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# EFFECTS OF HEAT-TREATMENT ON THE MICROSTRUCTURE OF TiAl-Nb PRODUCED WITH LASER METAL DEPOSITION TECHNIQUE

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## Abstract

Ti and its intermetallic alloys are light-weight and have good creep properties. Materials of this nature are desired for the manufacturing of parts for the aerospace, automotive and power plants. Titanium aluminides, in particular, are preferred due to their lightness and excellent creep properties when compared to nickel super-alloys. This paper looked into the production of titanium aluminide microstructures, the so-called ordered $\alpha_2$-$Ti_3Al$-Nb, making use of the *in-situ* laser metal alloying additive manufacturing approach, and the impact of heat treatment on microstructural evolution and hardness measurements. Ti-33Al-8Nb alloy was produced by melting the elemental powders in a laser melt-pool. The resulting microstructure was characterised for composition and hardness measurements before and after heat treatment. The As-produced sample was dense and had lamella with the observed grains. Micro-hardness of $555_{0.5}$ was reported. Heat treatment at 1200°C, as oppose to other heating profiles, resulted in a homogeneous structure with refined lamella observed inside the preserved grain structures. Contrary, non-homogeneous structure with coarsened lamella inside elongated grains was observed at 1400°C. Micro-hardness of $452_{0.5}$ was reported. These results are summarised in Figure 4.

## Introduction

New and attractive class of functionally gradable and intermetallic materials that can be formed into structures or used as surface barrier coating called titanium aluminides are under research and development [1]. According to the Ti-Al binary phase diagram, elemental Al and Ti can react to form several titanium aluminides intermetallics. It has however become accepted that only three stable phases are of engineering interest [2, 3]. The $\alpha_2$-$Ti_3Al$, $\gamma$-TiAl and ($\alpha_2$+$\gamma$/$\gamma$+$\alpha_2$) can be formed into parts that are of greatest significance and valuable to industries such as the aerospace, energy and automotive. These intermetallics are light weight (low density) and have excellent high-temperature properties making them necessary as material of choice for high temperature gas reactors, compressors and other high value components. It is recognised that if their room temperature ductility shortcomings were to be timely resolved and if their route to production can be repeatable (microstructure and mechanical properties) in a cost effective way, these aluminides would gain world interest over nickel super-alloys which are heavy and have limited creep properties [4].

Research on this field has primarily focused on the improvement and development of the manufacturing steps involved during the synthesis and manufacturing of the TiAl alloys using powder metallurgy processes. In general studies agree that casting, for example, will produce TiAl alloys with lower ductility and damaged tolerance at room temperature as well as low workability at elevated temperature [5-6]. More importantly, Imayev et al [5] indicated that cast TiAl alloys have a coarse-grained microstructure, a sharp casting texture and significant chemical inhomogeneity. The ternary phase TiAl alloys seems to be showing significant improvement on formability and performance of these aluminides at both room and high temperature [7]. There are several published research findings that particularly looked into the improvement of the $\alpha_2$-$Ti_3Al$ by adding niobium (Nb) [8-12]. Germann et al [6] presented a comprehensive introductory review on the development of the ternary TiAl alloys and their significance to forming processes, structural engineering and performance of the aluminides research herein.

In brief, it can be ascertained that the first study that showed the room temperature ductility improvement of the ordered $\alpha_2$-$Ti_3Al$ by adding Nb was by McAndrew and Simcoe in 1960 with the first patent registered by H. Winter in 1968 [6]. They [6] Studied the effects of composition on the material properties of newly developed $Ti_2AlNb$-based titanium aluminides and discovered that adding Nb to orthorhombic alloys improves their oxidation resistance behaviour, but beyond 15 at%, Nb the oxide becomes less protective and the oxide scale appeared less adhesive and more porous at 800°C. Other researchers would later add the fourth and fifth element to improve on the creep properties. To date a significant number of TiAl alloys, viz., General Electric (Ti4822), Plansee and GKSS exist. Many different manufacturing techniques have been investigated towards the improvement of the microstructure tailoring (homogeneity) and resulting mechanical properties [13]. Gasper et al [14] used the direct metal deposition (DMD) technique to study *in-situ* synthesis of TiAl alloy making use of the so called satellite process while others use the pre-alloyed powders. In this study a DMD process was used to produce a TiAl-Nb alloy by feeding a master powder of Ti-Nb and Al, independently, into a laser melt pool in which upon cooling a TiAl-Nb alloy would result. These current processes seem to be winning on the homogenisation and refinement phase while producing components with correct mechanical properties.

