

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

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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 was 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 α' 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 β -field during the DMLS process [9]. The acicular α' 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 α - and β -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 α -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 β -grains
53 to $\alpha+\beta$ grains during slow cooling from the high temperature single-phase β 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 (μm)	Layer thickness (μm)	Scanning speed (mm/s)	Laser diameter (μm)
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 α - to β -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 β -grains to α -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 μm 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 μm 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

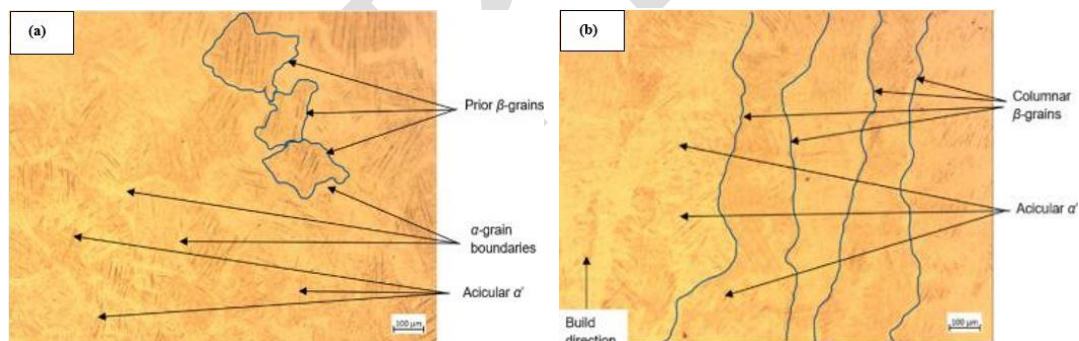
133 After tensile testing, small samples were cut from the uniaxial tensile fractured specimens, and an
134 electrical discharge machine (EDM) wire cutter was used to cut the specimens, along the longitudinal z-
135 build direction. The specimens were then cut in a transverse direction 15 mm away from 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.



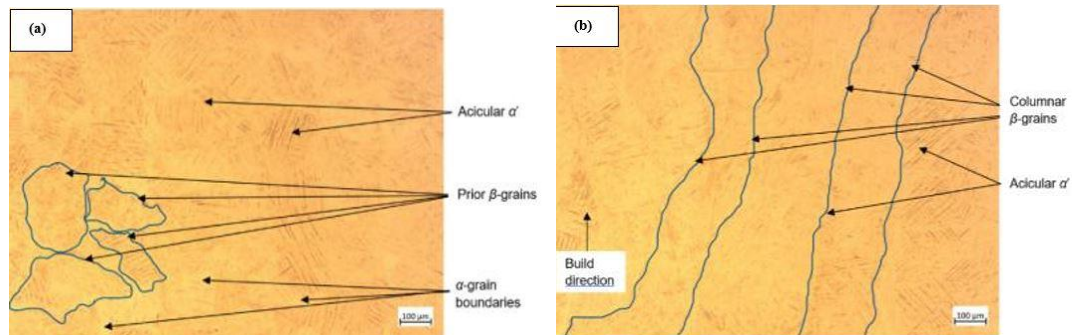
146 **Fig. 1.** Optical micrograph of the (a) top and (b) side view of an as-built DMLS Ti6Al4V(ELI) sample

148 The micrographs in the two figures show different morphologies of prior β -grains. Fig. 1(a), which
149 is the top view of an as-built sample, shows a lighter shade needle-like α' -lath referred to as α' martensite
150 microstructure. The figure also shows epitaxial (columnar) prior β -grains whose outlines are shown by
151 blue colour lines on the micrograph and which are separated by α -grain boundaries. Fig. 1(b) shows the
152 side view of the sample, consisting of columnar β -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 β -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 β -grains were found α'
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 α' martensitic microstructure is formed as a result of
161 rapid cooling (at rates in the range 10^4 - 10^6 K/s) of the alloy from the high temperature in the β -field
162 during the DMLS process [9]. Madikizela *et al.* [26], found α' -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 α' -laths varies from 1
164 to 3 μm . Phutela *et al.* [28], found an average columnar β -grains width of 90 μm in SLM as-built Ti6Al4V

165 samples, while Li *et al.* [29], found an average grain diameter of prior β -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.

