

# THE EFFECT OF TEMPERATURE ON THE MICROSTRUCTURES OF ADDITIVELY MANUFACTURED Ti6Al4V(ELI)

T.D. MOLOI <sup>1,\*</sup>, T.C. DZOGBEWU <sup>1</sup>, M. MARINGA <sup>1</sup>, AND A.M. MUIRURI <sup>2</sup>

<sup>1</sup> Department of Mechanical and Mechatronics Engineering, Central University of Technology, Free State, Private Bag X20539, Bloemfontein 9300, South Africa. Website: [www.cut.ac.za](http://www.cut.ac.za); Email: [moloitd7@gmail.com](mailto:moloitd7@gmail.com)\*, [dzogbewu@cut.ac.za](mailto:dzogbewu@cut.ac.za), [mmaringa@cut.ac.za](mailto:mmaringa@cut.ac.za)

<sup>2</sup> Department of Mechanical Engineering, Muranga University of Technology, P.O. Box 75-10200, Muranga, Kenya. Website: [www.mut.ac.ke](http://www.mut.ac.ke); E-mail: [amuiruri@mut.ac.ke](mailto:amuiruri@mut.ac.ke)

**Abstract:** The stability of microstructure at high temperatures is necessary for many applications. This paper presents investigations on the effect of changes in temperature on the microstructures of additively manufactured Ti6Al4V(ELI) alloy, as a prelude to high temperature fatigue testing of the material. In the present study, a Direct Metal Laser Sintering (DMLS) EOSINT M290 was used to additively manufacture test samples. Produced samples were stress relieved and half of these were then annealed at high temperatures. The samples were then heated from room temperature to various temperatures, held there for three hours and thereafter, cooled slowly in the air to room temperature. During tensile testing, the specimens were heated up to the intended test temperature and held there for 30 minutes, and then tensile loads applied to the specimens till fracture. Metallographic samples were then prepared for examination of their microstructures both at the fracture surfaces and away from them. The obtained results showed that changes in temperature do have effects on the microstructure and mechanical properties of Ti6Al4V(ELI) alloy. It is concluded in the paper that changes in temperature will affect the fatigue properties of the alloy.

**Keywords:** Effect of temperature, Microstructure, Mechanical properties, Additive manufacturing, Ti6Al4V(ELI), Heat treatment.

## 1. INTRODUCTION

Additive Manufacturing (AM) was established in 1987, as a solution for faster product development. Additive Manufacturing is the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies such as casting, moulding, forming, machining, or joining [1]. For metallic materials, AM is common to use powder feedstocks that are melted successively using a directed energy source such as a laser or electron beam [2]. Subtractive manufacturing methodologies of Ti6Al4V parts often forge or cast material before machining parts to final shapes and dimensions, which unavoidably results in a large amount of material waste and long lead times [3]. Additive manufacturing technologies are used to manufacture Ti6Al4V parts at high build temperatures in the ranges of 1660 to 1670 °C [3, 4]. Such high build temperatures with their rapid cooling rates result in parts with different microstructures and material properties, as compared to conventional manufacturing methodologies [3]. Laser-powder-bed-fusion (LPBF) technologies are widely used to produce titanium alloy parts. These technologies include selective laser melting (SLM), selective laser sintering (SLS), laser metal fusion (LMF), and direct metal laser sintering (DMLS) [5-6]. The DMLS is an additive manufacturing technology that can be used to prototype one-off parts or manufacture low-volume production that runs directly from Computer Aided Drawing (CAD) data [2]. The DMLS technology has the ability to produce fine features and thin walls with good accuracy; works well with a wide variety of elemental metals and alloys and provides excellent resolution of features [2].

During the building process, the printed layers go through repeated melting and solidification. When the printed layers restrain the contraction of the current layer, this results in the creation of residual stresses in fabricated parts. Residual stresses can cause initiation cracks and ultimately reduce the fatigue lives of parts [7]. The post heat treatment process of stress relieving is applied to remove residual stresses present in the DMLS as-built parts [8]. The rapid cooling inherent in DMLS results in the formation of a needle-like microstructure called acicular  $\alpha'$  martensitic microstructure. This microstructure is formed as a result of cooling at rates in the range of  $10^4 - 10^6$  K/s, of the alloy from the high temperatures in the  $\beta$ -field during the DMLS process [9]. The acicular  $\alpha'$  martensitic microstructure is undesirable due to its characteristics of brittleness. High temperature annealing heat treatment is applied to transform this unstable needle-like microstructure to stable mixture of  $\alpha$ - and  $\beta$ -phases. When the temperature is high enough, the thermal energy of atoms increases and causes the atoms to migrate and overcome the surface energy of the grain boundaries. This results in the movement of grain boundaries and the growth of

31 grains. Therefore, higher temperatures imposed on Ti6Al4V(ELI) lead to higher mobility of grains and  
32 the attendant coarsening of  $\alpha$ -laths [10-11].

33 Several industries such as aerospace, biomedical, and automobile, use titanium alloys for many  
34 applications at room and high temperatures. Stability at high temperatures is necessary for many  
35 applications [12-14]. The Ti6Al4V alloy is the most widely studied titanium alloy and is often regarded  
36 as the workhorse of the alloys of titanium. It is a dual-phase ( $\alpha+\beta$ )- titanium alloy with high strength,  
37 low density, high fracture toughness, excellent corrosion resistance and superior biocompatibility [3].  
38 The Ti6Al4V alloy is commonly used at normal room temperature and up to temperatures of 250-400  
39 °C [15]. Zhao *et al.* [15], reported that the application of the alloy at temperatures above 400 °C can  
40 affect its microstructures. To the contrary, Song *et al.* [16], reported that the microstructure of SLM-built  
41 samples has little sensitivity to a temperature below 500 °C and that only above 500 °C did changes  
42 occur. The difference between the two studies is the soaking time, Zhao *et al.* [15], soaked samples for  
43 four hours while Song *et al.* [16], soaked samples for half an hour. Zöllner [10], demonstrated that  
44 variation of temperature has a significant effect on the growth of grains.

45 The Ti6Al4V alloy occurs in the form of various microstructures, namely lamellae, equiaxed,  
46 martensitic, bimodal and Widmanstätten. Lamellae microstructure has good creep and high fatigue crack  
47 growth resistance, while equiaxed has high fatigue crack initiation resistance. The bimodal  
48 microstructure combines both the advantages of lamellar and equiaxed microstructures and possesses an  
49 excellent combination of strength and ductility [17-18]. High cooling rates of the alloy that are greater  
50 than 410 °C/s results in the formation of a complete martensitic microstructure [3]. This microstructure  
51 is characterized by high strength and hardness. The bimodal and equiaxed microstructures are formed  
52 during recrystallization. Lamellae microstructure is formed as a result of the transformation of  $\beta$ -grains  
53 to  $\alpha+\beta$  grains during slow cooling from the high temperature single-phase  $\beta$  region [17, 19]. The alloy's  
54 mechanical properties such as yield strength, ultimate tensile strength, and percentage elongation are  
55 dependent on the microstructure of the alloy [3, 20]. The microstructure of the alloy in turn is dependent  
56 on the properties of the powder used, process parameters and part geometry [21].

57 The mechanical properties of metallics define their response to applied loads. Key amongst these  
58 mechanical properties are strength, ductility, modulus of elasticity, and hardness [22]. Microstructural  
59 features such as grain morphologies and sizes as well as texture do determine the mechanical properties  
60 of metallics [23-24]. The mechanical properties of Ti6Al4V have been noted to be sensitive at all  
61 temperatures [16]. At high temperatures strength, hardness, and modulus of elasticity of the alloy  
62 decrease while ductility increases, whereas at low temperatures strength, hardness, and modulus of  
63 elasticity increase, while ductility decreases [16].

64 Many studies have focused on the effect of heat treatments on the microstructure and tensile  
65 properties and less work has been done on the effect of temperature. This study aims to investigate the  
66 effect of temperature on the microstructure and mechanical properties of the alloy. The novelty arising  
67 from this research lies in the emergence of a definitive statement based on systematic testing, of the  
68 dependence on test temperatures lying between 20 °C and 350 °C, of the microstructure of as-built, stress  
69 relieved, as well as stress relieved then high temperature annealed specimens of Ti6Al4V(ELI)  
70 specimens, for use in elevated temperature fatigue testing.

71

## 72 2. EXPERIMENTAL PROCEDURES

### 73 2.1. Preparation of Samples

74 In this study, a DMLS EOSINT M290 machine was used to manufacture all test samples. The  
75 machine was set to the optimum process parameters shown in Table 1, as recommended by the  
76 manufacturing company EOS GMBH for the production of Ti6Al4V parts.

77 **Table 1.** Process Parameters for Ti6Al4V(ELI) Manufactured By DMLS EOSINT M290

Parameters	Laser power (W)	Hatch spacing ( $\mu\text{m}$ )	Layer thickness ( $\mu\text{m}$ )	Scanning speed (mm/s)	Laser diameter ( $\mu\text{m}$ )
Ti6Al4V(ELI)	280	120	40	1300	80-100

78 Two sets of samples were manufactured. The first set of samples for soaking at different  
79 temperatures and were built as cube blocks with the dimensions of 10 mm. The tensile test samples were

80 manufactured as rods of 53 mm in length and 12 mm in diameter and then further machined to comply  
81 with ASTM E8 standards. Eighteen samples per set were manufactured.