The aim of this study was to demonstrate the feasibility of producing the TiAl-Nb alloy *in-situ* using the laser direct metal deposition technique and subsequently study their microstructure and hardness before and after heat-treatment. The microstructural and hardness profiling characterisation was such that the effects of heat treatment on the resulting microstructure of TiAl-Nb can be related to mechanical property.

## Experimental methods

To study the feasibility of *in-situ* laser metal alloying during the production of the stable $\alpha_2$-$Ti_3Al$ through the addition of Al powder to the master alloy Ti-Nb, a high power laser system was required. In our study a three kilowatt (3 kW), IPG continuous fibre laser system was used to generate a melt-pool on the base metal into which the depositing metal powders will be convectionally mixed then alloyed into a $\alpha_2$-$Ti_3Al$-Nb upon cooling. Our process set-up comprised of the powder feeding system with two hoppers, bulk argon supply system, IPG laser, copper 3-way coaxial nozzle, Kuka robot arm and the X-Y table. The feeding nozzle and the laser head were mounted onto the robot arm and controlled automatically from the pendant. The materials used were pure titanium, pure aluminium, pure niobium powders and the Ti-6Al-4V substrates. Titanium and aluminium were supplied by TLS, Technik GmbH & Co and had were spherical and particle size distribution of 45-90µm while the Nb powder was irregular and supplied by Weartech and had particle size distribution of about 45-120µm. The Ti-6Al-4V substrates with 70 x 70 x 5 $mm^3$ dimensions were used.

Before process the acetone cleaned Ti-6Al-4V plates were clamped on the process table and a stand-off distance of 12 mm and beam incident angle of 12° were set. This is such that the back-reflections can be avoided during processing. During processing, the Ar gas was used as both the process and shielding gas. The powders being processed were contained in two separate hoppers that were automatically controlled and allowed for efficiency in the delivery and deposition. One hopper contained the Ti-Nb powder (unalloyed or mechanical mixed) which was delivered to the processing zone at 1.8 l/min carrier gas flowrate while the feeding disc was rotating at 2 rpm. The other feeder contained pure Al powder which was delivered at 2 l/min carrier gas flowrate while the feeder disc was rotating at 1 rpm. The optimisation process parameters that were used are laser power (1-2 kW); beam spot size (2-4 mm); shielding gas flowrate (15 l/min); powder carrier gas flowrates (Ti-Nb and Al) and the scanning speed (0.5-2.5 m/min). The results reported here are on the laser power of 1.5 kW, 2 mm spot size and 0.5 m/min laser scanning speed.

The produced $\alpha_2$-$Ti_3Al$-Nb samples were cut and prepared for metallographic observations. The cut samples were then heat-treated at different heating profiles (800-1400°C) in order to determine the protection effects of Nb on the $\alpha_2$-$Ti_3Al$ phase. Most importantly, heat treatment was done so that the produced clads can be homogenised and detail their hardness at every heating temperature. STF 16/180 Carbolite Furnace (serial number: 20-900816) with maximum temperature of 1600°C manufactured by Parsons Lane, England was used for the heat treatment process. Heat treatment was carried out under Ar controlled environment at a constant heating rate of 20.0 K/min from 300-1400°C. The heating profile was such that the samples will be heated to the set temperature and at every interval a holding period of 2 hours will be implemented before cooling to room temperature. The As-produced and heat treated samples were prepared for metallographic observations and micro-hardness characterisation. Kroll's reagent was used for etching. The microstructures and elemental analyses were conducted using Joel JSM-6010PLUS/LA Scanning Electron Microscope (SEM) equipped with the Energy Dispersive X-ray Spectroscopy (EDS). The SEM-EDS used Intouch Scope software for analyses. The Matsuzawa Seiko Vickers model MHT-1 was used for the micro-hardness analyses conducted at indent-load of 500 grams and 10 seconds dwelling time. The results are reported and discussed next.

