IN-SITU ALLOYED LENS ADDITIVELY MANUFACTURED TiAl-Nb STRUCTURE

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ABSTRACT

Titanium aluminides (TiAl) are interesting intermetallic materials to study due to their enhanced high temperature and light weight properties. They are necessary as structural materials, but brittle to form with conventional methods. Hence big corporates are interested in developments that will lead to cost effective manufacturing technologies that are able to produce homogenous, defect free TiAl structures. Additive manufacturing is one promising technology hence it was explored here in studying process development of producing TNB alloy using laser *in-situ* alloying approach. The produced alloy, γ-TNB, appeared to be homogeneous after heat-treatment and had hardness of 576HV.

1. INTRODUCTION

Titanium alloys are necessary as aerospace structure materials. They are light weight, have improved corrosion and wear, and creep properties. The use of titanium aluminides (TiAl) as high temperature materials is becoming a reality now since their early research and development. Their light weight and mechanical properties at elevated temperatures make them attractive as high temperature structured materials necessary for the aerospace and automotive industries [1-2]. Binary TiAl structures lack ductility at room temperature hence they are impossible to form into structures by means of conventional technologies such as casting and forging. To improve on their ductility, research and development make use of ternary and quaternary alloys amongst other, e.g. GKSS and GE Alloys, in the manufacturing of durable and weight reduced structures [3]. To manufacture such high value components casting is still found to produce heterogonous microstructures hence for effective production cast components are homogenous via Hot Isostatic Pressing [4-7]. Meanwhile additive manufacturing technologies like Electron Beam Melting (EBM) and powder beds seem to be achieving desirable TiAl products with correct specifications [5]. Dilip et al [8] used binder jetting followed reactive sintering treatment to manufacture TiAl structure. Other technologies like selective electron beam melting [5], spark plasma sintering [9], and Laser metal deposition [10] have been used to advance the manufacturing technologies in the production of TiAl. Mallikarjuna et al [11] used the Laser Engineered Net Shaping technique to remelt the EMB produced TiAl sample looking at the effects of laser melt pool.

It is obvious that significant progress is made in the undertaking of manufacturing defect free TiAl structures. While advanced powder metallurgy and AM technologies are able to achieve somewhat desired results, they have a huge cost relating to the production of TiAl structures. Moreover, production still dependent on the preparation and production of master alloy powder, separately, before manufacturing can occur. It is therefore our take that the overall process to market could be expensive and prohibited by those who manufacture this master or pre-alloyed powders. In realising these shortcomings and concerns, The Additive Manufacturing Research Group in the Laser Enable Manufacturing Division of the NLC at the CSIR, Pretoria are looking to circumvent the dependence on those who make pre-alloy master powders by manufacturing TiAl structures, using the LENS Platform, from elemental powders. The approach they call "laser *in-situ* additive manufacturing alloying" was used in this research. This approach is still at its infant stage.

The phenomena in the proposed approach is that the laser created melt pool will be able to convectionally mix the elemental powders into a TiAl alloy when correct thermodynamics are met. It is premised by Wu and Hu [12] that the inability for cast TiAl components to be timely heated and cooled have presented the difficulties especially in the production of a finer TiAl microstructure. The fast heating and cooling achieved by cyclic heat treatments is apparent in the refinement of the microstructure being investigated but on the technological view point it seems such post steps limit cast production of TiAl structures [12]. It is in this understanding and the exiting knowledge, of the authors on laser-materials processing, that this paper present the production of TiAl by using the LENS. This paper reports and discusses a γ-TNB alloy that was produced using the proposed laser *in-situ* additive manufacturing alloying approach. The produced TNB alloy was studied for the microstructure using light optical and scanning electron microscopes. The composition was studied with SEM-EDS and XRD while overall hardness of the produced alloy was measured using the Vickers micro-harness machines.

2. METHODOLOGY

2.1. Methodology

2.1.1 Process set-up and materials

LENS manufacturing process set-up and materials used to manufacture the TiAl-Nb samples is presented by Figure 1. The Optomec, 1 kW laser power, Platform was used to produce the TNB alloy studied in this paper. The processed powders were pure aluminium and a master alloy consisting of Grade 1, commercially pure titanium and niobium. Argon gas was used as the carrier gas and for purging oxygen off the processing chamber during manufacturing. For processing, effects of laser power (W), carrier gas (l/min), powder flow-rates and composition were investigated. The results reported here are from the optimised process parameters when the laser power output was 400 W.