## 82 2.2. Post Heat Treatment Processes

83 Two samples were tested without any heat treatment and are referred to as, as-built samples. The  
84 two sets of samples were stress relieved and half of those were further annealed at a high temperature.  
85 Stress relieve heat treatment was done while the samples were still attached to the building substrate. In  
86 this process, the samples were heated to 600 °C at the rate of 0.08 °C/s, held at this temperature for 3  
87 hours and then furnace-cooled in a vacuum to room temperature. This stress relieving heat treatment  
88 regime was adopted from the works carried out over the past years, that showed it to effectively relieve  
89 residual stress without affecting the microstructure of the as-built part. Half of the stress relieved samples  
90 were then cut off from the building substrate for inspection. The remaining samples were exposed to  
91 high temperature annealing while still attached to the building substrate. In this process, the samples  
92 were heated temperature at the rate of 0.13 °C/s to the beta transus temperature of 980 °C and held at  
93 this temperature for 1 hour; then furnace cooled to 705 °C, held at this temperature for 2 hours; and then  
94 vacuum cooled back to room temperature. The temperature of 980 °C was selected ensure transformation  
95 of the microstructure from the  $\alpha$ - to  $\beta$ -grains and soaking carried out at this temperature to ensure  
96 homogenisation of the resulting microstructure. Furnace cooling at a temperature of 705 °C was carried  
97 out to ensure the transformation of  $\beta$ -grains to  $\alpha$ -grains and their growth in size to enhance ductility of  
98 the alloy.

## 99 2.3. Soaking of Samples at Various Elevated Temperatures

100 The samples that were heat treated were further soaked at these temperatures 133 °C, 241 °C and  
101 349 °C. These soaking temperatures were selected to fall below the maximum normal operating  
102 temperature of 400 °C noted in the literature, below which it has been observed in literature that  
103 temperature has no effect on the microstructure, and therefore, served to test this inference. Two stress  
104 relieved and two high temperature annealed samples were soaked at each temperature for three hours  
105 and then cooled slowly in the air to room temperature. Table 2 presents the DMLS Ti6Al4V(ELI) sample  
106 groups.

107 **Table 2.** DMLS Ti6Al4V(ELI) sample groups

Sample Group	Status/Heat Treatment
A	As-built
B	Stress relieved
C	Stress relieved and then high temperature annealed
D	Sample B soaked at 133 °C
E	Sample C soaked at 133 °C
F	Sample B soaked at 241 °C
G	Sample C soaked at 241 °C
H	Sample B soaked at 349 °C
I	Sample C soaked at 349 °C

## 108 2.4. Metallographic Examination

109 Metallographic examination was done on all the built specimens. To do this, the specimens that were  
110 cut from them were mounted in a mounting cylinder in Multifast resin using a Struers Mounting Press  
111 Citopress machine. Two samples that had undergone similar heat treatment processes were taken for  
112 metallographic analysis. For the soaked samples, the top surface (perpendicular to the build direction)  
113 of one sample and side surface (parallel to the build direction) of the other sample were left free during  
114 mounting for analysis; while for the tensile test samples, the surfaces that were cut along the longitudinal  
115 z-build direction were left free for analysis. The cut surfaces were ground using a 320-grit size silicon  
116 carbide (SiC) grinding paper with water as a lubricant using a Struers tegramin machine. This was  
117 followed by polishing at three stages to obtain a mirror finish surface. Polishing was carried out on  
118 Struers tegramin machine, using a Largo Diapro cloth, with a 9  $\mu$ m diamond suspension for 5 minutes,  
119 then using a Mol Diapro cloth for 2 minutes, and then the final stage of polishing was done using an  
120 MD-Chem cloth with a 4  $\mu$ m suspension of diamonds for 110 seconds. Kroll's reagent (Water 92.82 %, Nitric Acid 6.11 %, and Hydrofluoric Acid 1.07%) was used to etch the samples to reveal their

122 microstructures. Thereafter, a Zeiss Axio A1 optical microscope was used on the etched samples to  
123 examine their microstructures.

## 124 2.5. Tensile Testing

125 Tensile testing was conducted on a Servo Instron 1342, H7051 hydraulics testing machine. Two sets  
126 of specimens were tested at the three different temperatures of 20 °C, 175 °C, and 325 °C. Three  
127 specimens were tested at each temperature. During testing, each specimen was mounted on the testing  
128 machine, heated up to the intended test temperature, and kept at this temperature for 30 minutes,  
129 following which a tensile load was applied on the specimen at a displacement rate of 2 mm/min until  
130 failure and complete separation. The tensile properties investigated here were yield strength, ultimate  
131 tensile strength, percentage elongation, and modulus of elasticity.

## 132 2.6. Vickers Microhardness Testing

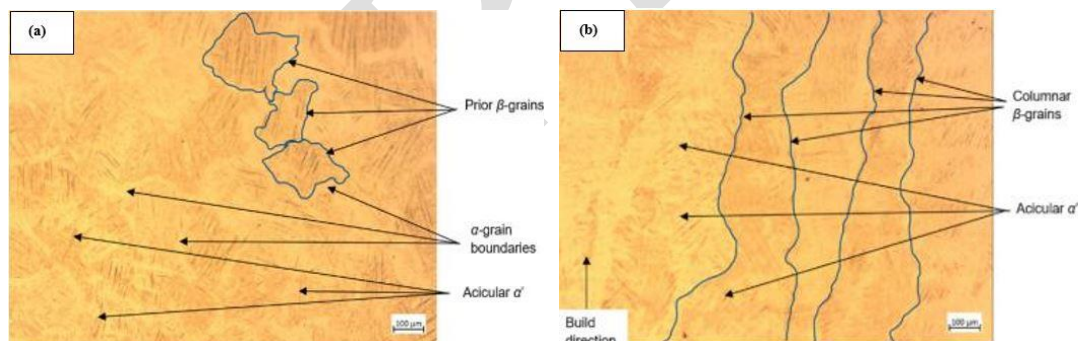
133 After tensile testing, small samples were cut from the uniaxial tensile fractured specimens, and an  
134 electrical discharge machine (EDM) wire cutter was used to cut the specimens, along the longitudinal z-  
135 build direction. The specimens were then cut in a transverse direction 15 mm away from the fracture  
136 surface. Indentations were made on the polished and etched surfaces of the Ti6Al4V(ELI) samples using  
137 the Vickers Future Tech FM 7E microhardness tester. Ten indentations were made on each sample under  
138 the same conditions, with a test load of 2942 mN and a dwell time of ten seconds.

# 140 3. RESULTS AND DISCUSSION

## 141 3.1. Metallographic Examination of Soaked Samples

### 142 3.1.1. The Microstructures of Sample Group A

143 Fig. 1 shows the top and side view optical micrographs of an as-built Ti6Al4V(ELI) sample  
144 produced by DMLS, respectively. It should be noted that the side view and top view are parallel and  
145 orthonormal to the build direction, respectively.



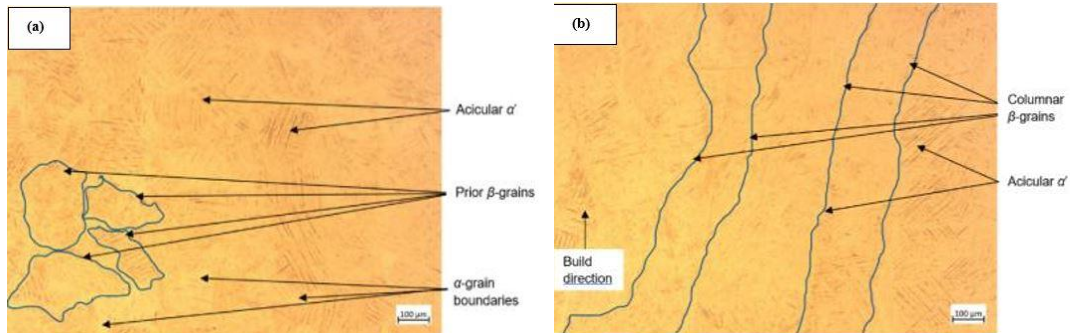
146  
147 **Fig. 1.** Optical micrograph of the (a) top and (b) side view of an as-built DMLS Ti6Al4V(ELI) sample

148 The micrographs in the two figures show different morphologies of prior  $\beta$ -grains. Fig. 1(a), which  
149 is the top view of an as-built sample, shows a lighter shade needle-like  $\alpha'$ -lath referred to as  $\alpha'$  martensite  
150 microstructure. The figure also shows epitaxial (columnar) prior  $\beta$ -grains whose outlines are shown by  
151 blue colour lines on the micrograph and which are separated by  $\alpha$ -grain boundaries. Fig. 1(b) shows the  
152 side view of the sample, consisting of columnar  $\beta$ -grains whose outlines are shown the continuous blue  
153 colour lines superimposed on the micrograph. These grains grow epitaxially perpendicular to the  
154 direction of travel of the laser beam and in the direction of the build [25]. This is mainly due to the  
155 temperature gradient of the molten pool being essentially perpendicular to the scanning direction during  
156 the DMLS process. The average width of the columnar  $\beta$ -grains was measured to be  $136 \pm 16 \mu\text{m}$  with a  
157 coefficient of variance of 11.8 %. Such a high value of the coefficient of variance (COV) implies that  
158 the data was spread out away from the mean value. Inside these columnar  $\beta$ -grains were found  $\alpha'$   
159 martensitic laths with an average width of about  $2.4 \pm 0.4 \mu\text{m}$ , and a COV of 16.7 %. This high COV  
160 implies a large scatter of the data. The acicular  $\alpha'$  martensitic microstructure is formed as a result of  
161 rapid cooling (at rates in the range  $10^4$ - $10^6 \text{ K/s}$ ) of the alloy from the high temperature in the  $\beta$ -field  
162 during the DMLS process [9]. Madikizela *et al.* [26], found  $\alpha'$ -laths with a width of  $0.3$ - $2.4 \mu\text{m}$  for SLM  
163 as-built Ti6Al4V samples, while Agius *et al.* [27], reported that the width of the  $\alpha'$ -laths varies from 1  
164 to 3  $\mu\text{m}$ . Phutela *et al.* [28], found an average columnar  $\beta$ -grains width of 90  $\mu\text{m}$  in SLM as-built Ti6Al4V

165 samples, while Li *et al.* [29], found an average grain diameter of prior  $\beta$ -grains of  $135.5 \mu\text{m}$  on as-  
 166 fabricated Ti6Al4V samples. The differences between these values are likely to be a result of different  
 167 process parameters used during the additive manufacturing process of respective specimens.