## Results and discussion

The microstructure of the laser alloyed TiAl-Nb coating is presented in Figure 1.

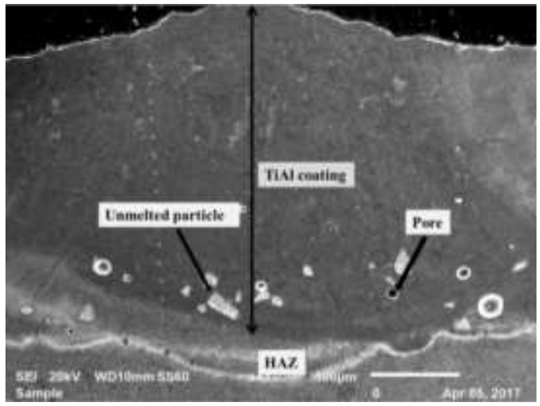


Figure 1: The As-produced TiAl-Nb coating.

The as produced TiAl-Nb coating, shown in Figure 1, reveals that the coating had few unmelted particles closer to the heat affected zone or the interface, and had pores that result after the trapped bubble escape after laser melting. The observed pores are traditional to laser cladding process as they have been observed before by current authors. The observed particles were neither irregular nor spherical. These were Nb particles. Intuitively, these particles look like they are secondary particles which fell onto the clad after solidification hence they did not melt to form part of the produced coating structure. Importantly, this coating looked bound to the base metal, homogeneous, continuous and no significant cracking. This is an indication that the laser alloying process was achieved. The higher magnification images of the produced coatings are presented in Figure 2.

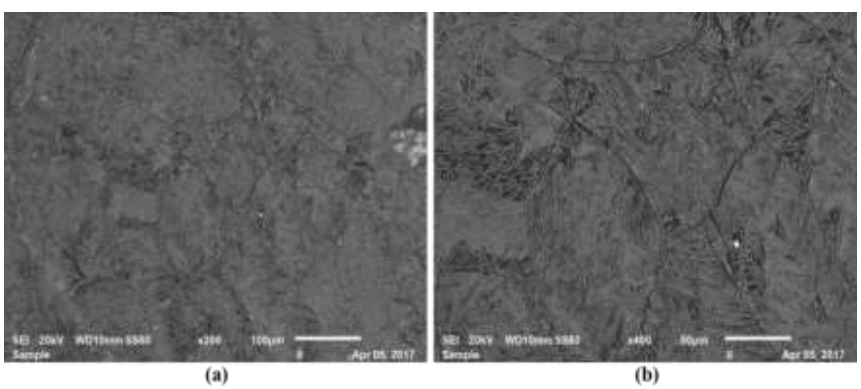


Figure 2: High magnification images of the as-produced TiAl-Nb coating.

Figure 2 shows the highly resolved images of the as-produced TiAl-Nb coating. The resulting microstructure seems to be of the $\alpha_2$-$Ti_3Al$ phase. The EDS analyses indicated that this alloy contained 32 % Al, 60 % Ti and 8 % Nb (at. %). According to the binary phase diagrams, a titanium aluminide alloy with Al content of about 22-39 %, Al (at. %) is a $\alpha_2$-$Ti_3Al$ alloy.

