ADDITIVE MANUFACTURING: CHARACTERIZATION OF TI-6AI-4V ALLOY INTENDED FOR BIOMEDICAL APPLICATION

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Abstract

Direct Metal Laser Sintering (DMLS) is one of the new Laser Additive Manufacturing (LAM) techniques used for producing complex topology components mostly found in medical applications. The work presented in this paper focuses on metallographic analyses of laser sintered Ti-6Al-4V samples. The samples were built by DMLS process from EOSINT Ti-6Al-4V powder. They were then heat treated at temperatures of 1000 and 1100°C and subsequently either cooled with the furnace or water quenched. Slow cooling of Ti-6Al-4V samples from 1000 and 1100^{o}C resulted in a microstructure constituted more by the alpha phase of lower hardness than the laser-sintered material. High hardness was obtained by water quenching. The water quenched evidenced martensitic transformation and high hardness when compared to furnace cooled samples.

Background

Ti-6Al-4V has found application as a bio-alloy and is widely used as an implant material due to its corrosion resistance and high strength to weight ratio [1]. The alloy has a two-phase (α-β) microstructure [2]. Titanium (Ti) undergoes α to β-transformation. Aluminium (Al~ 6 at. %) and vanadium (V~4 at. %) stabilise the alpha and the β-phases, respectively [2]. These phases can exist as lamellar, equi-axed or bimodal see Figure 1 below [3]. These microstructures have a strong influence on the mechanical behavior of the alloy.

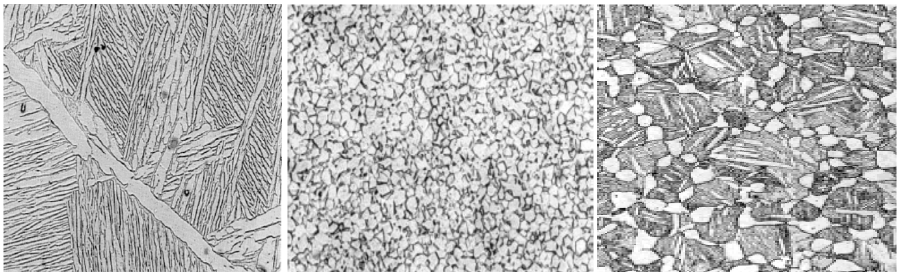

(a) (b) (c)

Figure 1: Microstructures of Ti-6Al-4V (a): an example of a lamellar structure; (b) an example of equi-axed structure; (c) an example of a bimodal structure [3].

Ti6Al4V components may be produced by a variety of methods such as casting [3], or sintering of powders among others [1]. There in turn, is a variety of powder methods such as injection moulding, hot and cold compaction and additive or direct laser sintering. Direct Metal Laser Sintering (DMLS) is one of the latest additive technologies used in manufacturing implants. This technology is capable of producing intricate customised biomaterial implants parts [1]. DMLS works by sintering very fine layers of metal powder layer by layer from the bottom up into a three dimensional component. Figure 2 below shows the schematic diagram of the DMLS process.

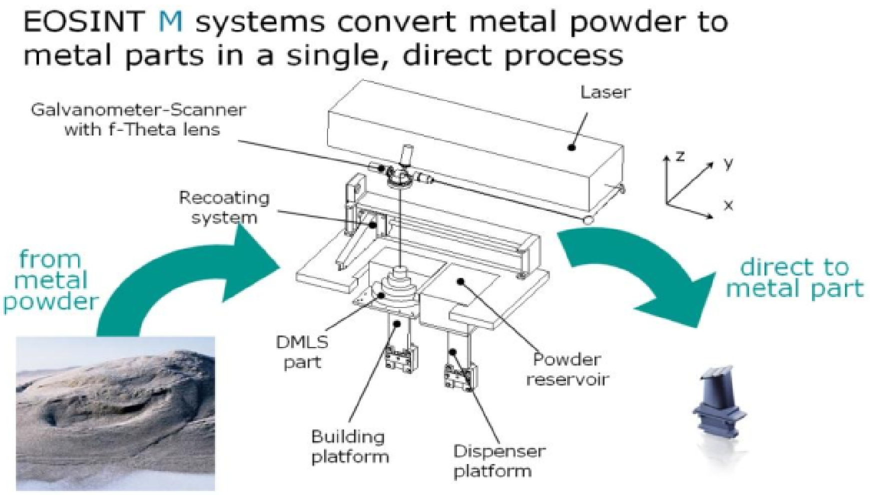


Figure 2: Schematic Diagram Showing DLMS Process on EOSINT M270 Machine [Courtesy to EOS GmbH]