### 168 3.1.2. The Microstructure of Sample Group B

169 Fig. 2 shows the top and side view optical micrographs of stress-relieved Ti6Al4V(ELI) samples,  
 170 respectively.

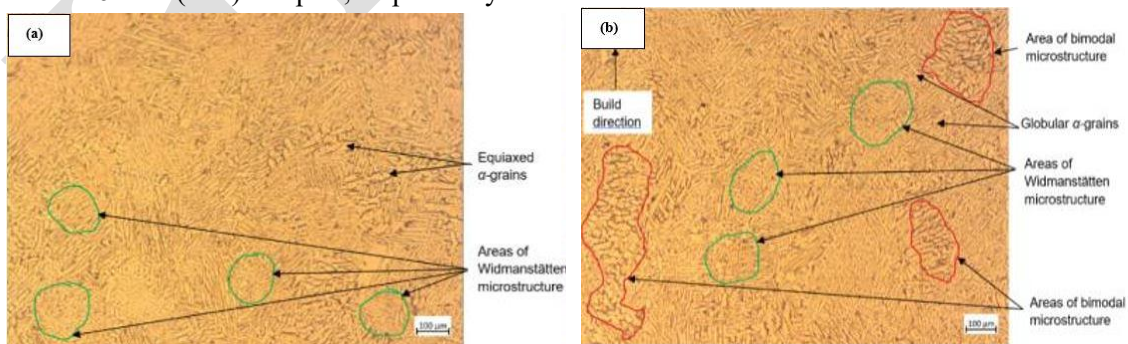


171  
 172 **Fig. 2.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to  
 173 the build direction) of a stress-relieved DMLS Ti6Al4V(ELI) sample

174 The microstructures in Fig. 2(a) and Fig. 2(b) show similar morphologies as those of the as-built  
 175 samples presented in Fig. 1(a) and Fig. 1(b), respectively. In Fig. 2(a), the acicular  $\alpha'$  laths are still visible,  
 176 with an average width of about  $2.6 \pm 0.47 \mu\text{m}$ . The COV for these statistical measures is 18.1 %, which  
 177 implies a large scatter of data. The mean and standard deviations are 0.2 and  $0.07 \mu\text{m}$  higher, respectively,  
 178 than the ones for as-built samples shown in Fig. 2(a), a respective increase of 8 and 17 %. Fig. 2(b)  
 179 shows columnar  $\beta$ -grains which were also evident in the side view of as-built samples. Within these  
 180 columnar  $\beta$ -grains exist acicular  $\alpha'$  laths suggesting that no noticeable microstructure transformation  
 181 took place during stress-relieving heat treatment as observed under an optical microscope. The average  
 182 width of the columnar  $\beta$ -grains in Fig. 2(b) was measured to be  $142 \pm 17 \mu\text{m}$ , giving rise to a COV of 11.9  
 183 %. Jazdzewska *et al.* [30], reported that stress relieving does not affect the mechanical properties of  
 184 titanium alloys such as strength or ductility but rather only reduces residual stresses. This is consistent  
 185 with the observation made here that there was no variation of microstructure with stress relieving heat  
 186 treatment.

### 187 3.1.3. The Microstructure of Sample Group C

188 Fig. 3 shows the top and side view optical micrographs of stress relieved and then high temperature  
 189 annealed Ti6Al4V(ELI) samples, respectively.



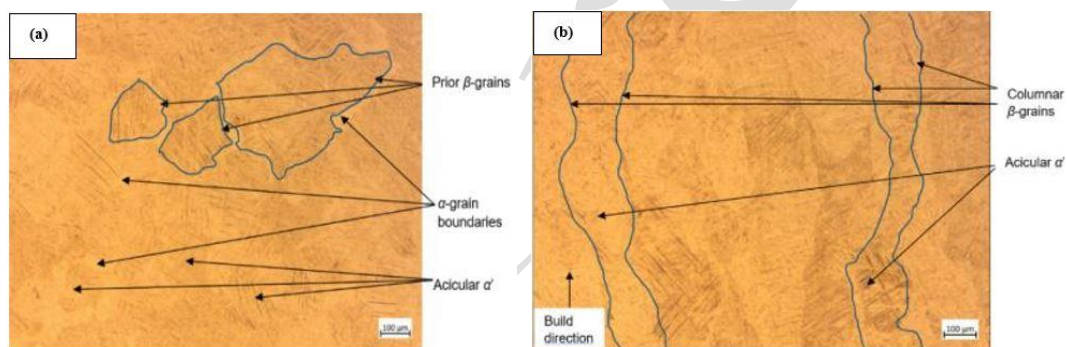
190  
 191 **Fig. 3.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to  
 192 the build direction) of a sample stress relieved and then at high temperature annealed DMLS Ti6Al4V(ELI)  
 193 sample

194 The morphologies of the microstructure in Fig. 3 are completely different from the ones in Fig. 1  
 195 and Fig. 2. This difference in microstructure is due to the differences in the heat treatment processes. In  
 196 Fig. 3(a), acicular  $\alpha'$  needles-like structures no longer exist and are replaced by larger globular  $\alpha$ -laths  
 197 that are longer and wider. When the temperature is high enough, the thermal energy of atoms increases  
 198 and causes the atoms to migrate and overcome the surface energy of the grain boundaries. This results  
 199 in the movement of grain boundaries and the growth of grains. Therefore, higher temperatures imposed

200 on Ti6Al4V(ELI) lead to higher mobility of grains and the attendant coarsening of  $\alpha$ -laths [10-11]. Fig.  
 201 3(a) exhibits a mixture of equiaxed and lamellar  $\alpha$ -laths. Both the micrographs in Fig. 3 consist of  
 202 globular  $\alpha$ -laths and Widmanstätten microstructure, the latter shown inside green colour circles  
 203 boundaries on the micrographs. Fig. 3(b) shows part of the micrographs to exhibit bimodal  
 204 microstructures some of which are shown inside red coloured contours lines. The average width of the  
 205  $\alpha$ -laths is about  $8.2 \pm 1.2 \mu\text{m}$ , and therefore, a COV of 14.6 %. The average width of the  $\beta$ -laths is about  
 206  $1.2 \pm 0.25 \mu\text{m}$ , which gives a COV 20.8 %. Such a COV implies a large scatter of the data. Malefane *et*  
 207 *al.* [31], in their study of the microstructures on DMLS Ti6Al4V high-temperature annealed samples,  
 208 observed equiaxed and basket weave microstructures. It was further reported in their research that the  
 209 average width of the  $\alpha$ -laths built in the y-direction was  $6.7 \mu\text{m}$ . The different result between the present  
 210 study and that of Malefane *et al.* [31], is likely due to differences in the annealing processes used. In  
 211 their study the annealing was done at a temperature of  $950 \text{ }^\circ\text{C}$ , soaking for two hours and cooling at  $0.13$   
 212  $^\circ\text{C/s}$  to room temperature. In the present study annealing was done at  $980 \text{ }^\circ\text{C}$ , soaking was done for one  
 213 hour, followed by very rapid cooling to a temperature of  $705 \text{ }^\circ\text{C}$ , and then soaked for 2 hours followed  
 214 by cooling at  $0.03 \text{ }^\circ\text{C/s}$  to room temperature. The higher initial soaking temperature and slower eventual  
 215 cooling rate are likely causes of the coarser  $\alpha$ -laths obtained in the present study. Zöllner [10], stated that  
 216 higher temperatures lead to even coarser  $\alpha$ -laths. Jovanovic' *et al.* [32], reported that the thickness of the  
 217  $\alpha$ -laths increases as the annealing temperature is increasing. They found that the average thickness of  
 218 the  $\alpha$ -laths at temperatures of  $1100 \text{ }^\circ\text{C}$ ,  $950 \text{ }^\circ\text{C}$  and  $800 \text{ }^\circ\text{C}$  were  $9 \mu\text{m}$ ,  $7 \mu\text{m}$  and  $6 \mu\text{m}$ , respectively.

### 219 3.1.4. The microstructure of Sample Group D

220 Fig. 4 shows the top and side view optical micrograph of stress relieved Ti6Al4V(ELI) samples,  
 221 respectively, that were soaked at a temperature of  $133 \text{ }^\circ\text{C}$  for three hours and allowed to cool slowly in  
 222 the air.