Such a microstructure consists of or can be identified by hexagonal structure (DO19 structure). Figure 2(a) clearly defines this as the $\alpha_2$-$Ti_3Al$ structure while Figure 2(b) shows the lamella within the identified grains. These observations concluding that in deed the $\alpha_2$-$Ti_3Al$ structure was formed at the selected laser *in-situ* metals alloying process. The need to study the $\alpha_2$-$Ti_3Al$ alloyed with Nb is in the promotion of their room temperature ductility. Germann et al [5] reviewed that an alloy with Al in the range 25-26, at. % will be brittle and in effect increasing Al beyond this range will lead to a decrease in room temperature elongation, but an increase in Nb will result in a positive effect on room temperature elongation. Moreover, Ref [5] concluded that Nb will only have a positive effect on the oxidation behaviour of the $\alpha_2$-$Ti_3Al$ structure only when added up to 15 at. %. Yang et al [11] studied the effects of heat treatment and mechanical behaviour on a similar alloy as the one studied herein. They reviewed that $\alpha_2$-$Ti_3Al$ structure will exhibit optimal super-plasticity behaviour at temperature below 1000°C and an optimum super-plasticity deformation at temperatures above 1000°C. Refs [10] and [11] indicated that the general features of super-plasticity include grain boundary sliding among others and concluded that grain grown impact on the structural elongation above 600°C-1000°C. Such a structure will have lower ductility when compare to structure with small grains.

The heat treatment microstructures of the $\alpha_2$-$Ti_3Al$-Nb structure being studied are given in Figure 3. The ternary phase diagram presented in [11] indicated that the structure $\alpha_2$-$Ti_3Al$ structure, by extrapolation, should be stable up to 1200°C when alloyed with Nb.

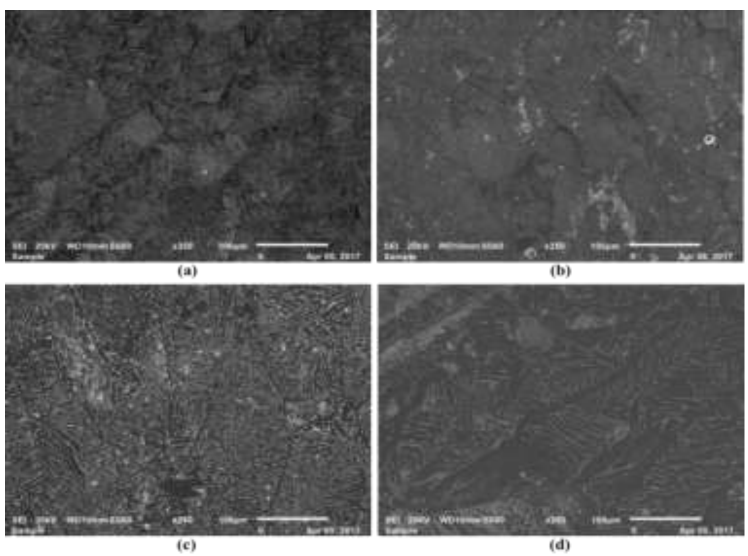

Figure 3: Microstructure of TiAl-Nb coating after heat treatment and oven cooled at: (a) 800, (b) 1000, (C) 1200 and (d) 1400.

The images shown on Figure 3 present the microstructures of the $\alpha_2$-$Ti_3Al$-Nb after heat treatment at different conditions as indicated. It is evident that the lamella of the produced $\alpha_2$-$Ti_3Al$-Nb structure within the observed smaller equiaxed grains at 800°C [11] were thick and clustered (a), became refined at 1000°C (b), fine and well aligned at 1200°C with some diffusion or grain movement being slightly evident and finally at 1400°C the grain boundary sliding is evident with the initially observed equiaxed grains having totally elongated. From these analyses, the observations lead to an inference that this alloy will be ductile and perform well in high temperature until at about 1200°C. The hardness measurement was performed on these coatings. Vickers micro-hardness results indicated that the $HV_{0.5}$ values of the coatings decreased, in a wavy pattern, with the increase in the heat treatment temperature. Three micro-hardness tracks were measured across the sample and the average was determined. From the determined average hardness values, overall hardness (HV) value, for each sample, was calculated and plotted against the corresponding heat treatment temperature (°C). In addition the standard deviation error was obtained. Figure 4 summarises the effects of heat treatment on the resulting microstructure and hardness of the coatings that are studied.

