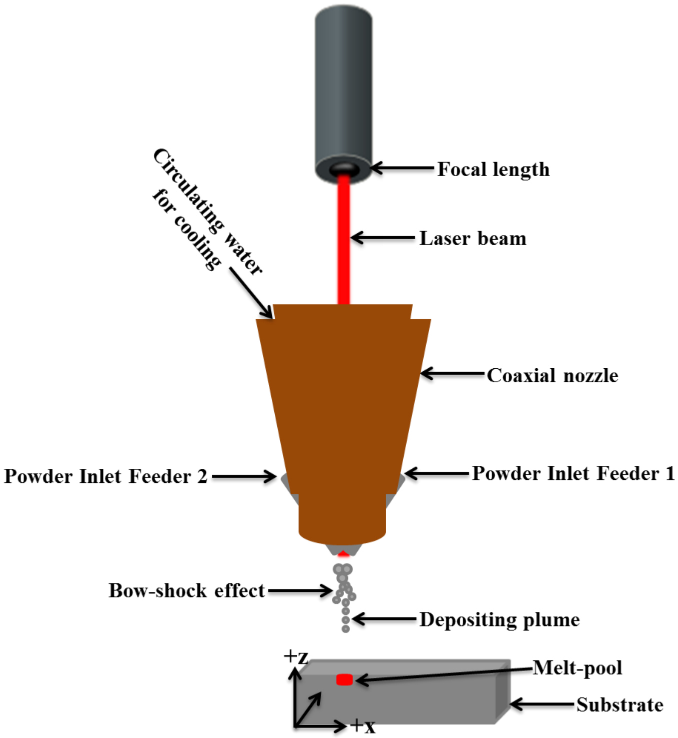

Figure 1: The LENS process set-up illustration.

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 were fed from hoppers 1&2 simultaneously. The software executed 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 was manipulated by adjusting the LENS deposition head speed which is in % terms. The chamber was filled Argon gas for oxygen depletion and as a carrier gas. The produced cubes comprised 90% Ti and 10% Al.

2.2 Sample preparation and characterisation

After the deposition process, the samples were sectioned along the transverse direction across the clad layers 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 an Olympus light optical microscope which was 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 AND DISCUSSION

Material characteristics have impact on their overall performance and durability. Moreover, materials needed for structures generally undergo rigorous qualification process before they can be qualified and adopted. The aerospace vehicles will use TiAl materials that are ductile and that will be able to withstand high temperatures. The ability for these intermetallic materials to perform is linked directly to the resulting microstructure. A homogeneous, single phase microstructure is required when manufacturing parts for the intended applications. Knowing this, the results obtained on the microstructures and elemental by SEM and phase composition by XRD are discussed collectively. The microstructures results presented here were taken from the top, middle and bottom of the sections of the produced TiAl cube.

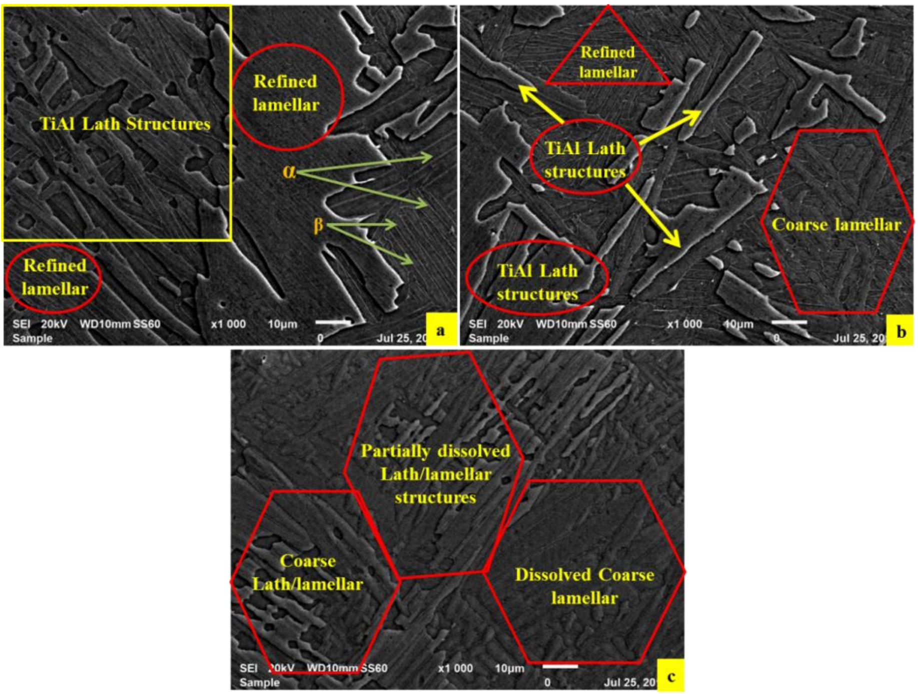

Figure 2: SEM images of the LENS produced TiAl cube (a) top, (b) middle and (c) bottom.

Figure 4 presents the SEM images of the LENS produced TiAl structure taken at different regions. The top structure of the built cube consists mainly of refined lamellar TiAl structures and agglomerated lamellar or colonies of lath TiAl structures. The beta and alpha phases of the lamellar structures can be identified were a defined spacing between the lamellae can be identified. The bottom, right corner in Figure 4(b), shows a refined lamellar structure that can be identified as γ-TiAl. Similar structures can be seen in Figure 4(b), but here the lath formed structure seems to be form thinner stripes compared to the lath structures present in Figure 4(a). In fact, this image reveals that three possible kinds of lamellar structures are formed during processing: coarse, refined and lath. Figure 4(c) indicates that the most dominating type of lamellar that formed at the bottom of the produced cube was coarse lamellae, partially dissolved laths and dissolved laths.