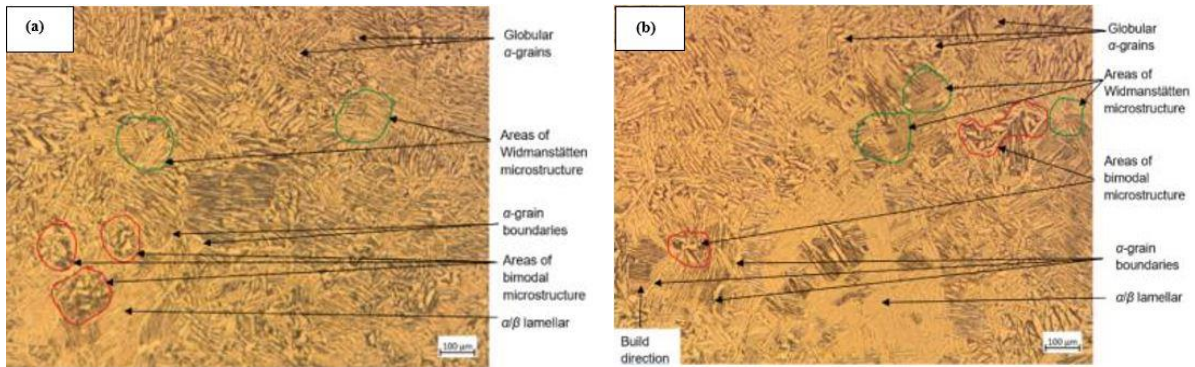


223  
 224 **Fig. 4.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to  
 225 the build direction) of a DMLS Ti6Al4V(ELI) sample stress relieved and then soaked at  $133 \text{ }^\circ\text{C}$  for three hours

226 The morphology of the microstructure in Fig. 4(a) is similar to the one in Fig. 2(a). Fig. 4(a) consists  
 227 of needle-like  $\alpha'$  martensite laths, with an average width of about  $2.64 \pm 0.55 \mu\text{m}$  and therefore, a COV  
 228 of 20.8 %. This high COV implies a wide dispersal of data. The mean standard deviations are 0.24 and  
 229 0.15 higher respectively, than the ones for as-built samples shown in Fig. 2(a), a respective increase of  
 230 10 and 38 %. This implies a modest increase of the size of laths and a significant increase in the scatter  
 231 of data of size between this case and the one for Fig. 2(a). Fig. 4(a) exhibits prior  $\beta$ -grains whose outlines  
 232 are shown in blue colour lines and are separated by  $\alpha$ -grain boundaries. As was the case in Fig. 2(b), the  
 233 columnar  $\beta$ -grains in this figure have within them  $\alpha'$  martensitic laths. The average width of the columnar  
 234  $\beta$ -grains, in this case, is about  $144 \pm 16 \mu\text{m}$ . Giving rise to a COV of 11.1 %. This is a further increase  
 235 above the values of  $142 \pm 17 \mu\text{m}$  and  $136 \pm 16 \mu\text{m}$ , recorded for the as-built and stress relieved specimens,  
 236 respectively. Whilst there is a fair increase in the average width of the epitaxial grains from the as-built  
 237 to stress relieved samples (4 %), the further increase due to soaking at a temperature of  $133 \text{ }^\circ\text{C}$  after  
 238 stress relieving is small (1 %). Clearly, the soaking temperature is too low to have a notable influence  
 239 on the microstructure as was observed in the study done by Zhao *et al.* [15] and Song *et al.* [16].

### 240 3.1.5. The Microstructure of Sample Group E

241 Fig. 5, shows the top and side views optical micrographs respectively, of DMLS Ti6Al4V(ELI)  
 242 samples that were stress relieved and then annealed at high temperature and then further soaked at a  
 243 temperature of  $133 \text{ }^\circ\text{C}$  for three hours, followed by slow cooling in air.



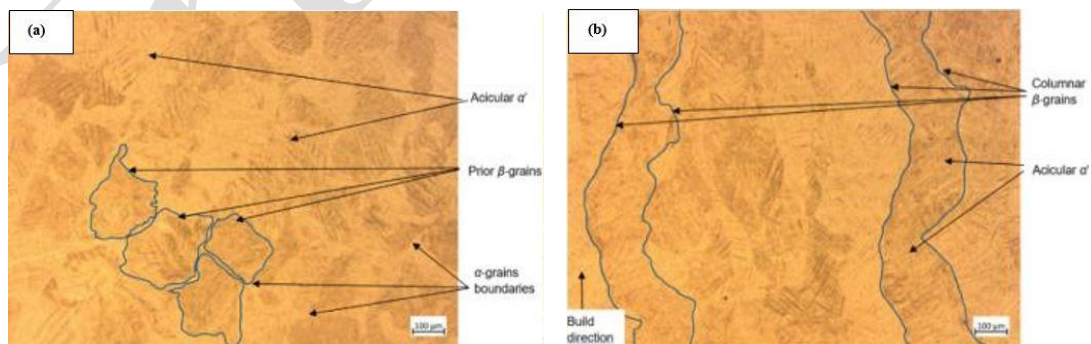
**Fig. 5.** Optical micrograph of the top view (perpendicular to the build direction) of a stress relieved, high temperature annealed and then soaked at 133 °C DMLS Ti6Al4V(ELI) sample

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245  
246

In Fig. 5, the  $\alpha$ - and  $\beta$ -phases are clear, with the  $\alpha$ -laths being brighter and  $\beta$ -laths darker as shown by the arrows in these figures. In both these two figures,  $\alpha$ -grain boundaries, globular  $\alpha$ -laths, Widmanstätten and lamellar as well as bimodal microstructures are present. The Widmanstätten and bimodal microstructures are outlined with green and red colour lines on the micrographs, respectively. It can be noted that the micrograph in Fig. 5(b) is dominated by a Widmanstätten microstructure. This microstructure is known to have low strength and poorer ductility among the different microstructures of Ti6Al4V [33]. Fig. 5(a) and Fig. 5(b) show an average width of  $\alpha$ -laths of  $8.3 \pm 1.4 \mu\text{m}$ , giving rise to a COV of 16.9 %. The average width of  $\alpha$ -laths has increased from 8.2 to 8.3  $\mu\text{m}$  when compared with the value recorded from Fig. 3(b) with only stress relieving followed by high temperature annealing. This is a small negligible increase of 1 % that indicates the effect of soaking at a temperature of 133 °C, on the width of  $\alpha$ -laths is inconsequential. Moreover, the two means fall within the standard deviations of one another. The average width of  $\beta$ -laths was measured at  $1.8 \pm 0.45 \mu\text{m}$ , implying a COV of 25 %. This high percentage implies a large spread of data away from the mean value. It is noted that the mean value of width for  $\beta$ -laths has increased from 1.2 to 1.8  $\mu\text{m}$  when compared with the value obtained from Fig. 3(b). This is a much greater (50 %) than was recorded for the  $\alpha$ -laths and is a sign that the effect of soaking at 133 °C on the growth of  $\beta$ -laths is significant effect of. This increase in the width of  $\beta$ -laths is a sign of a significant increase in the mobility of atoms and is expected to give rise to improved ductility of the alloy [34].

### 265 3.1.6. The Microstructure of Sample Group F

266 Fig. 6, shows the top and side view optical micrographs of stress relieved DMLS Ti6Al4V(ELI)  
267 samples, respectively that were then soaked at a temperature of 241 °C and thereafter cooled slowly in  
268 the air.



**Fig. 6.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to the build direction) of a DMLS Ti6Al4V(ELI) stress relieved sample, soaked at a temperature of 241 °C

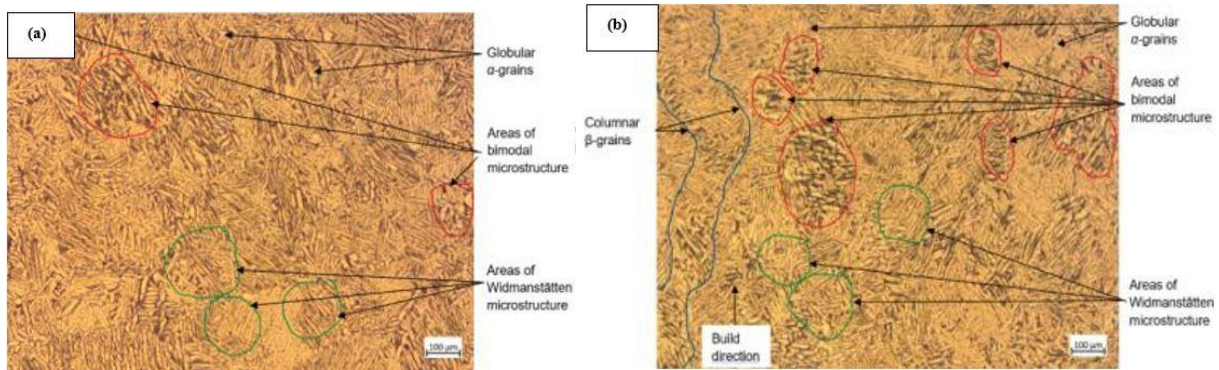
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Prior  $\beta$ -grains are evident on the micrograph in Fig. 6(a), whose outlines are shown by blue colour lines and are separated by  $\alpha$ -grain boundaries on the micrograph. The acicular  $\alpha'$  martensitic microstructure, with an average width of the  $\alpha'$  laths of  $2.9 \pm 0.6 \mu\text{m}$  exists within these grains, which gives a COV of 20.7 %. This high percentage implies a high dispersion away from the mean value. It is noted that average width of the  $\alpha'$  laths has increased from 2.64 to 2.9  $\mu\text{m}$  when compared with the value obtained from Fig. 4(a). This is a small increase of about 1 %, that falls within the standard deviations of both values of mean and therefore, is negligible. Fig. 6(b) shows columnar  $\beta$ -grains whose outlines

279 are shown by continuous blue lines within which exist  $\alpha'$  martensite grains. The average thickness of the  
 280 columnar prior  $\beta$ -grains is about  $147\pm 26 \mu\text{m}$ , giving rise to COV of 17.7 %. This high percentage  
 281 indicates that the data was highly spread out. The morphology in the above figure shows a predominance  
 282 of grains that are a bit darker than the one in Fig. 4(a). This is likely due to the higher soaking  
 283 temperature, which caused an increase in the content of  $\beta$ -laths. Shaikh *et al.* [18], observed an increase  
 284 in the content of  $\beta$ -laths as the soaking temperature increased.