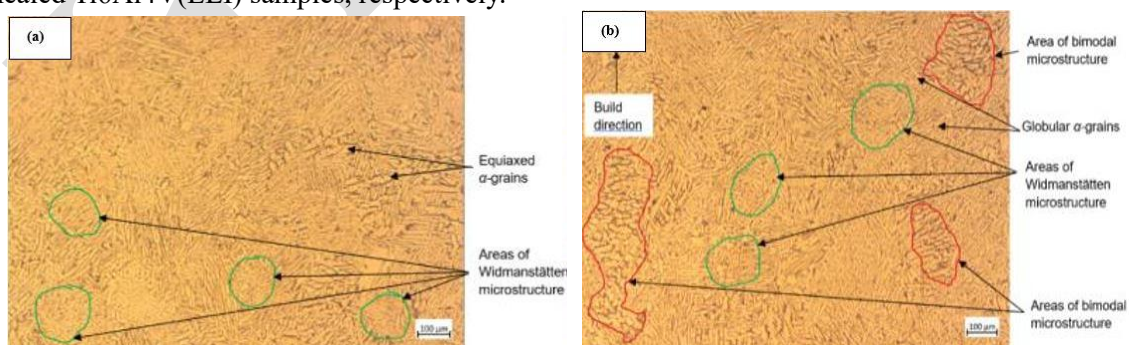


171
172 **Fig. 2.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to
173 the build direction) of a stress-relieved DMLS Ti6Al4V(ELI) sample

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 α' 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 β -grains which were also evident in the side view of as-built samples. Within these
180 columnar β -grains exist acicular α' 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 β -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.



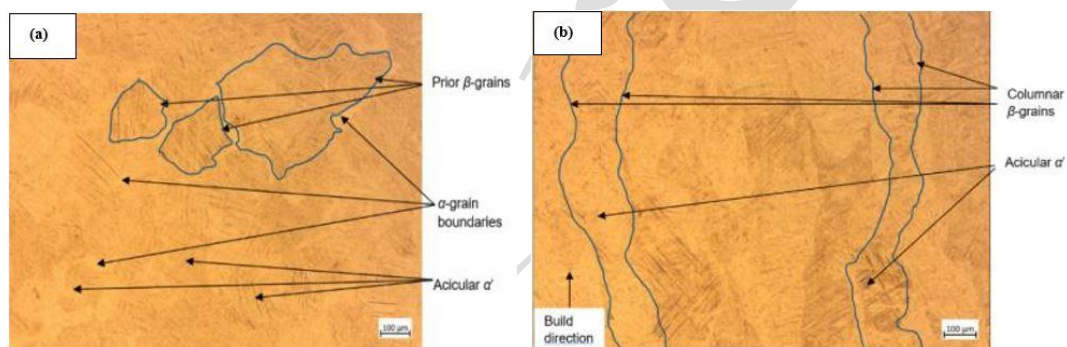
190
191 **Fig. 3.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to
192 the build direction) of a sample stress relieved and then at high temperature annealed DMLS Ti6Al4V(ELI)
193 sample

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 α' needles-like structures no longer exist and are replaced by larger globular α -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 α -laths [10-11]. Fig.
 201 3(a) exhibits a mixture of equiaxed and lamellar α -laths. Both the micrographs in Fig. 3 consist of
 202 globular α -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 α -laths is about $8.2 \pm 1.2 \mu\text{m}$, and therefore, a COV of 14.6 %. The average width of the β -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 α -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 α -laths obtained in the present study. Zöllner [10], stated that
 216 higher temperatures lead to even coarser α -laths. Jovanovic' *et al.* [32], reported that the thickness of the
 217 α -laths increases as the annealing temperature is increasing. They found that the average thickness of
 218 the α -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 α' 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 β -grains whose outlines
 232 are shown in blue colour lines and are separated by α -grain boundaries. As was the case in Fig. 2(b), the
 233 columnar β -grains in this figure have within them α' martensitic laths. The average width of the columnar
 234 β -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.

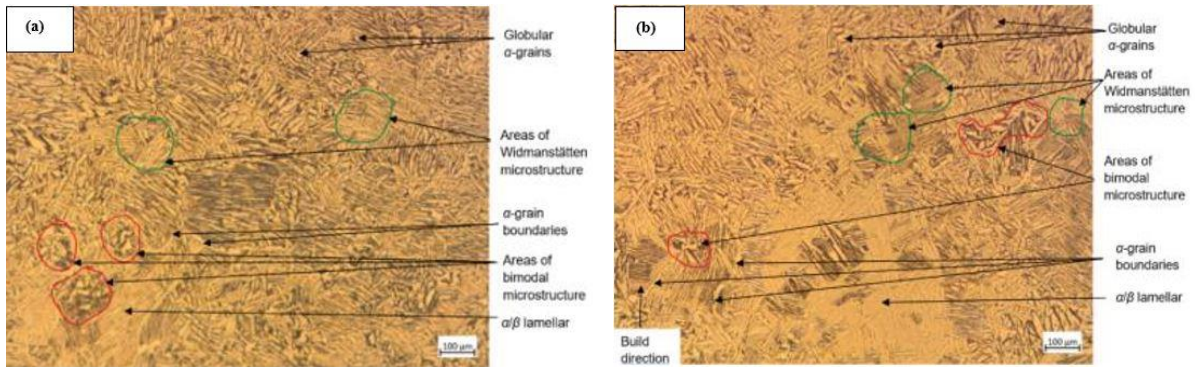


Fig. 5. Optical micrograph of the top view (perpendicular to the build direction) of a stress relieved, high temperature annealed and then soaked at 133 °C DMLS Ti6Al4V(ELI) sample