The microstructure of Ti-6Al-4V alloy is influenced by the manufacturing process and /or any post-manufacturing process such as heat treatment. Figure 3 below illustrates pseudo-binary phase diagram of Ti-6Al-4V alloy at 4% per weight of vanadium together with a section on heating temperatures and cooling rates which results in different microstructures as shown.

Direct Metal Laser Sintering (DMLS) is capable of producing customised biomaterial implants parts using EOSINT M270 machine. The main purpose of this work to evaluate the microstructural characteristics of a direct laser sintered Ti-6Al-4V alloy after various heat treatments. An understanding of the relationship of microstructures and hardness is of high importance since it is a fundamental mechanical property of the material. Hardness test in most cases is regarded as a non-destructive test that often indicates tensile and wear properties of a material [6]. It is expected that the results will aid in creating phenomenological model that relates microstructure and mechanical properties in order to tailor-make, inspect, predict deformation and/or failure of the components build at Centre for Rapid Prototyping and Manufacturing (CRPM).

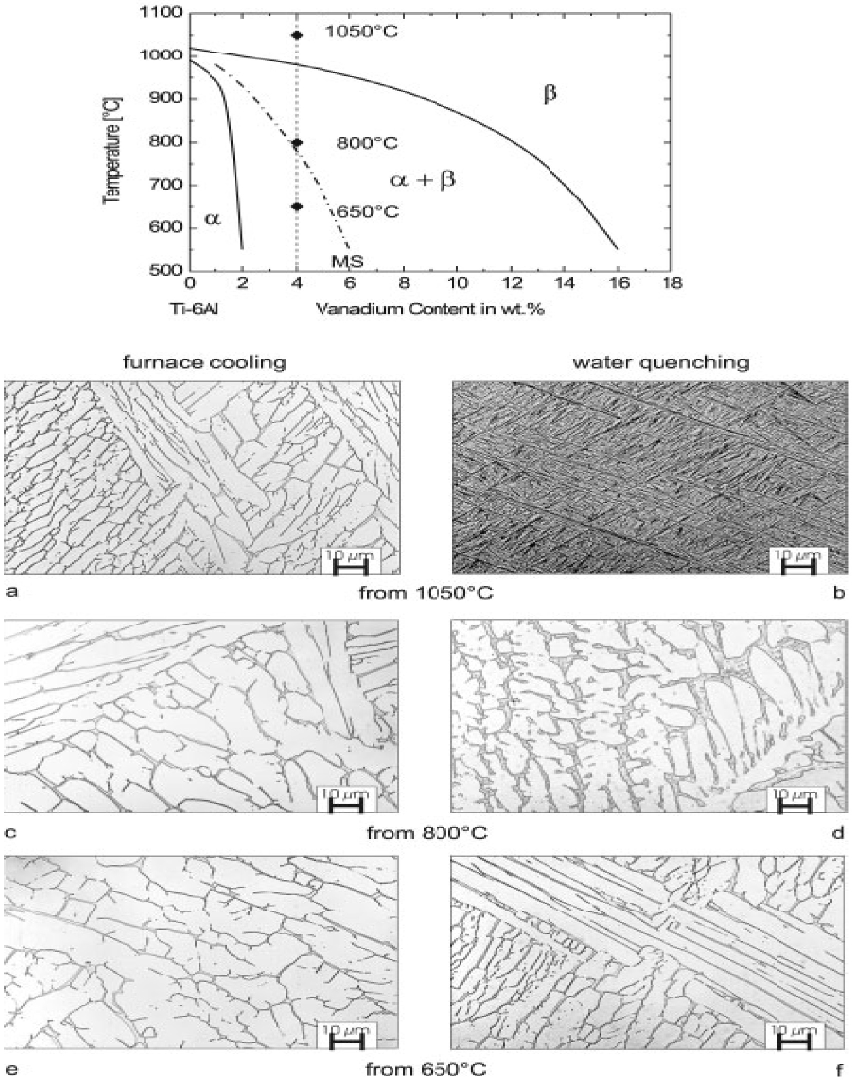


Figure 3: Schematic ternary phase diagram Ti-6Al-V (MS: martensite start temperature); microstructure of Ti-6Al-4V after slow cooling (50ºC/h) and water quenching from 1050ºC, 800ºC, and 650º[3].