### 285 3.1.7. The Microstructure of Sample Group G

286 Fig. 7, shows the top and side view optical micrographs of stress relieved and high temperature  
 287 annealed Ti6Al4V (ELI) samples, respectively that were further soaked at a temperature of 241 °C for  
 288 three hours and then cooled slowly in the air.

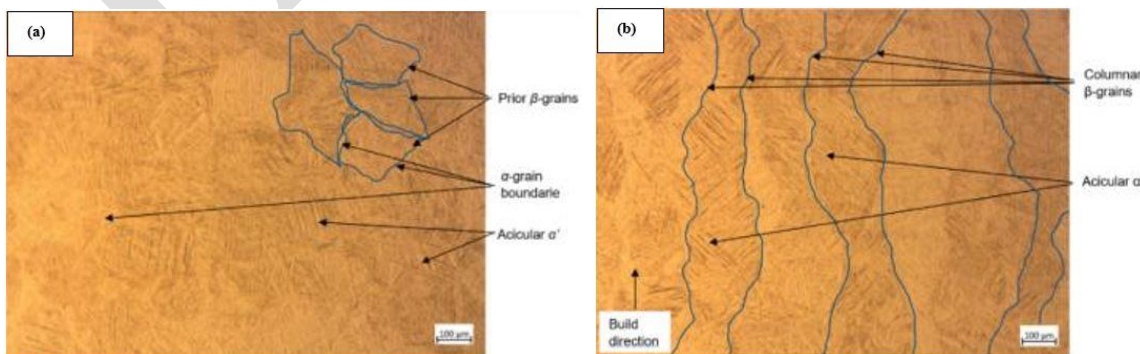


289  
 290 **Fig. 7.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to  
 291 the build direction) of a sample soaked at 241 °C for three hours, after stress relieving followed by high  
 292 temperature annealing

293 Fig. 7 shows the  $\alpha$ -laths being brighter and  $\beta$ -laths darker. Both the two figures show globular  $\alpha$ -  
 294 laths, areas of Widmanstätten and bimodal microstructures. The Widmanstätten and bimodal  
 295 microstructures are circled by green and red colour lines, respectively. A columnar  $\beta$ -grain is visible in  
 296 Fig. 7(b), whose outline is shown by blue lines, within which are colonies of  $\alpha$ - and  $\beta$ -laths. The  
 297 micrograph in Fig. 7(b) has a high incidence of the bimodal microstructure. This microstructure is known  
 298 to possess a higher ductility among the microstructures of Ti6Al4V [35-36]. The average width of the  
 299  $\alpha$ -laths was determined as  $8.3\pm 2.3 \mu\text{m}$ , while the measured average width of the  $\beta$ -laths was  $1.9\pm 0.7 \mu\text{m}$ .  
 300 The coefficient of variation for the width of the  $\alpha$ -laths and  $\beta$ -laths are calculated as 27.7 % and 36.8 %,  
 301 respectively. Such high percentages suggest a high dispersal of data. It is noted that the width of  $\beta$ -laths  
 302 had increased by  $0.1 \mu\text{m}$  from the value of  $1.8 \mu\text{m}$  measured in Fig. 5(b), an increase of 6 %.

### 303 3.1.8. The Microstructure of Sample Group H

304 Fig. 8, shows the top and side view optical micrographs of stress relieved Ti6Al4V(ELI) samples,  
 305 respectively, that were soaked at a temperature of 349 °C for three hours and then slowly cooled in the  
 306 air.



307  
 308 **Fig. 8.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to  
 309 the build direction) of a stress relieved DMLS Ti6Al4V(ELI) sample soaked at a temperature of 349 °C for three  
 310 hours

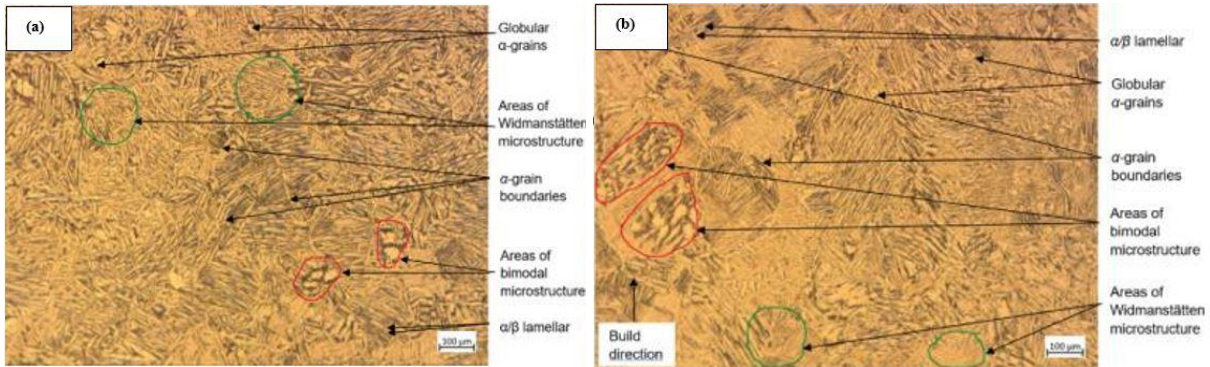
311 Fig. 8 shows similar morphologies of microstructure as observed in Fig. 6, respectively. Fig. 8(a)  
 312 shows prior  $\beta$ -grains, with  $\alpha'$  martensite laths within them. These prior  $\beta$ -grains are separated by  $\alpha$ -grain  
 313 boundaries. The width of the martensitic  $\alpha'$  laths within the prior  $\beta$ -grains in this figure measure to



314  $3.1 \pm 1.2 \mu\text{m}$  in width, giving rise to a very high COV of 38.7 %. In Fig. 8(a), it is noted that the width of  
 315 the  $\alpha'$  laths has increased by  $0.2 \mu\text{m}$  from the value of  $2.9 \mu\text{m}$  measured in Fig. 6(a), an increase of 7 %.  
 316 Agius *et al.* [27], reported that the width of the acicular  $\alpha'$  laths vary from 1 to  $3 \mu\text{m}$  for as-built samples.  
 317 Slightly higher values of the width of the  $\alpha'$  martensitic laths were recorded in the present study, which  
 318 can be ascribed to the higher soaking temperatures in the present work. The columnar prior  $\beta$ -grains in  
 319 Fig. 8(b) measure of approximately  $137 \pm 25 \mu\text{m}$  in the width, giving rise to a COV of 18.2 %. Such a  
 320 high percentages implies a high dispersal of data.

### 321 3.1.9. The Microstructure of Sample Group I

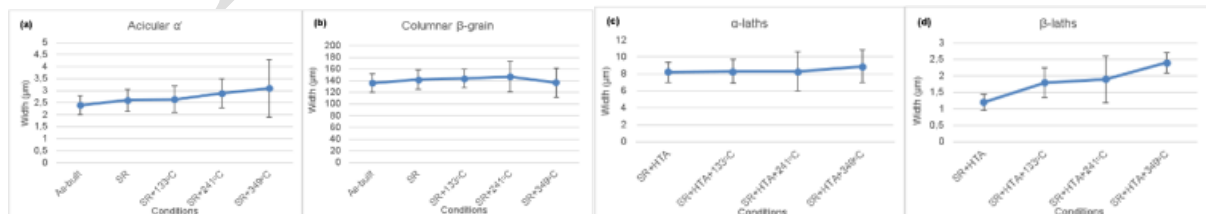
322 Fig. 9, shows the top and side view optical micrographs of stress relieved and high temperature  
 323 annealed Ti6Al4V(ELI) samples, respectively that were soaked at a temperature of  $349 \text{ }^\circ\text{C}$  for three  
 324 hours before being cooled in the air.



325 **Fig. 9.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to  
 326 the build direction) of DMLS Ti6Al4V(ELI) samples that were stress relieved and then annealed at high  
 327 temperature followed by soaking at  $349 \text{ }^\circ\text{C}$  for three hours  
 328

329 After being soaked at  $349 \text{ }^\circ\text{C}$  for three hours, the micrographs in Fig. 9 shows a further increase in  
 330 darker areas compared to Fig. 7. This could be a result of the increase in soaking temperature, which  
 331 from the transformation phase diagram of the alloy implies higher content of the  $\beta$ -laths. The  
 332 micrographs show areas of  $\alpha$ - and  $\beta$ -phases that are stacked parallel to each other forming a lamellar  
 333 microstructure. There are also areas of globular  $\alpha$ -grains and Widmanstätten microstructure whose  
 334 outlines are shown by blue colour circles. The two micrographs have areas of bimodal microstructures  
 335 whose outlines are shown by red colour circles. The bimodal microstructure is known to exhibit a well-  
 336 balanced strength and ductility [37]. The average width of the  $\alpha$ -lath was measured at  $8.9 \pm 1.9 \mu\text{m}$ , giving  
 337 rise to a COV of 21.3 %. The average width of the  $\beta$ -lath measured  $2.4 \pm 0.3 \mu\text{m}$ , giving rise to a COV of  
 338 12.5 %. Such high percentages suggests that the data was spread out. It is noted that the mean width of  
 339 the  $\beta$ -lath increased by  $0.5 \mu\text{m}$  from the value of  $1.9 \mu\text{m}$  measured in Fig. 7(a), an increase of 28 %.  
 340 Shaikh *et al.* [18], observed a similar trend, as the soaking temperature increased. Xing *et al.* [38], stated  
 341 that as the powder bed temperature increases the width of the  $\beta$ -lath increases as well.