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In Fig. 5, the α - and β -phases are clear, with the α -laths being brighter and β -laths darker as shown by the arrows in these figures. In both these two figures, α -grain boundaries, globular α -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 α -laths of $8.3 \pm 1.4 \mu\text{m}$, giving rise to a COV of 16.9 %. The average width of α -laths has increased from 8.2 to 8.3 μm 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 α -laths is inconsequential. Moreover, the two means fall within the standard deviations of one another. The average width of β -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 β -laths has increased from 1.2 to 1.8 μm when compared with the value obtained from Fig. 3(b). This is a much greater (50 %) than was recorded for the α -laths and is a sign that the effect of soaking at 133 °C on the growth of β -laths is significant effect of. This increase in the width of β -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].

3.1.6. The Microstructure of Sample Group F

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

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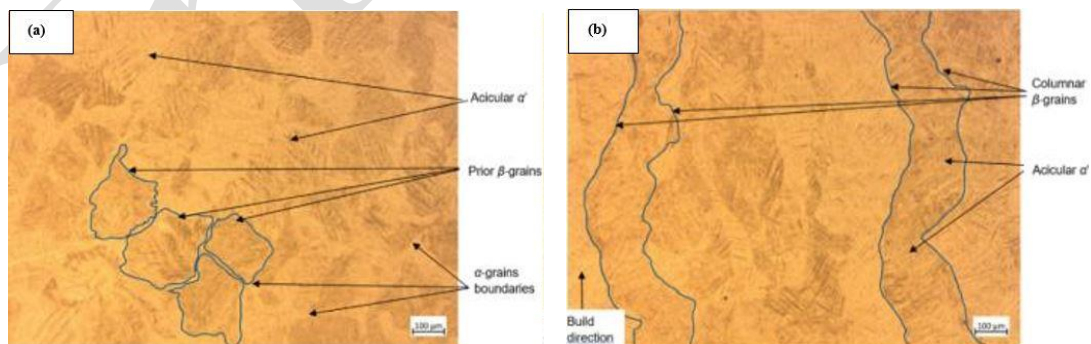


Fig. 6. Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to the build direction) of a DMLS Ti6Al4V(ELI) stress relieved sample, soaked at a temperature of 241 °C

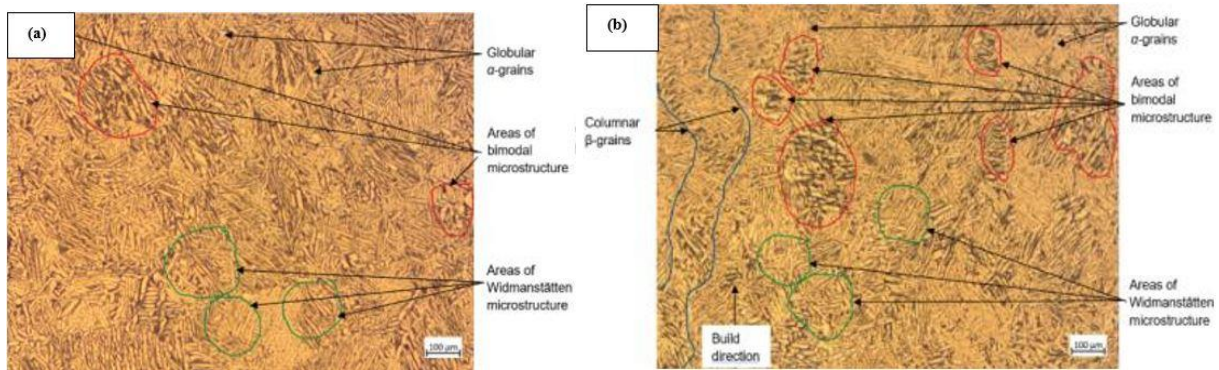
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Prior β -grains are evident on the micrograph in Fig. 6(a), whose outlines are shown by blue colour lines and are separated by α -grain boundaries on the micrograph. The acicular α' martensitic microstructure, with an average width of the α' laths 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 α' laths has increased from 2.64 to 2.9 μm 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 β -grains whose outlines

279 are shown by continuous blue lines within which exist α' martensite grains. The average thickness of the
 280 columnar prior β -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 β -laths. Shaikh *et al.* [18], observed an increase
 284 in the content of β -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.