Experimental Work

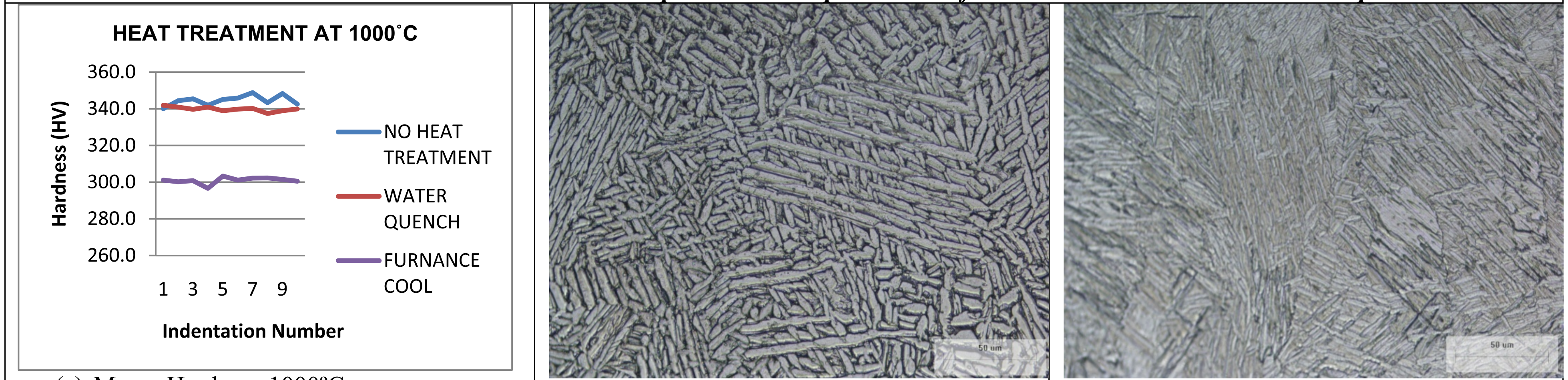
The Ti6Al4V alloy powder used for this work was supplied by EOS GmbH. Samples were manufactured at CRPM in Bloemfontein using the DMLS process on EOSINT M270 machine set at standard building parameters. The samples were built to represent the alloy in order to study the mechanical properties, but no specific implant was produced. Some of the directly sintered samples were heat treated at temperatures of 1000ºC and 1100ºC and others were left untreated. All the heat treated samples were soaked at the set temperature for an hour, after which some were quenched in water and others were slowly cooled with the furnace. Soaking temperatures were selected based on pseudo-binary phase diagram (figure 3) whereby 1000ºC is just above the β-transus temperature while 1100ºC is a high temperature in the β phase-field [2-5]. A carbolite tube furnace was used. The heat treatments were done in an atmosphere with a protective argon gas. Samples were prepared for metallographic analyses using standard methods and etched using Keller's reagent. Metallographic analyses was performed on optical microscope (OM) and scanning electron microscope (SEM) to reveal phases of the microstructure on both the as-sintered and the heat treated samples. Macro hardness measurements were taken using a digital low load tester for Vickers.

Results and Discussions

The photomicrographs of microstructures captured from an OM are shown in figure 4 b, c, e, and f. There is evidence of martensite laths in water-quenched samples as compared to furnace-cooled samples. Slow cooling or equilibrium cooling rates result in the formation of more alpha (α) phase with a lamellae microstructure. This phase dominates as observed in figure 4 (b and e). Fast cooling, as performed by water quenching, results in a martensitic transformation in agreement with Filip et al studies [2]. By furnace cooling, materials follow equilibrium while fast cooling disturbs the thermodynamic path. The fast cooling processes such as quenching in either water or air induces metastable phases with good mechanical properties.