342 Fig. 10 summarizes the observation made in Fig. 1 to Fig. 9. The symbol SR and HTA denote stress  
 343 relieved and high temperature annealed, respectively.



344 **Fig. 10.** The average width of the (a) acicular  $\alpha'$  laths, (b) columnar  $\beta$ -grains, (c)  $\alpha$ -laths and (d)  $\beta$ -laths  
 345

346 Fig. 10(a) shows that as the soaking temperature increases from room temperature to a temperature  
 347 of  $349 \text{ }^\circ\text{C}$ , the widths of  $\alpha'$  laths increase as well. Fig. 10(b) shows that as the soaking temperature  
 348 increases from room temperature to a temperature of  $241 \text{ }^\circ\text{C}$ , the width of the columnar prior  $\beta$ -grains  
 349 increases as well. However, at  $349 \text{ }^\circ\text{C}$  the width of the columnar prior  $\beta$ -grains experiences a drop in  
 350 value. Fig. 10(c) and Fig. 10(d) show that as the soaking temperature increases the width of the  $\alpha$ - and

351  $\beta$ -laths increase. This result is similar to the study done by Zöllner [10], who stated that higher  
 352 temperatures lead to even coarser  $\alpha$ -laths. However, the measured values of mean in all but a few cases  
 353 in the above figures fall within the error bars, which implies that the differences observed in the graphs  
 354 are insignificant. It is right to conclude, therefore, that soaking temperatures below 350 °C have little to  
 355 no effect on the microstructure of the Ti6Al4V(ELI) alloy.

356 **3.2. Mechanical Properties**

357 Table 3 shows the obtained results from tensile and hardness testing of stress relieved DMLS  
 358 Ti6Al4V(ELI) specimens tested at different temperatures.

359 **Table 3.** Mechanical Properties of Specimens that were Stress Relieved

Temperature °C	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Vickers Micro-Hardness (HV)	Percentage Elongation (%)
20	1266.7±60.4	1311±52	123.8±5.2	348.6±12.7	5.9±1.6
175	1008.6±85.5	1073.2±66	116.9±0.9	327.3±12.5	7.8±0.4
325	913.8±21.1	993.3±6.2	105.9±3.4	-	10.8±2.9

360  
 361 The data in Table 3 shows that increasing the testing temperature from 20 °C to 175 °C and 325 °C  
 362 led to a reduction of yield strength, ultimate tensile strength, modulus of elasticity, and hardness, as well  
 363 as an increase in ductility.

364 Table 4 shows the obtained results from tensile and hardness testing of stress relieved followed by high  
 365 temperature annealing DMLS Ti6Al4V(ELI) specimens tested at different temperatures.

366 **Table 4.** Mechanical Properties of Specimens that were Stress Relieved Followed by High Temperature  
 367 Annealing

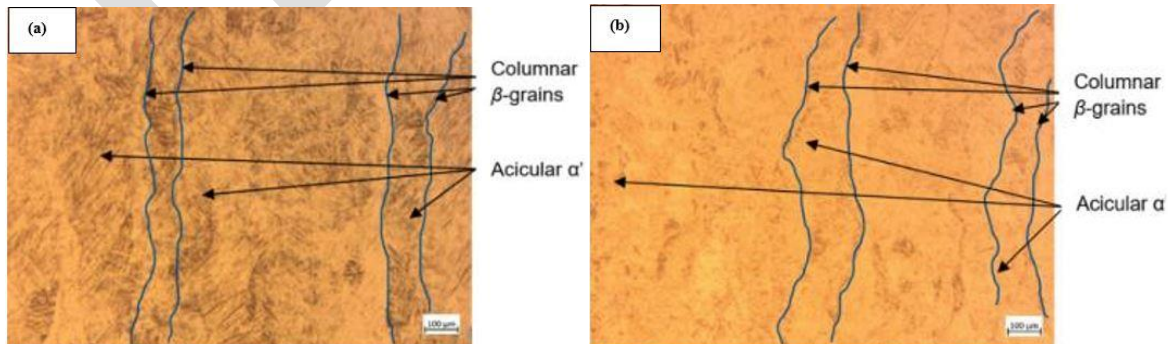
Temperature °C	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Vickers Micro-Hardness (HV)	Percentage Elongation (%)
20	888.4±33.9	953.7±59	99.5±17	312.5±25.3	13.8±2.1
175	597.2±84.3	674.8±83	91.7±13.5	310.4±33.9	13.9±1.6
325	544.3±33.4	654.9±6.7	83.3±1.1	-	17.3±4.6

368  
 369 Similar to the specimens that were stressed relieved only, increasing the testing temperature led to a  
 370 reduction of yield strength, ultimate tensile strength, modulus of elasticity, and hardness, as well as an  
 371 increase of ductility. It is noted that annealing heat treatment led to a reduction of yield strength, ultimate  
 372 tensile strength, modulus of elasticity, and an increase in ductility.

373 **3.3. Metallographic Examination of Tensile Tested Samples**

374 **3.3.1. Microstructures of Samples that were Stress Relieved and then Tested at a Temperature of 20 °C.**

375 Fig.11 show optical micrographs of stress relieved Ti6Al4V(ELI) samples that were tested at a  
 376 temperature of 20 °C



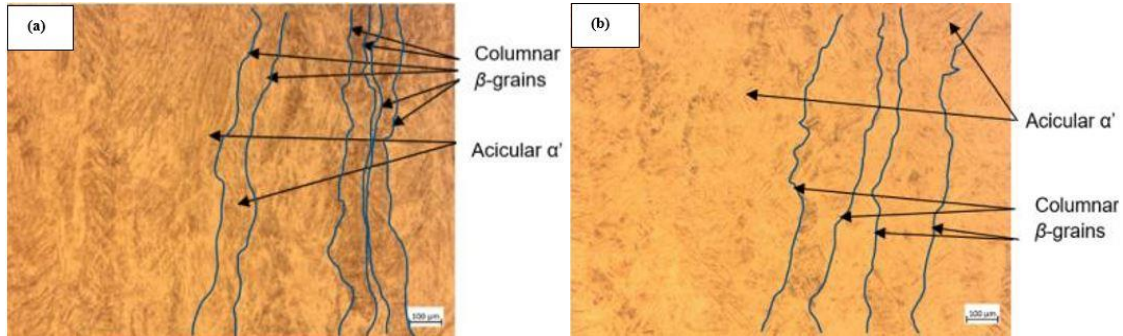
377 **Fig. 11.** Optical micrograph of a stress relieved DMLS Ti6Al4V(ELI) sample and tested at a temperature of 20  
 378 °C (a) near the fracture surface and (b) 15 mm away from the fracture surface

379  
 380 The two figures show a needle-like  $\alpha'$  lath known (dark lines) martensite microstructure, inside  
 381 columnar  $\beta$ -grains. The average widths of the columnar  $\beta$ -grains in Fig. 11(a) and Fig. 11(b) are 135±14  
 382  $\mu\text{m}$  and 136±11  $\mu\text{m}$ , respectively, with coefficients of variance (COVs) of 10.4 % and 8.1 %, respectively.  
 383 The average widths of  $\alpha'$  martensitic laths in Fig. 11(a) and Fig. 11(b) are 3.8±0.3  $\mu\text{m}$  and  
 384 3.7±0.4  $\mu\text{m}$ , respectively, with COVs of 7.9 % and 10.8 %, respectively. It is noted that the COVs are

385 high, which implies that the data was spread out away from the mean value. There are small differences  
 386 in the average values of columnar  $\beta$ -grains and  $\alpha'$  martensitic that fall within the respective standard  
 387 deviations given above for both values of the two means. In Fig. 11(a), the micrograph is darker, and the  
 388 columnar  $\beta$ -grains are more visible than in Fig. 11(b), this is likely to be a result of lateral (transverse)  
 389 strains at the neck.

390 *3.3.2. Microstructures of Specimens that were Stress Relieved and Tested at a Temperature of 175 °C.*

391 Fig. 12 shows optical micrographs of stress relieved Ti6Al4V(ELI) samples that were tested at a  
 392 temperature of 175 °C.