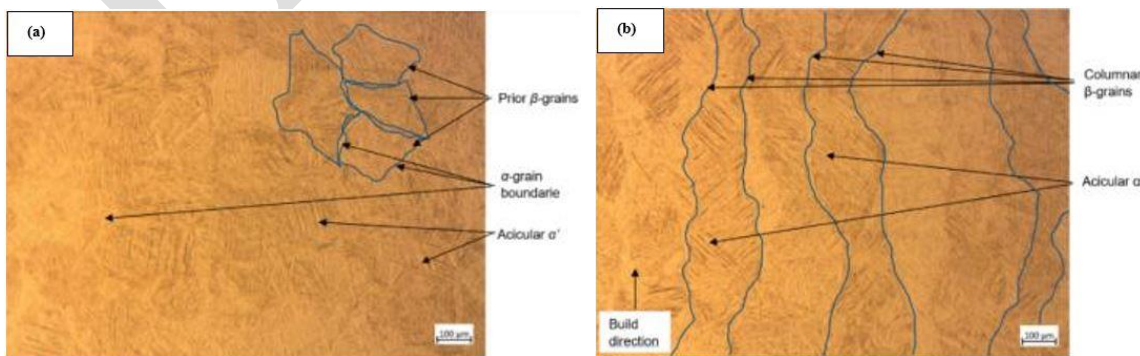


289
 290 **Fig. 7.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to
 291 the build direction) of a sample soaked at 241 °C for three hours, after stress relieving followed by high
 292 temperature annealing

293 Fig. 7 shows the α -laths being brighter and β -laths darker. Both the two figures show globular α -
 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 β -grain is visible in
 296 Fig. 7(b), whose outline is shown by blue lines, within which are colonies of α - and β -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 α -laths was determined as $8.3\pm 2.3 \mu\text{m}$, while the measured average width of the β -laths was $1.9\pm 0.7 \mu\text{m}$.
 300 The coefficient of variation for the width of the α -laths and β -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 β -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.



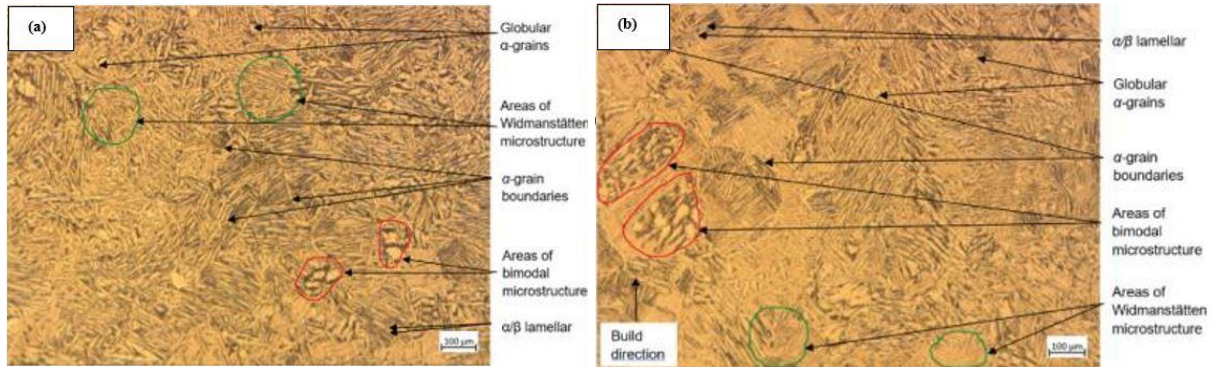
307
 308 **Fig. 8.** Optical micrograph of the (a) top view (perpendicular to the build direction) and (b) side view (parallel to
 309 the build direction) of a stress relieved DMLS Ti6Al4V(ELI) sample soaked at a temperature of 349 °C for three
 310 hours

311 Fig. 8 shows similar morphologies of microstructure as observed in Fig. 6, respectively. Fig. 8(a)
 312 shows prior β -grains, with α' martensite laths within them. These prior β -grains are separated by α -grain
 313 boundaries. The width of the martensitic α' laths within the prior β -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 α' 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 α' laths vary from 1 to $3 \mu\text{m}$ for as-built samples.
 317 Slightly higher values of the width of the α' 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 β -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

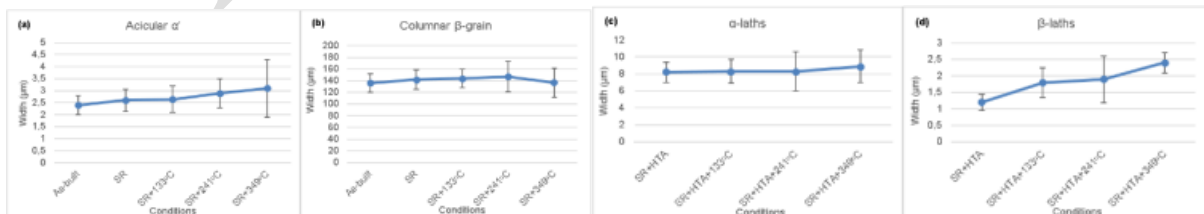
322 Fig. 9, shows the top and side view optical micrographs of stress relieved and high temperature
 323 annealed Ti6Al4V(ELI) samples, respectively that were soaked at a temperature of $349 \text{ }^\circ\text{C}$ for three
 324 hours before being cooled in the air.