The SEM micrographs in figure 5 a, b, c, d and e show martensitic laths of the Ti-6Al-4V alloy in the as sintered and heat treated states, respectively. It follows that the cooling rate induced during laser sintering promoted the martensitic transformation. This implies that the cooling was fast enough (air quenching) to induce the martensitic transformation. Since the process involves melting of thin layers approximately 10 µm, high cooling rates enables martensitic transformation. Subsequent deposition of layers also provide the stress release on previously deposited layers and this is evidenced by the varying contrast in figure 5 a. Titanium metal show a polymorphic transformation from low temperature alpha to high temperature beta phase. In order to exploit this behavior and to improve the mechanical properties, aluminium (Al) and vanadium (V) are added. It has been found that Al in 6 wt% stabilized the low temperature while 4 wt% V promote the existence of the high temperature beta phase. Among other properties Al increase chances of martensite formation while V is responsible for corrosion resistance and strength. It then follow that for beta alloys, Al amount will be decreased while beta-stabilizing elements such as V, and Nb are added.

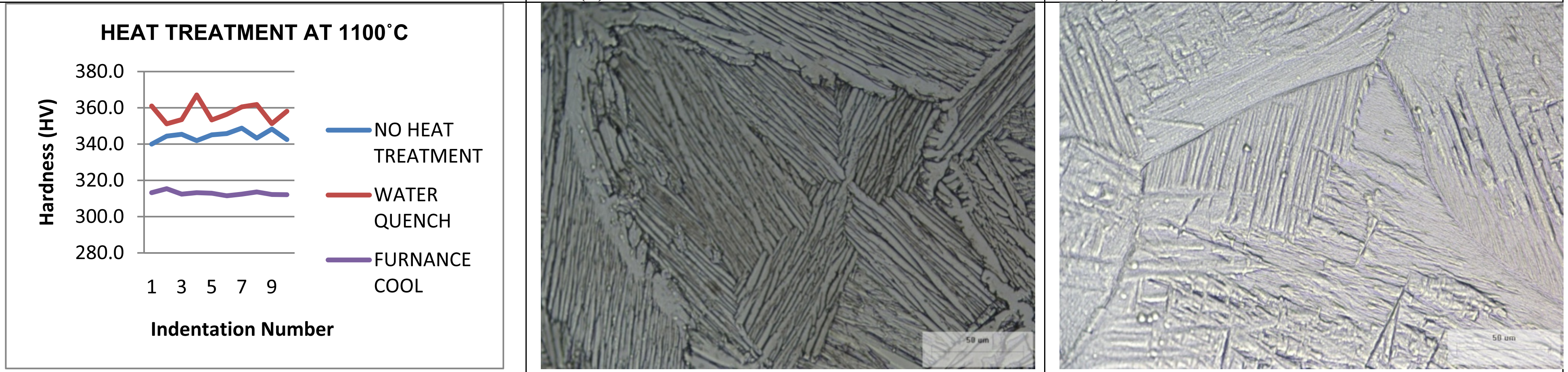
Macro Hardness Results and Optical Microscope Pictures of Direct Laser Sintered Ti-6al-4v Samples