393 **Fig. 12.** Optical micrograph of a stress relieved DMLS Ti6Al4V(ELI) sample and tested at a temperature of 175  
 394 °C (a) near the fracture surface and (b) 15 mm away from the fracture surface

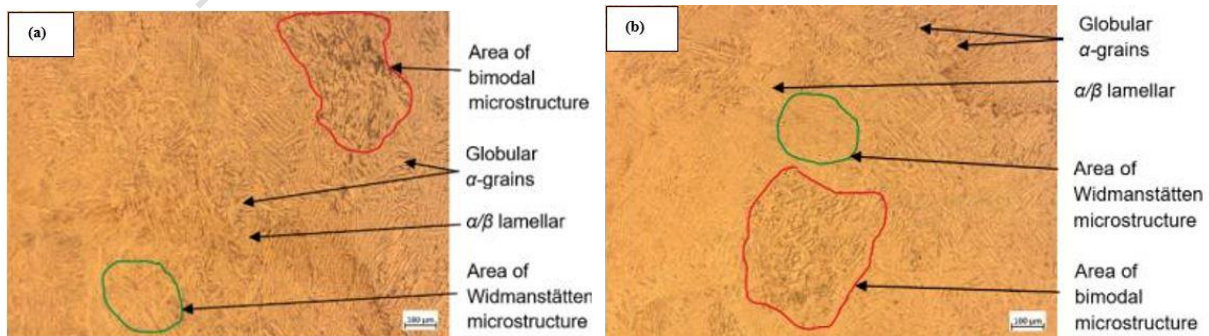
395 The two figures show the presence of columnar  $\beta$ -grains within them  $\alpha'$  lath martensitic  
 396 microstructure. In Fig. 12(a), the average widths of the columnar  $\beta$ -grains and  $\alpha'$  laths are  $146 \pm 13 \mu\text{m}$   
 397 and  $3.8 \pm 0.4 \mu\text{m}$ , respectively, with COVs of 8.9 % and 10.5 %, respectively. In Fig. 12(b), the average  
 398 widths of the columnar  $\beta$ -grains and  $\alpha'$  laths are  $136 \pm 14 \mu\text{m}$  and  $3.7 \pm 0.6 \mu\text{m}$ , respectively, with COVs  
 399 of 10.3 % and 16.2 %, respectively. Similar to Fig. 11(a) and Fig. 11(b), the calculated values of the  
 400 COV are high, which reduces the reliability of the calculated values of mean. It is noted that in Fig.  
 401 12(a), the columnar  $\beta$ -grains are closely packed and narrower than in Fig. 12(b), which is thought to be  
 402 a result of the severity of deformation taking place during necking and the attendant transverse, Poisson's  
 403 ratio contraction.  
 404

405 *3.3.3. Comparing Microstructures of Specimens that were Stress Relieved and then Tested at*  
 406 *Temperatures of 20 °C and 175 °C.*

407 Both Fig. 11(a) and Fig. 11(a) show micrographs that consist of the columnar  $\beta$ -grains and  $\alpha'$  laths.  
 408 However, in Fig. 11(a) the needle-like structures are smaller, more randomly oriented and dense than in  
 409 Fig. 12(a). It is reported that these needle-like structures exhibit high strength and hardness [17, 19].  
 410 This agrees with the finding from the tensile testing conducted here that, as the test temperature was  
 411 increased there was a reduction in the values of yield and ultimate strength as well as hardness.

412 *3.3.4. Microstructures of Specimens that were Stress relieved Followed by High Temperature Annealing*  
 413 *and then Tested at a Temperature of 20 °C.*

414 Fig. 13 shows optical micrographs of stress relieved followed by high temperature annealing  
 415 Ti6Al4V(ELI) samples that were tested at a temperature of 20 °C.

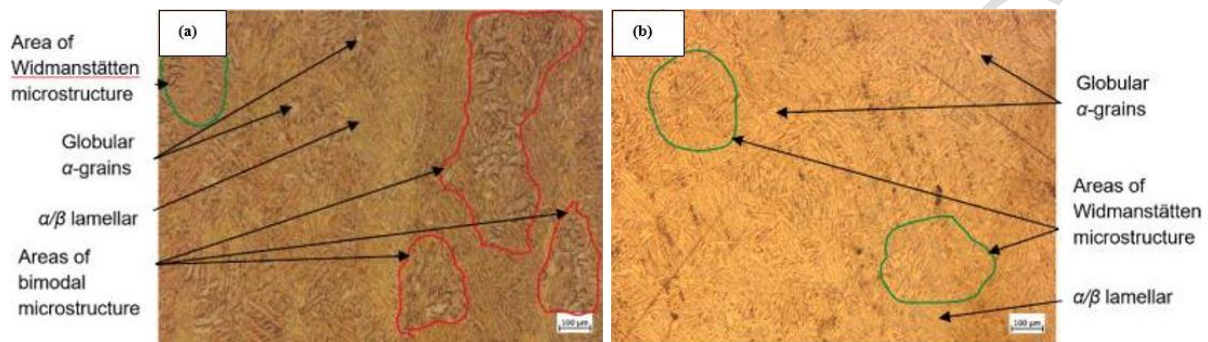


416 **Fig. 13.** Optical micrograph of a stress relieved followed by high temperature annealed DMLS Ti6Al4V(ELI)  
 417 sample and tested at a temperature of 20 °C (a) near the fracture surface (b) 15 mm away from the fracture  
 418 surface  
 419

420 The two figures show similar micrographs with areas having  $\alpha$ - and  $\beta$ -phases that are packed parallel  
 421 to each other forming lamellar microstructures. There are also areas with Widmanstätten and bimodal  
 422 microstructures. In Fig. 13(a), the average widths of the  $\alpha$ -laths and  $\beta$ -laths were measured at  $10.1\pm 0.9$   
 423  $\mu\text{m}$  and  $3.4\pm 0.3 \mu\text{m}$ , respectively, with COVs of 8.9 % and 8.8 %, respectively. In Fig. 13(b), the average  
 424 widths of the  $\alpha$ -lath and  $\beta$ -lath were measured at  $9.1\pm 1.2 \mu\text{m}$  and  $3.4\pm 0.6 \mu\text{m}$ , respectively, with COVs  
 425 of 13.2 % and 17.6 %, respectively. It is noted that the average width of  $\alpha$ -laths is higher near the fracture  
 426 surfaces than at 15 mm away from the fracture surface, and the average width of  $\beta$ -laths is  $3.4 \mu\text{m}$  in  
 427 both regions. However, the difference in the first case is small and falls within the standard deviations  
 428 of the two means.

429 **3.3.5. Microstructures of Specimens that were Stress relieved Followed by High-Temperature Annealing**  
 430 **and then Tested at a Temperature of 175 °C.**

431 Fig. 14 shows optical micrographs of stress relieved followed by high temperature annealed  
 432 Ti6Al4V(ELI) samples and tested at a temperature of 175 °C.



433 **Fig. 14.** Optical micrograph of a stress relieved followed by high temperature annealed DMLS Ti6Al4V(ELI)  
 434 sample and tested at a temperature of 175 °C (a) near the fracture surface (b) 15 mm away from the fracture  
 435 surface  
 436

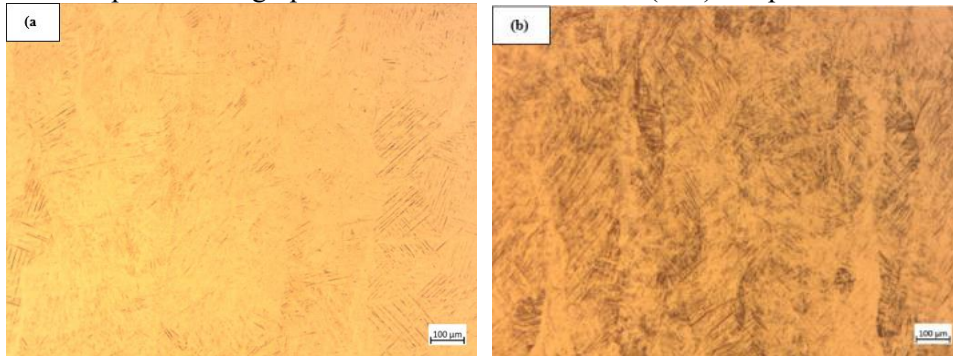
437 The two figures show both lamellar and Widmanstätten microstructures, with some areas having  
 438 globular  $\alpha$ -grains. Fig. 14(a) shows the presence of bimodal microstructures. In Fig. 14(a), the  
 439 micrograph is darker than in Fig. 14(b), possibly as a result of transverse strains at the neck. In Fig.  
 440 14(a), the average width of the  $\alpha$ -lath and  $\beta$ -lath was measured at  $13.1\pm 1.2 \mu\text{m}$  and  $4.0\pm 0.4 \mu\text{m}$ ,  
 441 respectively, with COVs of 9.2 % and 10 %, respectively. In Fig. 14(b), the average widths of the  $\alpha$ -laths  
 442 and  $\beta$ -laths was measured at  $9.1\pm 1.0 \mu\text{m}$  and  $3.2\pm 0.4 \mu\text{m}$ , respectively, with COVs of 11 % and 12.5 %,  
 443 respectively. These high values of COV, imply data that was scattered away from the mean value. Similar  
 444 to the samples that were tested at a temperature of 20 °C, the average widths of  $\alpha$ -laths and  $\beta$ -laths were  
 445 greater near the fracture surfaces than at 15 mm away from the fracture surface. This is thought to be  
 446 because of heating effects resulting from the mechanism of necking and fracture.