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

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 β -laths. The
 332 micrographs show areas of α - and β -phases that are stacked parallel to each other forming a lamellar
 333 microstructure. There are also areas of globular α -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 α -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 β -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 β -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 β -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
 345 **Fig. 10.** The average width of the (a) acicular α' laths, (b) columnar β -grains, (c) α -laths and (d) β -laths

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 α' 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 β -grains
 349 increases as well. However, at $349 \text{ }^\circ\text{C}$ the width of the columnar prior β -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 α - and

351 β -laths increase. This result is similar to the study done by Zöllner [10], who stated that higher
 352 temperatures lead to even coarser α -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

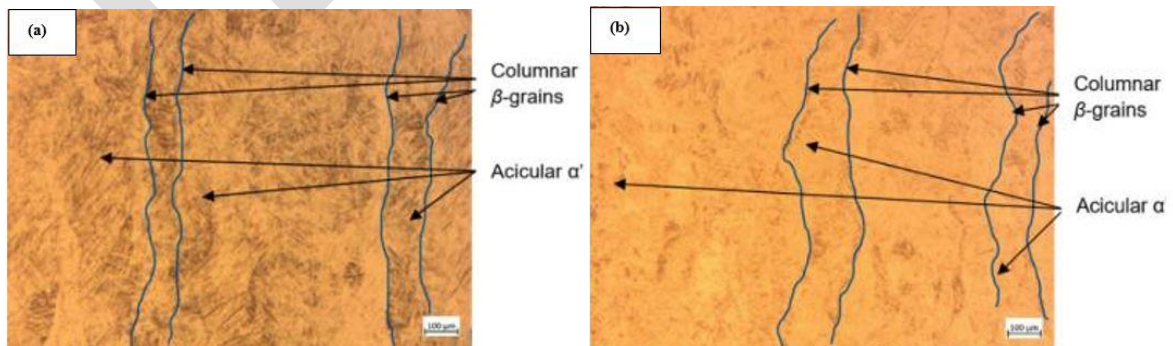
Temperature °C	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Vickers Micro-Hardness (HV)	Percentage Elongation (%)
20	888.4±33.9	953.7±59	99.5±17	312.5±25.3	13.8±2.1
175	597.2±84.3	674.8±83	91.7±13.5	310.4±33.9	13.9±1.6
325	544.3±33.4	654.9±6.7	83.3±1.1	-	17.3±4.6

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



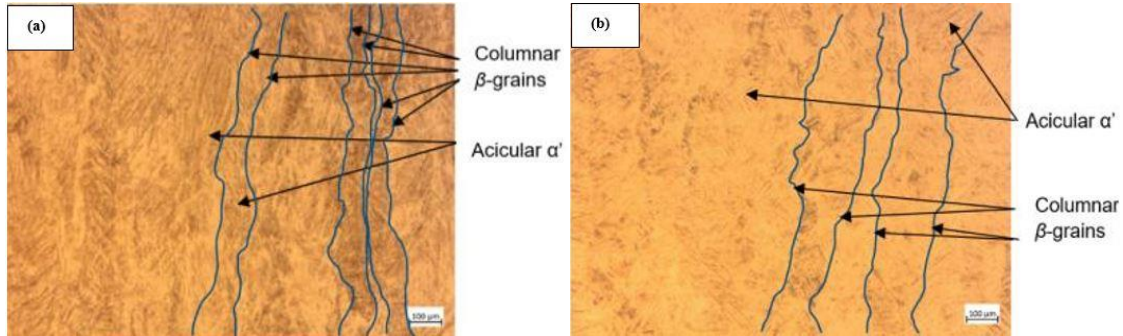
377 **Fig. 11.** Optical micrograph of a stress relieved DMLS Ti6Al4V(ELI) sample and tested at a temperature of 20
 378 °C (a) near the fracture surface and (b) 15 mm away from the fracture surface

379
 380 The two figures show a needle-like α' lath known (dark lines) martensite microstructure, inside
 381 columnar β -grains. The average widths of the columnar β -grains in Fig. 11(a) and Fig. 11(b) are 135±14
 382 μm and 136±11 μm , respectively, with coefficients of variance (COVs) of 10.4 % and 8.1 %, respectively.
 383 The average widths of α' martensitic laths in Fig. 11(a) and Fig. 11(b) are 3.8±0.3 μm and
 384 3.7±0.4 μm , 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 β -grains and α' 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 β -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