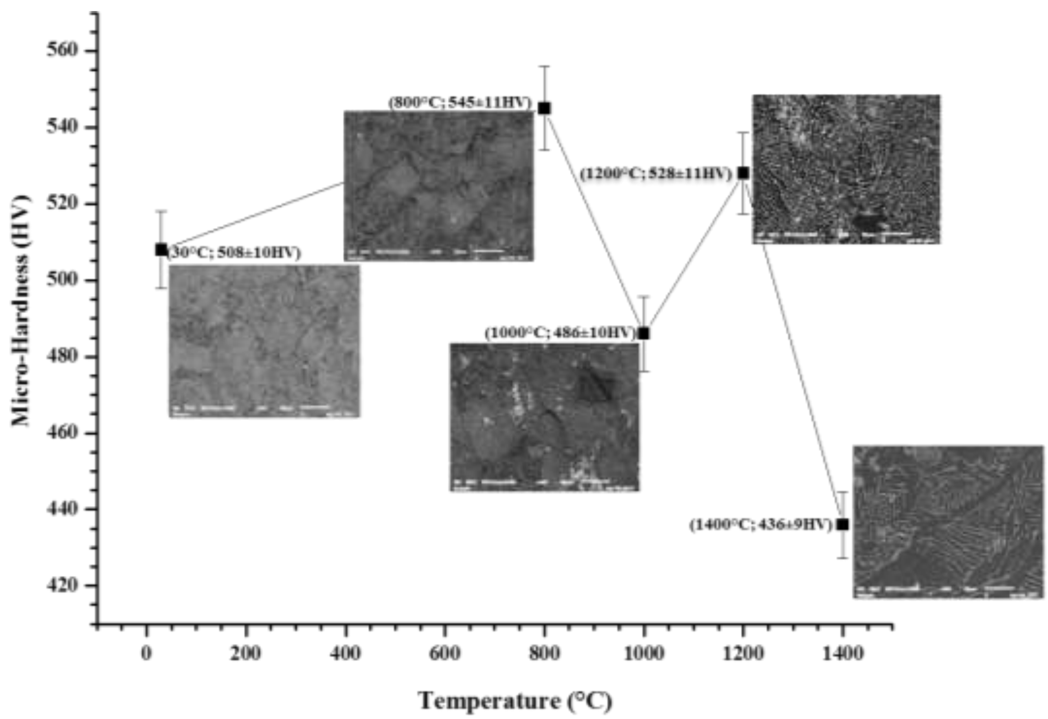


Figure 4: Effects of heat-treatment on microstructure and hardness.

Figure 4 reveals that a hard structure is obtained at temperatures of 800°C and 1200°C. A soft structure is obtained at 1000°C and this is attributable to the retained initial equiaxed grains and refined lamella. Structural weakening, as seen with the hardness of the microstructure at 1400°C, is due to grain and lamella elongation. The latter structure will have lower ductility, if it was to be obtained, when compared to others.