447 **3.3.6. Comparing Microstructures of Specimens that were Stress Relieved followed by High Temperature**  
 448 **Annealing and then Tested at Temperatures of 20 ° and 175 °C.**

449 Both Fig. 13(a) and Fig. 14(a) exhibit globular  $\alpha$ -grains, as well as areas of lamellar, Widmanstätten,  
 450 and bimodal microstructures. The measured average width of the  $\alpha$ -lath is greater in Fig. 14(a) than in  
 451 Fig. 13(a). This is thought to be because of the higher testing temperature. Zöllner [10], reported that  
 452 increasing temperatures led to coarser  $\alpha$ -laths and a reduction in strength. This is consistent with the  
 453 findings in the present work from tensile and hardness testing, which showed that as the test temperature  
 454 was raised there was a drop in values of yield and ultimate strength as well as hardness. The average  
 455 width of the  $\beta$ -laths is wider in Fig. 14(a) than in Fig. 13(a). This is thought to have been due to higher  
 456 testing temperature, which caused an increase in the width and the content of  $\beta$ -laths as well. This is  
 457 consistent with the study by Shaikh *et al.* [18]. It has been reported that the  $\beta$ -phase with its body-  
 458 centered-cubic (BCC) crystal structure is responsible for the ductility of the alloy [15, 32]. The  
 459 micrograph in Fig. 14(a) has a higher incidence of the bimodal microstructure than the micrographs in  
 460 Fig. 13(a). The bimodal microstructure is known to possess the highest ductility among the  
 461 microstructures of Ti6Al4V [35-36]. Again, this agrees with the findings from tensile testing in the  
 462 present work that as the test temperature was raised the ductility increased.

463 3.4. Comparing the Microstructures of the Soaked Samples to the Microstructures of Tensile Tested  
464 Samples Near the Fracture Surface.

465 Fig. 15 shows optical micrographs of stress relieved Ti6Al4V(ELI) samples.

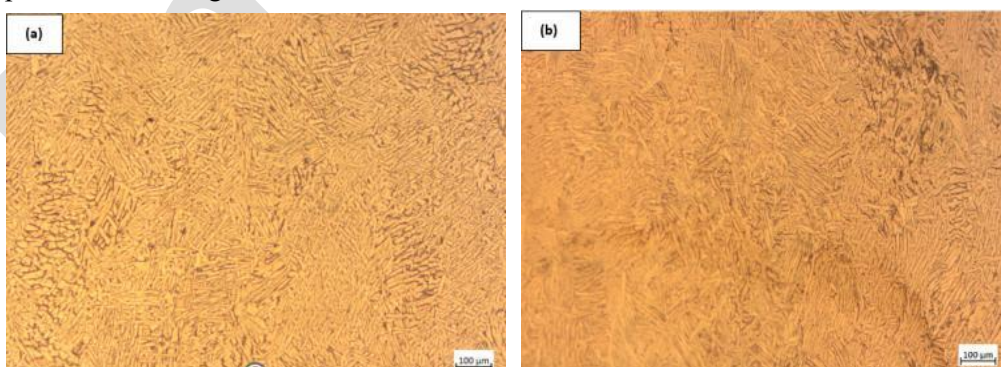


466  
467 **Fig. 15.** Optical micrographs of stress relieved DMLS Ti6Al4V(ELI) samples (a) soaked at 20 °C (b) soaked and  
468 tested till fracture at a temperature of 20 °C

469 The two micrographs show the presence of an  $\alpha'$  martensite microstructure and columnar  $\beta$ -grains.  
470 It is noted that Fig. 15(b) is darker than Fig. 15(a), which is thought to be a result of transverse strain at  
471 the neck. The average widths of the  $\alpha'$  lath were measured to be  $2.6\pm 0.47 \mu\text{m}$  for the micrograph in Fig.  
472 15(a) and  $3.8\pm 0.3 \mu\text{m}$  for the micrograph in Fig. 15(b). The average width of the  $\alpha'$  laths for the samples  
473 tested at a temperature of 20 °C is wider than that of the samples that were only soaked at the same  
474 temperature. This is thought to be due to the heating effects during the necking and fracturing of the  
475 specimen.

476 The samples that were soaked at a temperature of 133 °C, as well as soaked at 175 °C and tested in  
477 tension showed the presence of the  $\alpha'$  martensite microstructure and columnar  $\beta$ -grains. The average  
478 widths of the  $\alpha'$  laths for the sample that was only soaked at a temperature of 133 °C and the sample that  
479 was tested at a temperature of 175 °C were  $2.64\pm 0.55 \mu\text{m}$  and  $3.8\pm 0.4 \mu\text{m}$ , respectively. This is likely  
480 due to higher soaking temperature, as the sample in Fig. 15(b) was tested at a temperature of 175 °C  
481 after being soaked for 30 minutes at that temperature, while the sample in Fig. 15(a) was only soaked at  
482 a temperature of 133 °C. Both the sample's average widths of the  $\alpha'$  laths were observed to increase with  
483 the increase in testing and in soaking temperature. The average width of the columnar  $\beta$ -grains varies  
484 along the length for both the soaked samples and the tensile tested samples.

485 Fig. 16 shows optical micrographs of Ti6Al4V(ELI) samples that were stress relieved followed by  
486 high temperature annealing.



487  
488 **Fig. 16.** Optical micrographs of stress relieved followed by high temperature annealed DMLS Ti6Al4V(ELI)  
489 samples (a) soaked at 20 °C (b) soaked and tested till fracture at a temperature of 20 °C

490 The two figures show similar micrographs, both which exhibit globular  $\alpha$ -grains, areas of lamellar,  
491 Widmanstätten, and bimodal microstructures. In Fig. 16(a), the average widths of the  $\alpha$ -laths and  $\beta$ -laths  
492 were measured at  $8.2\pm 1.2 \mu\text{m}$  and  $1.2\pm 0.25 \mu\text{m}$ , respectively; while in Fig. 16(b) the average widths of  
493 the  $\alpha$ -laths and  $\beta$ -laths were measured at  $10.1\pm 0.9 \mu\text{m}$  and  $3.4\pm 0.3 \mu\text{m}$ , respectively. The average widths  
494 of the  $\alpha$ -laths and  $\beta$ -laths of the samples soaked at 20 °C and then loaded in tension till fracture were  
495 observed to be wider than those of the sample that was only soaked at 20 °C. This is likely due to the  
496 heating effect due to loading and fracture of the specimens.

497 It was observed that as the soaking temperature and the testing temperature increased from 20 °C to  
498 175 °C, the average widths of the  $\alpha$ -laths and  $\beta$ -laths increased as well. This is consistent with the study  
499 by Shaikh *et al.* [18]. The recorded average widths of the  $\alpha$ -laths and  $\beta$ -laths for the sample that was  
500 tested at a temperature of 175 °C, were  $13.1\pm 1.2 \mu\text{m}$  and  $4.0\pm 0.4 \mu\text{m}$ , respectively; while for the sample  
501 that was soaked at the temperature of 133 °C they were  $8.3\pm 1.4 \mu\text{m}$  and  $1.8\pm 0.45 \mu\text{m}$ , respectively. The  
502 higher average widths of the  $\alpha$ -laths and  $\beta$ -laths for the former samples is noted. This is consistent with  
503 the studies by Zöllner [10], who reported that higher temperatures lead to coarsening of  $\alpha$ -laths, while  
504 Shaikh *et al.* [18], observed an increase in the content of  $\beta$ -laths as the soaking temperature increased,  
505 as is observed in Fig. 15 and some parts of Fig. 16.

#### 506 4. CONCLUSIONS

508 The aim of this study was to investigate the effect of temperature on the microstructure of the  
509 Ti6Al4V(ELI) alloy. The following conclusions were derived from the study.

- 510 • Stress relieve heat treatment does not change the original microstructure of acicular  $\alpha'$  laths  
511 existing within columnar  $\beta$ -grain.
- 512 • High temperature annealing heat treatment significantly influences the microstructure of  
513 Ti6Al4V(ELI), as the  $\alpha'$  laths are replaced by  $\alpha$ - and  $\beta$ -laths.
- 514 • As the soaking temperature and the testing temperature are increased, the width of the  $\alpha'$  lath,  
515  $\alpha$ -laths and  $\beta$ -laths increase as well.
- 516 • Increased tensile test temperature causes a decrease in the values of yield strength, ultimate  
517 tensile strength, modulus of elasticity, and hardness, as well as an increase in the values of  
518 ductility.
- 519 • For the samples that are tested in uniaxial tension, fracture comes with an increase of  
520 temperature, and an attendant increase in the widths of the  $\alpha'$  laths,  $\alpha$ -laths, and  $\beta$ -laths, which  
521 increase is greater near the fracture surface than at a distance of 15 mm away from the fracture  
522 surface.
- 523 • Fracture is accompanied by an increase in temperature and an attendant increase in the widths  
524 of the  $\alpha'$  laths,  $\alpha$ -laths, and  $\beta$ -laths compared to those of samples that are only soaked at  
525 different temperatures.
- 526 • Stress-relieved samples tested in tension at a temperature of 20 °C, had the highest yield and  
527 tensile strength, modulus of elasticity and hardness but lowest ductility. Samples that were  
528 stress relieved followed by high temperature annealing and then tested in tension at 350 °C had  
529 the highest ductility but lowest yield strength, ultimate tensile strength, modulus of elasticity,  
530 and hardness.

531 Clearly, changes in temperature does influence the microstructure and mechanical properties of  
532 Ti6Al4V(ELI). As the present work was a prelude to elevated temperature fatigue testing to determine  
533 whether these elevated test temperatures would affect the microstructures of the Ti6Al4V(ELI)  
534 samples, it can be said that changes in testing temperature are expected to influence the fatigue  
535 properties of the alloy. Moreover, the known heating effects arising from loading and failure of  
536 materials are expected to be exacerbated by fatigue loading.

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