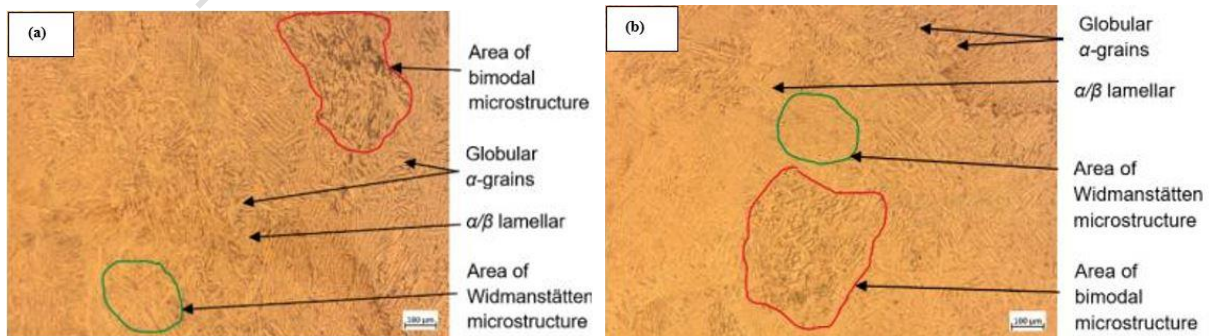
395 The two figures show the presence of columnar β -grains within them α' lath martensitic
 396 microstructure. In Fig. 12(a), the average widths of the columnar β -grains and α' laths are $146 \pm 13 \mu\text{m}$
 397 and $3.8 \pm 0.4 \mu\text{m}$, respectively, with COVs of 8.9 % and 10.5 %, respectively. In Fig. 12(b), the average
 398 widths of the columnar β -grains and α' 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 β -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.

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 β -grains and α' 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.

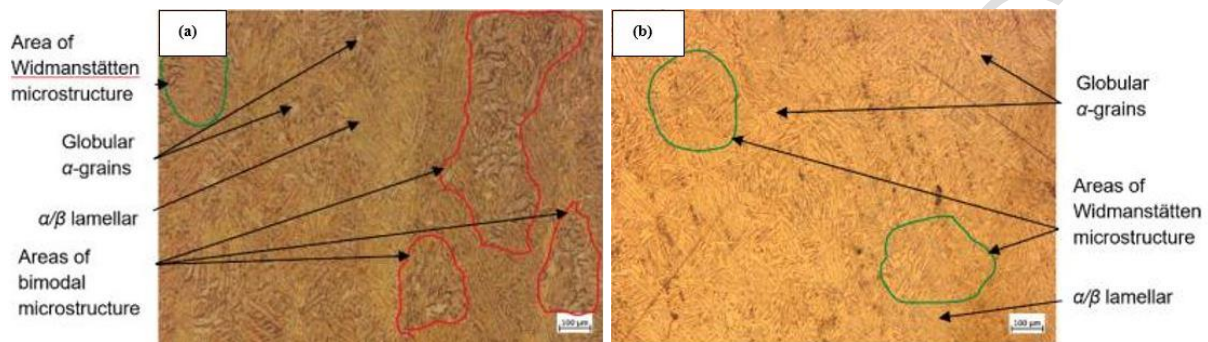


416 **Fig. 13.** Optical micrograph of a stress relieved followed by high temperature annealed DMLS Ti6Al4V(ELI)
 417 sample and tested at a temperature of 20 °C (a) near the fracture surface (b) 15 mm away from the fracture
 418 surface
 419

420 The two figures show similar micrographs with areas having α - and β -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 α -laths and β -laths were measured at 10.1 ± 0.9
423 μm 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 α -lath and β -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 α -laths is higher near the fracture
426 surfaces than at 15 mm away from the fracture surface, and the average width of β -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

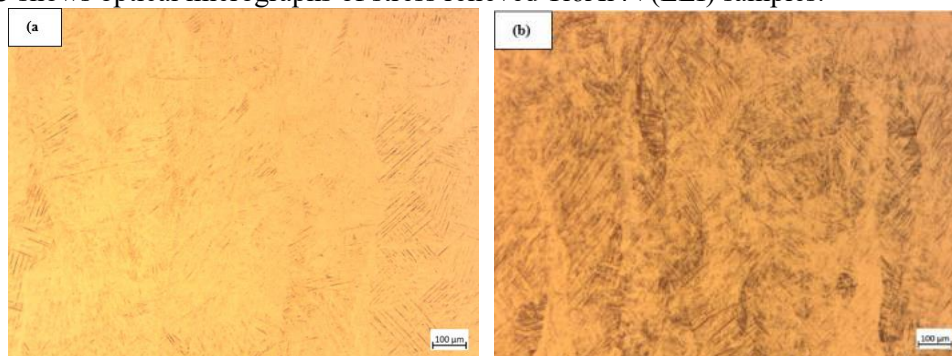
437 The two figures show both lamellar and Widmanstätten microstructures, with some areas having
438 globular α -grains. Fig. 14(a) shows the presence of bimodal microstructures. In Fig. 14(a), the
439 micrograph is darker than in Fig. 14(b), possibly as a result of transverse strains at the neck. In Fig.
440 14(a), the average width of the α -lath and β -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 α -laths
442 and β -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 α -laths and β -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 α -grains, as well as areas of lamellar, Widmanstätten,
450 and bimodal microstructures. The measured average width of the α -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 α -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 β -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 β -laths as well. This is
457 consistent with the study by Shaikh *et al.* [18]. It has been reported that the β -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.