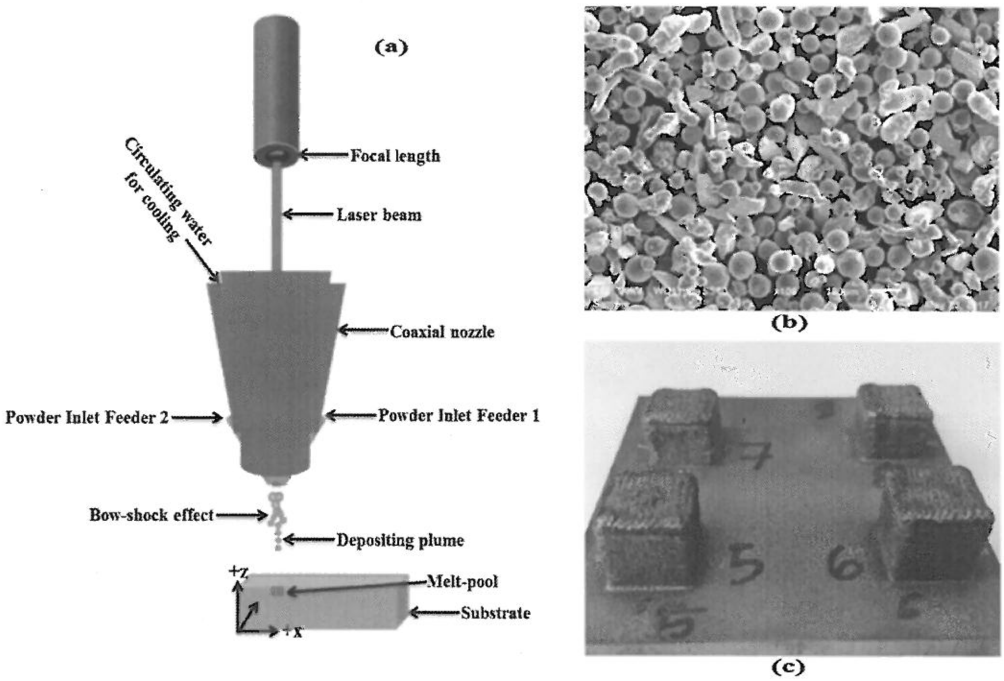

Figure 1: Process set-up (a) and materials (b) used to produce the TiAl-Nb samples (c)

2.2. Characterisation

LENS manufactured TiAl-Nb samples were sectioned along the transverse direction across the clad layer for phase and microstructure analyses. The samples where then mounted and polished to a 0.04 micron (OP-S suspension) surface finish using a Struers TegrsForce-5 auto/manual polisher. After polishing the samples were etched with Keller's reagent and then analysed for microstructures. The manufactured samples were characterised for appearance and macrostructure using Olympus optical light microscope and microstructure using the Joel JSM-6010PLUS/LA SEM that is equipped with the energy-dispersive X-ray spectroscopy (EDS). Composition and phase identification was obtained with the SEM-EDS and Panalytical Empyrean X-ray Diffraction system that is equipped with a Cu kα X-ray source. The produced samples were heat treated at 1400°C under Argon environment. Zwick/Roell Indetec (ZHVμ) was used for measuring the micro-hardness of the produced alloy.

3. RESULTS

The reserach work being reported here sought to investigate the possibility of producing a homogeous microstrture of the ternary TiAl-Nb alloy by a process of laser *in-situ* alloying using the additive manufacturing platfrom called 850-R Optomec LENS that uses the 1kW IPG Fibre laser. The process development results are reported here.

3.1. Microstructures

The sample as produced is presented in Figure 2.