## Conclusion

The study to understand the effects of Nb content on the stability of the ordered $\alpha_2$-Ti3Al alloy post processing with heat treatment was undertaken. The microstructure and hardness measurements were conducted on the As-produced and heat treated samples. The As-produced sample had no cracks, inherent porosity or semi melted particles. TiAl (binary) are crack sensitive due to the lack of ductility at processing conditions. This observation make it known that Nb serves as heat sink metal in the ordered $\alpha_2$-Ti3Al-Nb alloy. The microstructures showed a well refined and ordered $\alpha_2$-Ti3Al structure at the temperature range of 1000-1200°C. Structural grain-coarsening, elongation and possible slip movement was observed at 1400°C. These results led to the following remarks:

- The $\alpha_2$-Ti33Al-8Nb, at% has an order structure with equiaxed grains.
- This structure is stable up to 1200°C.
- There formed lamella within the observed grains were coarse in temperatures between 800-1000°C, became finer at 1200°C and then elongated at 1400°C.
- The observed grains became elongated, coarsened and grain boundary slips were observed at temperatures above 1200°C.
- The hardness values increased with the ordering and refinement of the $\alpha_2$-Ti3Al structure. Structural deformation observed at 1400°C led to a soft structure being produced.
- Overall, this alloy might be having good room temperature and high temperature properties.

## Future work

In the future we wish to extend towards studying dual phase alloys of the same phase using the same technique and 3D printing systems present at our laboratories.

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## References

[1] E. Illeková et al., “Kinetics of intermetallics phase formation in the Ti/Al multilayers,” *Thermochimica Acta*. 469 (2008) 77-85.

[2] N. Kosova et al., “The preparation of the Ti-Al alloys based on intermetallic phases, *IOP Conference Series: Materials Science Engineering*, 112 (2016), 1-5.

[3] E. Hamzah, M. Kanniah, M. Harun, “Microstructural characterization of as-cast binary gamma titanium aluminides,” *Jurnal. Teknologi*. 43 (2005), 113-124.

[4] M. Tlotleng, B. Masina, S. Pityana, “Characteristics of Laser *in-situ* Alloyed Titanium Aluminides Coatings,” *Procedia Manufacturing*, 7 (2017),” 39-45.

[5] R.M. Imayev, V.M. Imayev, M. Oehring, F. Appel, “Alloy design concepts for refined gamma titanium aluminides based alloys,” *Intermetallics*, 15 (2007),” 451-460.

[6] L. Germann, D. Banerjee, J.Y. Guédou, J.-L. Strudel, “Effect of composition on the mechanical properties of newly developed Ti2AlNb-based titanium aliminide,” *Intermetallics*, 13 (2005),” 920-924.

[7] F. Appeal, M. Oehring, J.D>H. Paul, “A novel in situ composite structure in TiAl alloys,” *Materials Science Engineering A*, 493 (2008),” 232-236.

[8] L. Song, X. Xu, L. You, Y. Liang, Y. Wang, J. Lin, “Ordered $\alpha_2$ to $\omega_0$ phase transformations in high Nb-containing TiAl alloys,” *Acta Materialia*, 91 (2015),” 330-339.

[9] A.K. Gogia, T.K. Nandy, D. Banerjee, T. Carisey, J.L. Strudel, J.M. Franchet, “Microstructure and mechanical properties of orthorhombic alloys in the Ti-Al-Nb system,” *Intermetallics*, 6 (1998),” 741-748.

[10] R.M. Imayev, G.A. Salishchev, O.N. Senkov, V.M. Imayev, M.R. Shagiev, N.K. Gabdullin, A.V. Kuznetsov, F.H. Froes,” *Materials Science Engineering A*, 300 (2001),” 263-277.

[11] K.L. Yang, J.C. Huang, Y.N. Wang, “Phase transformation in the β phase of super α2 Ti3Al base alloys during static annealing and superplastic deformation at 700-1000°C,” *Acta Materialia*, 51 (2003), 2577-2594.

[12] A.G. Paradkar et al., “Various stress-strain curve of Ti-Al-Nb alloys undergoing SIMT,” *Materials Science Engineering A*, 456 (1-2) (2007), 292-299.

[13] Y. Ma, D. Cuiuri, N. Hoye, H. Li, Z. Pan, “ Characterization of *In-Situ* Alloyed and Additively Manufactured Titanium Aluminides,” *Metallurgical and Materials Transactions B*, 45B (2014), 2299-2303.