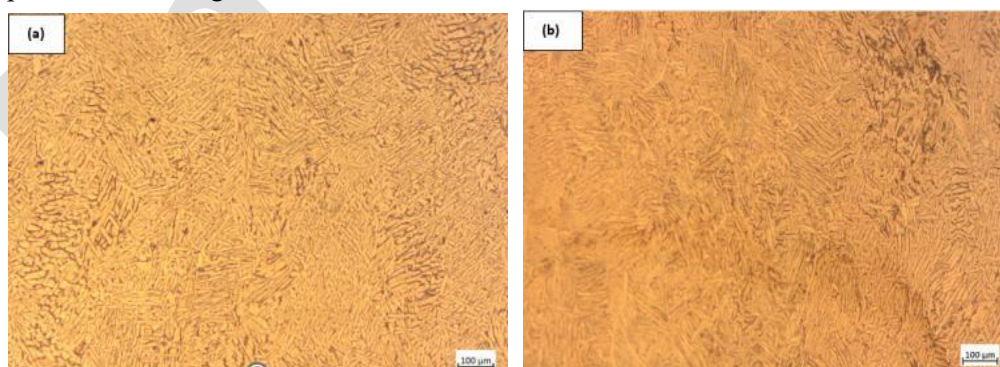


466
467 **Fig. 15.** Optical micrographs of stress relieved DMLS Ti6Al4V(ELI) samples (a) soaked at 20 °C (b) soaked and
468 tested till fracture at a temperature of 20 °C

469 The two micrographs show the presence of an α' martensite microstructure and columnar β -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 α' 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 α' 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 α' martensite microstructure and columnar β -grains. The average
478 widths of the α' 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 α' laths were observed to increase with
483 the increase in testing and in soaking temperature. The average width of the columnar β -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 α -grains, areas of lamellar,
491 Widmanstätten, and bimodal microstructures. In Fig. 16(a), the average widths of the α -laths and β -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 α -laths and β -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 α -laths and β -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 α -laths and β -laths increased as well. This is consistent with the study
499 by Shaikh *et al.* [18]. The recorded average widths of the α -laths and β -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 α -laths and β -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 α -laths, while
504 Shaikh *et al.* [18], observed an increase in the content of β -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 α' laths
511 existing within columnar β -grain.
- 512 • High temperature annealing heat treatment significantly influences the microstructure of
513 Ti6Al4V(ELI), as the α' laths are replaced by α - and β -laths.
- 514 • As the soaking temperature and the testing temperature are increased, the width of the α' lath,
515 α -laths and β -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 α' laths, α -laths, and β -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 α' laths, α -laths, and β -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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541 21-MOA-CUT-03).

542 REFERENCES

- 543 [1] Wohlers Associates, *What is Additive Manufacturing*, Washington DC, 2010.
- 544 [2] Dehghanghadikolaei, A., Namdari, N., Mohammadian, B. and Fotovvati, B., "Additive
545 manufacturing methods, A brief overview," *Journal of Scientific and Engineering Research*,
546 2018, 5, 123-131.