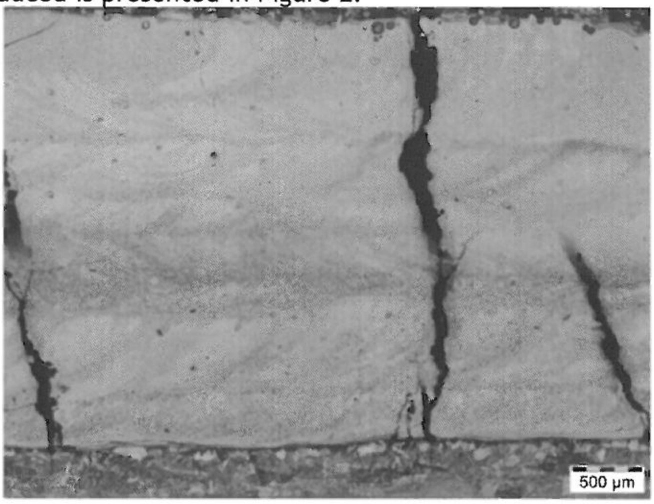

Figure 2: The As-produced TiAl-Nb sample

Figure 2 illustrate the macrostructure of the samples after laser *in-situ* manufacturing. The samples were produced with surface and deep cracks. TiAl materials are naturally brittle and therefore are typically produced with cracks. Refs [10 & 11] have reported on how these cracks can be mitigated during laser manufacturing of Ti-47/48Al-2Cr-2Nb. They concluded that by optimising the process these cracks can be circumvented. The current authors are looking at the optimisation process of these cracks which include, in addition to metal alloy process, using pre-placed heating stage which would cause a retained heat during processing therefore reducing the rapid cooling involved with the laser manufacturing process. The high magnification of the produced sample taken on the sample, excluding the crack, is shown in Figure 3.

Figure 3 shows the macro- and micro-images of powder (a) and TNB alloy that was produced (b) and its heat treatment images (c and d). The sample is characterised of micro pores and unidentifiable structure before heat treatment (b). Post heat-treatment the macrostructure of the alloy could be identified (c and d). The obvious $\gamma+\alpha$ grain with lamella inside were obvious at higher magnification (d). It would seem that the grains are small which could mean the chosen process conditions and the heat treatment done at 1400°C led to a refined macrostructure. The structure looks similar to published microstructures of the y-TiAl cast and EBM manufactured [1-2, 7 & 12-14]. Most importantly it is similar to that of the Ti-46Al-9Nb alloy that is reported by Clemens et al [2] at the GKSS Research Centre, except for the grain sizes s theirs was coarser.

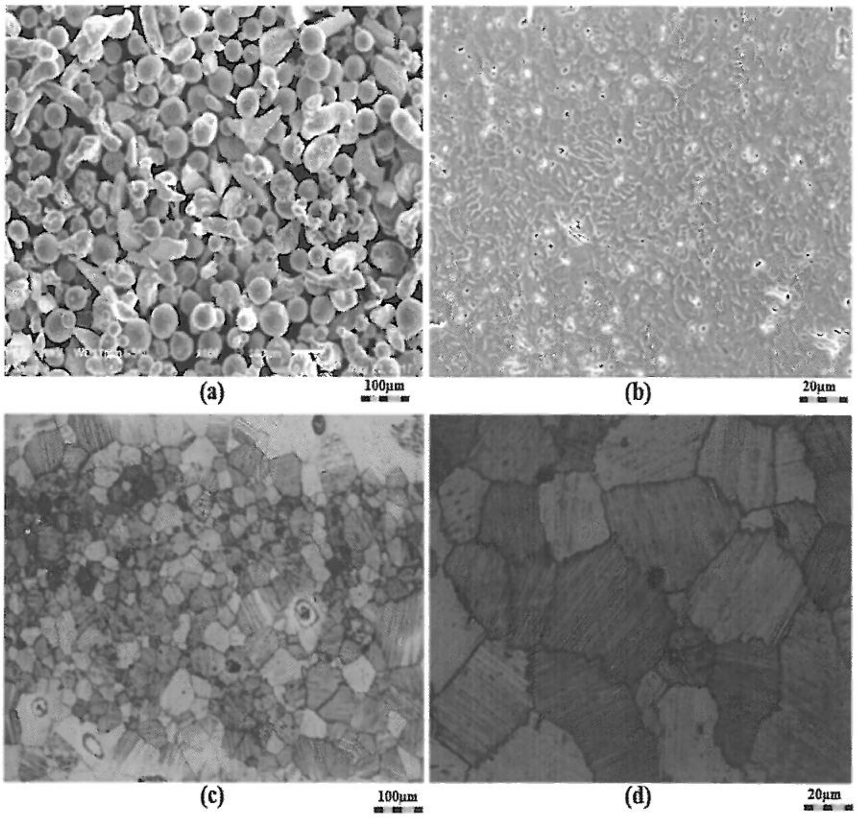

Figure 3: LENS Synthesised γ-TNB alloy (a) TiAlNb powder, (b) As-produced, (c) Heat-treated, and its (d) High magnification

3.2. Composition and phase identification

3.2.1. Composition (SEM-EDS)

The composition of the powder before manufacturing and the corresponding TiAl-Nb built at 1.69 g/min are reported in Table 1.