(a) Macro Hardness 1000ºC

(b) HT at 1000ºC/1hr/ FC

(c) HT at 1000ºC/1hr/ WQ



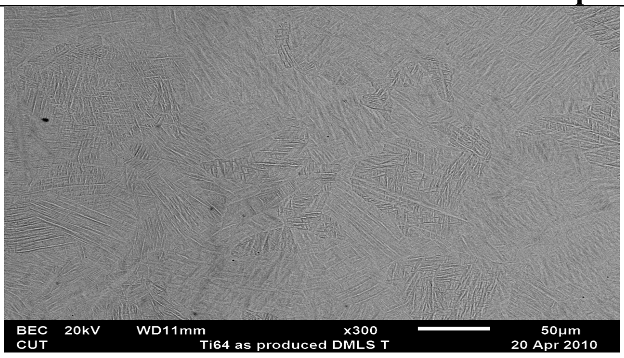
(d) Macro Hardness at 1100ºC

(e) HT at 1100ºC/1hr/ FC

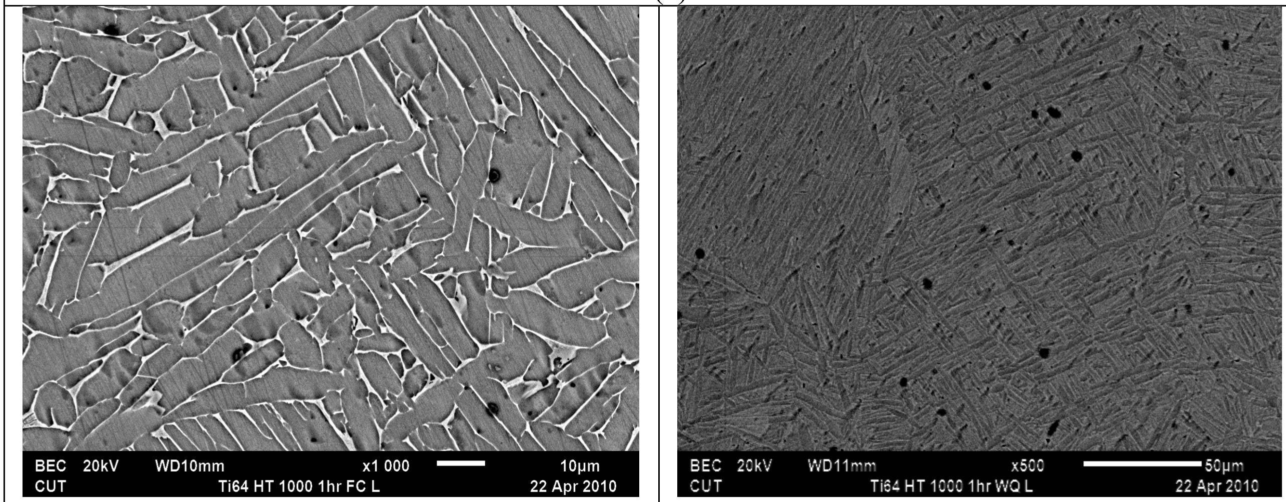
(f) HT at 1100ºC/1hr/ WQ

Figure 4: Optical Microscope of Both Un-Heat Treated and Heat Treated Direct Laser Sintered Ti-6Al-4V Samples

SEM micrograph of Direct Laser Sintered Ti-6Al-4V Samples

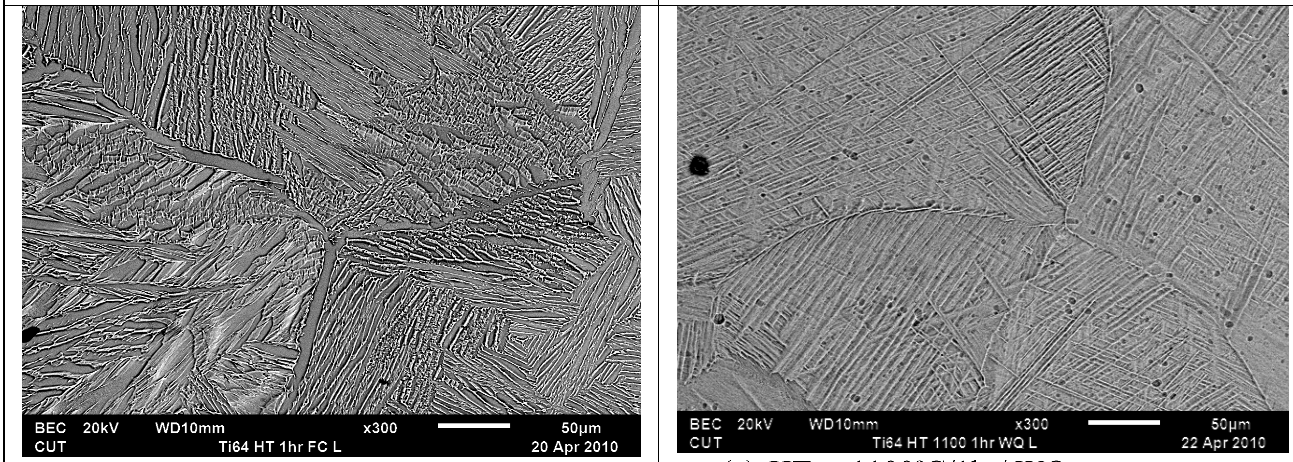


(a) No heat treatment



(b) HT at 1000ºC/1hr/ FC

(c) HT at 1000ºC/1hr/ WQ



(d) HT at 1100ºC/1hr/ FC

(e) HT at 1100ºC/1hr/ WQ

Figure 5: SEM micrograph of both un-heat treated and heat-treated Direct Laser Sintered Ti-6Al-4V Samples