Figure 4(a-c) reveals that a homogenous TiAl structure can be produced using the LENS machine but with different types of the lamellar structures. The LENS process is a heat intense process and this heat is not equally distributed in every layer during building. It appears that the first layer (bottom) experiences more heat as the second layer is deposited on it and process continues like that until the structure is completed. The process of layer by layer building is known to cause remelting on the previous layer which then would have a different microstructure when compared to the subsequent overlaid layer [6]. This is affirmed by the images presented in Figure 4. The remelted lamellae are only identified at the bottom (image c) of the structure which may have been repeatedly melted with every layer being built under the completion of process. Moreover, this layer experiences most heat which is retained by the base metal and cools over a longer period than the last layer which would cool off immediately. It is already established that fine lamellae form in air cooling environments or rapid cooling whereas coarse lamellae form under slow cooling conditions [6]. This phenomenon is supported by the images presented in Figure 4.

3.1 XRD: Phase composition

The phase compositions of the laser *in-situ* produced TiAl coatings were obtained by means of analysing the diffraction peaks (Figure 7). The assignment of the diffraction peaks were performed according to the PDF files no: $TiAl_3$ (04-018-4873), Ti_3Al_5 (00-042-0810), $TiAl_2$ (00-042-1136), $Ti_{1.5}Al_{2.5}$ (04-007-2384), Al (04-012-3403), Ti (00-005-0682), TiO_2 (00-015-0875), Al_2O_3 (00-004-0877), $AlTiO_2$ (00-052-1557), α_2-$Ti_3AlO_{0.01}$ (04-013-6696), α_2-$Ti_3AlO_{0.16}$, α_2-$Ti_3AlO_{0.31}$, $Al_3Ti_5O_2$ (43-4473), $Ti_{2.78}Al_{1.22}O_{0.173}$ (01-074-9926) and $Al_{1.24}O_{0.359}Ti$ (01-074-9927). The X-ray diffraction pattern of the structure is presented in Figure 5 and the peaks' positions and possible compounds are summarised in Table 2.

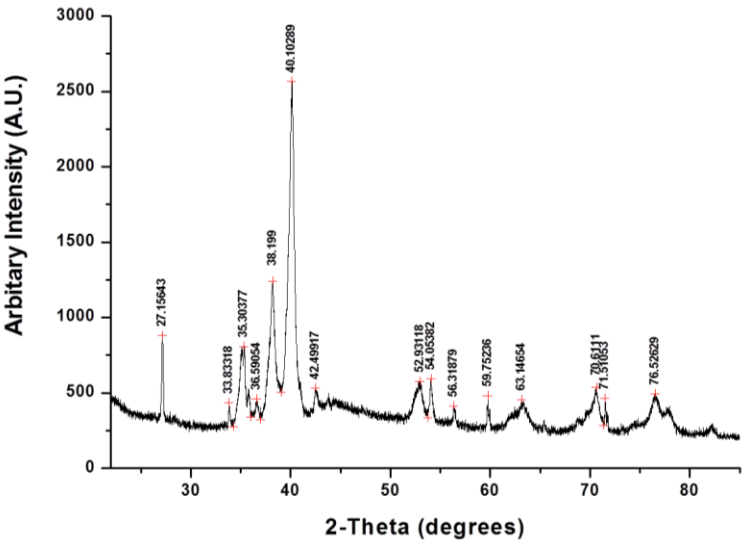

Figure 3: X-ray diffraction peaks of the LENS produced structure.

Table 1: The summary of the diffraction peak positions

Al	**38.20 (1 1 1)**
Ti	35.30 (1 0 0); 40.10 and 52.93
Ti_3Al	27.16; 70.61 (2 0 3) and 71.51 (3 1 1)
Ti_3Al_5	33.83; 36.59; 54.05 (3 2 2); 56.31 (4 0 2) and 59.75 (6 3 1)
TiO_2	42.49
$Al_3Ti_5O_2$	63.15 (3 2 2)

According to the results of the diffraction peak summarised in Table 2, the major peaks of the coating are of pure titanium. Thepeak at 2θ value of 27.16 corresponds to Ti_3Al, whichis one of the main stable phases in the Ti-Al system. The data in Table 2 indicate that Ti_3Al_5 was also present in the LENS product. Ti_3Al_5 seems to be significant in this structure even though it was not detected as one of the most intense group of peaks. The presence of Ti_3Al_5 as oppose to the γ-TiAl phase has been reported in the literature [7. This phase is known to form when Ti and Al react under high temperatures. A titanium aluminide oxide ($Al_3Ti_5O_2$) phase is also present making this material interesting for high temperature applications [2]. The presence of $Al_3Ti_5O_2$ suggests oxidation during layer deposition by way for the following reation:

$$Ti_3Al + 2TiAl + O_2 \rightarrow Ti_5Al_3O_2 \quad (1)$$

4. CONCLUSION

The possibility of synthesising TiAl structure from elemental powders of Ti and Al using LENS making use of the *in-situ* alloying approach was presented in this paper. The microstructural and XRD results indicate that the LENS product composition was consistent throughout the products and consisted mainly of Ti and Ti3Al phases with additional (minor?) Ti_3Al_5 and the oxide phases TiO_2 and $Al_3Ti_5O_2$. However, no quantitative analysis was done on the XRD data to provide conatraints on the relative proportions of the different phases. Nevertheless, these results are promising and present a process window development for further research development.

5. ACKNOWLEDGEMENTS

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