Table 1 : Composition identification with the EDS (Atomic, %)

Identity	Ti	Al	Nb
Powder	59.66	27.87	12.47
As-produced	38.01	52.33	9.66
Heat-treated (1400°C)	37.76	52.34	9.90

Table 1 reports the EDS composition of the powder and the sample before and after heat treatment. The composition before and after heat treatment are similar and indicated that this alloy composed of 52Al, 38Ti and 10Nb (at, %) making it an early gamma γ-TNB alloy as shown in the phase diagram in section 3.2.2.

3.2.2. Phase Identification (XRD)

The diffraction peaks were obtained by accelerating the voltage of 45kV and current of 40mA. The step size of 0.02 degrees was used. The diffraction pattern of this alloy is given in Figure 4.

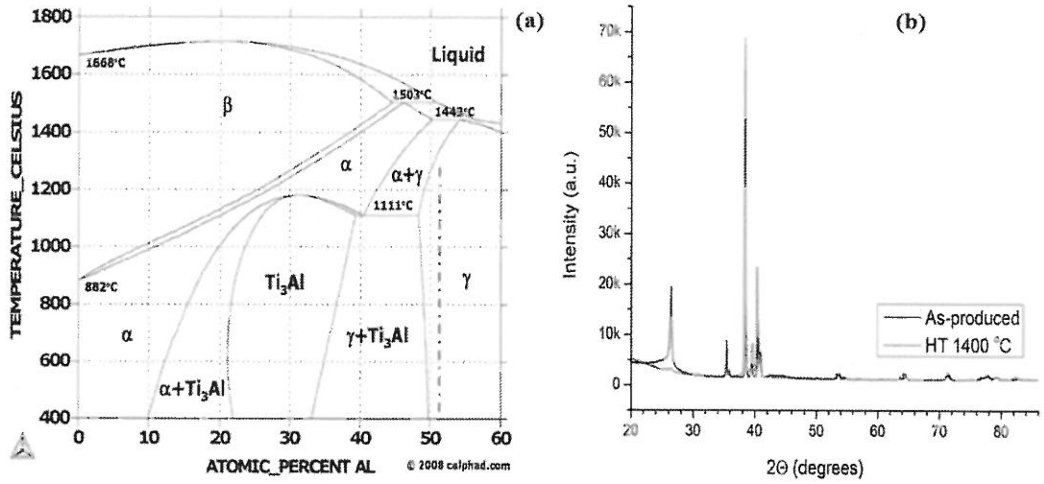

Figure 4: The phase diagram identifying our produced alloy (a) and its diffractogram (b)

According to the phase diagram (a) the as produced structure was of the early γ-TNB alloy (red line) which is an appropriate composition for structural engineering. The phase composition was identifies as shown (b). Figure 4b compares As-produced alloy to its heat treated. The peaks were identified as 26.5° (α_2-Nb), 35.5° (α_2-Nb), 38.5° (γ-Nb); the major peak, 39.0° (γ-Nb), and 40.5° (α_2-Nb). To study these peaks feather they were zoomed into as shown in Figure 5.