An average hardness of 345 HV_{10} was obtained as laser-sintered was comparable to the hardness of the as-casted Ti-6Al-4V alloy after solution treated at 800ºC/1hr/water quenched as reported in literature [7]. The comparable hardness values suggest that the additive process by direct laser sintering has achieved competitive hardness value both the powder sintering and the sintered part quench-treatment.

Volume fraction of α and β phase can be predicted by the hardness values presented in table 1. It is documented that β possesses high hardness than α-phase [2-5]. Water quenched samples (from 1000 and 1100°C) are harder than furnace cooled, but not entirely surpassing the laser sintered material. Quenching from 1000 and 1100°C above β-transus temperatures resulted in harder alloy with 339 and 357 hardness, respectively, although the former is softer than the latter. The amount of beta phase trapped during quenching increase the hardness. Therefore, the presence of α/β beta lamellae is higher than in furnace-cooled samples. The beta transus temperatature is ~1000°C, hence quenching at this temperature may induce the alpha phase since the sample is slightly exposed to the atmosphere before the operation. It should be noted that on pure Ti the beta transus occur at 882°C indicating that in Ti6Al4V, Al has increased this temperature. At 1100°C, the alloy has completely transformed to beta phase, hence higher residual beta in martesitic structure after quenching. The furnace cooled samples from 1000 and 1100°C have 301 and 312 HV_{10} and are softer than as-sintered samples due to high alpha phase with small amount of intergranular beta. In this instance the transformation β→α follow a thermodynamic route.

Table 1*: The influence of heat treatment on the hardness of the Ti-6Al-4V alloy*

Heat treatment	As-Sintered	1000 ºC/ FC	1000 ºC/ WQ	1100 ºC/ FC	1100 ºC/ WQ
Hardness HV_{10} (average)	345	301	339	312	357

Conclusion

Slow cooling of Ti-6Al-4V samples from 1000ºC and 1100°C resulted in more alpha than beta phases of lower hardness than untreated (as-sintered) material. The highest hardness was obtained by water quenching hence the hardness increases with higher β-phase in the alloy. Quenching from 1000ºC allowed small fraction of alpha phase, probably due to decrease in sample temperature prior to cooling process. The samples quenched from 1100°C illustrated martensitic structure as well as higher hardness than annealed samples.

Further Work

The authors seek to create a phenomenological model that relates to microstructure and mechanical properties in order to tailor-make, inspect, predict deformation and/or failure of Ti6Al4V components built at the CRPM. Therefore other relations between microstructure and mechanical properties such as tensile properties (yield strength, ductility), fracture toughness, low cycles fatigue as well as creep will be established.

Acknowledgements

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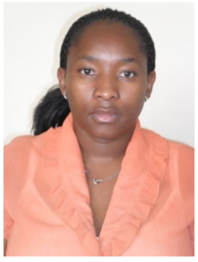
The Author



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I have registered with Engineering Council of South Africa (ECSA) as a candidate engineering technologist. I worked for Product Development Technology Station (PDTS) CUT from 2006 to 2008 then changed to the school of mechanical engineering where I am presently a junior lecturer. I am currently pursuing M-tech in mechanical engineering. My research work focuses on characterization of Ti-6Al-4V components particularly for biomedical application. I specialize on Ti-6Al-4V components that are manufactured by Direct Metal Laser Sintering (DMLS) process on EOSINT M270 machine. The work done so far has been well received and was presented at RAPDASA 2009 and CUT 2010 conferences. I was attached for two weeks at CSIR in the department of materials science and manufacturing under supervision of Dr. Hilda Chikwanda whereby I did a metallographic analysis work.