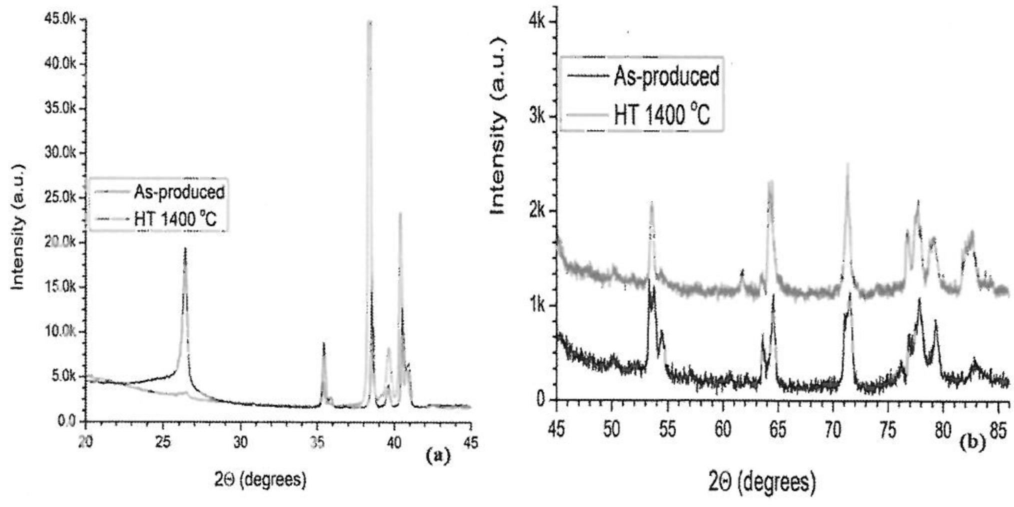

Figure 5: Resolved diffraction peaks of the TiAl-Nb alloy

Typical gamma TiAl spectrum was achieved before and after heat treatment. After heat treatment, Figure 5a, the (γ) major peak's intensity increased when compared to the As-produced sample. The α_2-Nb peak at 2θ value of 26.5° deconvoluted with heat treatment into doublets (26°and 26.5°) and had reduced in intensity. These doublets are of same broad intensity as the original peak and did not move to lower or increased 2θ values. This then suggesting that this deconvoluted α_2-Nb peak is stable in the temperature range chosen for heat treatment. The α_2 peak at 35.5°reduced in intensity to an extended that the initially observed shoulder almost disappeared. Contrary to this, at 40.5° an increase in peak intensity was observed with a slight shift to the lower theta values. Figure 5b shows peak refinement only no obvious change in intensity, broadening could be identified. In general then it would seem that heat treatment at 1400°C led to the precipitation of the α_2 which became intense and were without shoulders as was before heat treatment. Moreover, heat treatment led to the resolution of the entangled peak into easily identifiable α_2 twins while intensifying of the initially observed γ characteristic peak of the alloy. These diffraction spectra show conclusively that a reaction between Ti (40.153°), Nb (38.610°) and Al (38.473°) to form various phase of TiAlNb with γ-TiAl (38.5°) being the most intense peak and termed the alloy characteristic peak. No other phases were identified: β-TiAl/Ti_3Al_5 (39.069°), $TiAl_3$ (39.255°) and $TiAl_2$ (38.904°). These results are similar to those presented in Ref [7].

3.3. Micro-Hardness

Figure 6 presents the micro-hardness profile of the produced sample before and after heat treatment.

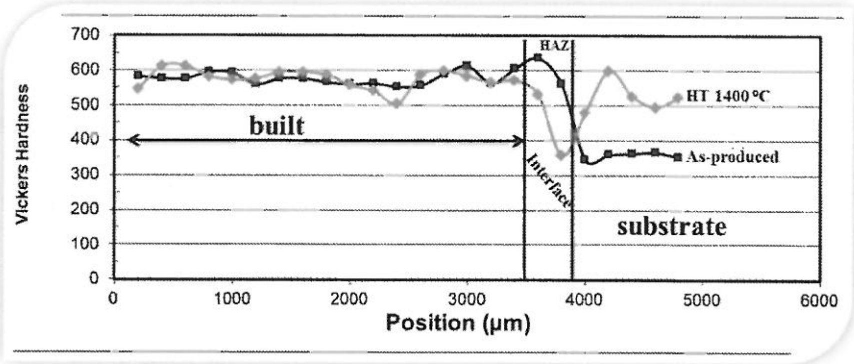

Figure 6: Overall hardness profile of the sample

Figure 6 report on the Vickers' micro-hardness of the produced sample before and after heat treatment, as indicated on the figure. The hardness profile reveals that the produced γ-TNB alloy had similar hardness before and after heat treatment. The overall hardness value ($HV_{0.5}$) was 577 and 576 for the As-produced and heat treated sample, respectively.

4. CONCLUSION

We have successfully studied the laser *in-situ* alloying technique in the quest to producing TiAl alloy(s). The γ-TNB alloy was produced in this regard using the laser *in-situ* additive manufacturing approach. The studied sample had cracks, surface pores and unidentifiable microstructure before heat treatment. Post heat treatment the microstructure, now revealing micron-grains with aligned lamellar inside, was found to be homogenous, refined and had no pores. Heat treatment led to the precipitation and deconvolution of the α_2 phase and intensified the γ phase which was said to be characteristic phase peak of the alloy. The sample had same hardness before and after heat treatment. These results are promising and dictate that a functional structure, e.g. micro-gas turbine be built and tested for thermo-mechanical performance.

REFERENCES

[1] **Imaev, R.M., Imaev, V.M. and Khismatullin, T.G.** 2006. Refining of the microstructure of cast intermetallic alloy Ti-43% Al-X (Nb, Mo, B) with the help of heat treatment, *Metal Science and Heat Treatment*, 48(1-2), pp 81-84.

[2] **Clemens, A., Bartels, A., Bystrzanowski, S., Chladil, H., Leitner, H., Dehm, G., Gerling, R., and Schimansky, F.P.** 2006. Grain refinement in γ-TiAl based alloys by solid state phase transformations, *Intermetallics*, 14, pp 1380-1385.

[3] **Nochovnaya, N.A., Panin, P.V., Kochetkov, A.S., and Bokov, K.A.** 2014. Modern Refractory alloys based on titanium gamma-aluminide: Prospects of Development and Application, *Metal Science and Heat Treatment,* 56(8), pp 23-27.

[4] **Gerling, R.** Clemens, H. and Schimansky, F.P. 2004. Power metallurgy processing of intermetallic gamma titanium aluminides, *Advanced Engineering Materials*, 6(1-2), pp 23-38.

[5] **Tang, H.P.**, Yang, G.Y., Jia, W.P., He, W.W., Lu, S.L. and Qian, M. 2015. Additive manufacturing of high niobium-containing titanium aluminide alloy by selective electron beam melting, *Materials Science & Engineering A*, 636, pp 103-107.

[6] **Gerling, R.**, Bartels, A., Clemens, H., Kestler, H. and Schimansky, F.P. 2004. Structural characterization and tensile properties of a high niobium containing gamma TiAl sheet obtained by powder metallurgical processing, *Intermetallics,* 12, pp 275-280.

[7] **Wang,** Y.H., Lin, J.P., He, Y.H., Lu, X., Wang, Y.L. and Chen, G.L. 2008. Microstructure and mechanical properties of high Nb containing TiAl alloys by reactive hot pressing, *Journal of Alloys and Compounds*, 461, pp 367-372.

[8] Dilip, J.J.S., Miyanaji, H., Lassell, A., Starr, T.L. and Stucker, B. 2017. A novel method to fabricate TiAl intermetallic alloy 3D parts using additive manufacturing, *Defence Technology,* 13, pp 72-76.

[9] Couret, A., Molenat, G., Galy, J. and Thomas, M. 2008. Microstructure and mechanical properties of TiAl alloys consolidated by spark plasma sintering, *Intermetallics*, 16, pp 1134-1141.

[10] Thomas, M., Malot, T. and Aubry, P. 2017. Laser metal deposition of the intermetallic TiAl allo, *Metallurgical and Materials Transactions A*, 48A, pp 3143-3157.

[11] Mallikarjuna, P.K., Balla, V.K., Das, M. Bontha, S. 2016. Understanding thermal behavior in laser processing of titanium aluminides alloys, Proceedings of 6th International & 27th India Manufacturing Technology, Design and Research Conference, pp 73-77.

[12] Wu, X. and Hu, D. 2005. Microstructural refinement in cast TiAl alloys by solid state transformation, *Scripta Materialia,* 25, pp 731-734.

[13] Nansaarng, S. and Srichandr, P. 2012. Microstructure and transformation characteristics of a TiAlNb alloy with Cr and Mo addition, *Materials Research*, 15(1), pp 63-69.

[14] Imayev, R.M., Imayev, V.M., Oehring, M. and Appel, F. 2007. Alloy design concepts for refined gamma titanium aluminide based alloys, *Intermetallics*, 15, pp 451-460.