- 547 [3] Liu, S. and Shin, Y.C., "Additive Manufacturing of Ti6Al4V alloy: A review," *International*
548 *Journal of Fatigue*, 2019, 164, 107552.
- 549 [4] Chadstand, V., Quagebeur, P., Maia, W. and Charkaluk, E., "Comparative study of fatigue
550 properties of Ti-6Al-4V specimens built by electron beam melting (EBM) and selective laser
551 melting (SLM)," *Materials Characterization*, 2018, 143, 76-81.
- 552 [5] Huang, H., Zhang, T., Chen, C., Hosseini, S.R.E., Zhang, J. and Zhou, K., "Anisotropy in the
553 Tensile Properties of a Selective Laser Melted Ti-5Al-5Mo-5V-1Cr-1Fe Alloy during Aging
554 Treatment", *Materials*, 2022, 15, 5493.
- 555 [6] Zhang, T., Huang, H., Hosseini, S.R.E., Chen, W., Li, F., Chen, C., Zhou, K., "Obtaining
556 heterogeneous α laths in selective laser melted Ti-5Al-5Mo-5V-1Cr-1Fe alloy with high
557 strength and ductility", *Materials Science and Engineering: A*, 2022, 835, 142624.
- 558 [7] Patterson, A.E., Messimer, S.L. and Farrington, P.A., "Overhanging Features and the
559 SLM/DMLS Residual Stresses Problem: Review and Future Research Need," *Technologies*, 2017,
560 5, 15-38.
- 561 [8] Becker, T.H., Beck, M. and Scheffer, S., "Microstructure and Mechanical Properties of Direct
562 Metal Laser Sintered Ti6Al4V," *South African Journal of Industrial Engineering*, 2015, 26, 1-10.
- 563 [9] Muiruri, A. M., "Investigation of the High Strain Rate Behaviour and Impact Toughness of
564 Ti6Al4V (ELI) Parts Built by the EOS M280 DMLS System with Standard Process Parameters;
565 As-Built and Stress Relieved," MEng dissertation, Department of Mechanical and Mechatronics
566 Engineering at Central University of Technology Free State, Bloemfontein, 2018.
- 567 [10] Zöllner, D., "Impact of a strong temperature gradient on growth in films," *Modelling and*
568 *Simulation in Material and Engineering*, 2022, 30, 16.
- 569 [11] Campbell, F., "Fatigue and Fracture, Understanding the basics", ASM International, Ohio, USA
570 2012, 415-447.
- 571 [12] Sterling, A., Shamsaei, N., Torries, B. and Thapson, S.M., "Fatigue Behaviour of Additively
572 Manufactured Ti-6Al-4V," The 6th Fatigue Design Conference, Mississippi, 2015, 133, 576-589.
- 573 [13] Basuki, E.A., Prajitno, D.H. and Muhammad, F., "Alloys Developed for High Temperature
574 Applications," Proceedings of the 1st International Process Metallurgy Conference (IPMC 2016),
575 Bandung, 2017.
- 576 [14] Huo, Y., Yu, W., He, T., Hosseini, S.R.E., Ji, H., Bai, J., Chen, H. and Zhou, Z., "Numerical
577 Prediction and Experimental Investigation of Ductile Damage During Cold Upsetting of TC16
578 Titanium Alloy for Manufacturing Fasteners", *Transactions of the Indian Institute of Metals*,
579 2023, 76, 2963-2973.
- 580 [15] Zhao, J., Hung, F., Lui, T. and Wu, Y., "The Relationship of Fracture Mechanism between High
581 Temperature Tensile Mechanical Properties and Particle Erosion Resistance of Selective Laser
582 Melting, Ti-6Al-4V Alloy," *Metals*, 2019, 9, 501-5015.
- 583 [16] Song, J., Han, Y., Fang, M., Hu, F., Ke, L., Li, Y., Lei, L. and Lu, W., "Temperature sensitivity of
584 mechanical properties and microstructure during moderate temperature deformation of selective
585 laser melted Ti-6Al-4V alloy," *Material Characterization*, 2020, 165, 110342.
- 586 [17] Sieniawski, J., Ziąja, W., Kubiak, K. and Motyka, M., "Microstructure and Mechanical
587 Properties of High Strength Two-Phase Titanium Alloys," InTech, Poland, 2013.
- 588 [18] Shaikh, A., Kumar, S., Dawari, A., Kirwai, S., Patil, A. and Singh, R., "Effect of Temperature and
589 Cooling Rates on the $\alpha+\beta$ Morphology of Ti-6Al-4V Alloy," The 2nd International Conference on
590 Structural Integrity and Exhibition, Pune, 2018, 14, 782-789.
- 591 [19] Zherebtsov, S., Murzinova, M.A., Klimova, M.V., Salishchev, G.A., Popov, A.A. Semiatin, S.L.,
592 "Microstructure evolution during warm working of Ti-5Al-5Mo-5V-1Cr-1Fe at 600 and 800°C,"
593 *Material Science and Engineering*, 2013, 563, 168-176.
- 594 [20] Xu, J., Zhu, J., Fan, J., Zhou, Q., Peng, Y. and Guo, S., "Microstructure and mechanical
595 properties of Ti-6Al-4V alloy fabricated using electron beam freeform fabrication," *Vacuum*,
596 2019, 167, 2019, 364-373.
- 597 [21] Ter Haar, G.M., Becker, T.H., Blaine, D.C., "Influence of Heat Treatments on the Microstructure
598 and Tensile Behaviour of Selective Laser Melting-Produced Ti-6Al-4V," *South African Journal*
599 *of Industrial Engineering*, 2016, 27, 174-183.
- 600 [22] ASM International, "Heat treating," ASM International, Ohio, 2015.

- 601 [23] Reiners, W., Pyzalla, A.R., Schreyer, A. and Clemens, H., "Microstructure and Properties of
602 Engineering Materials," *Neutrons and Synchrotron Radiation in Engineering Materials Science:
603 From Fundamentals to Applications*, Wiley-VCH Verlag GmbH & Co. KGaA, 2017, 3-20.
- 604 [24] Pippan, R., Scheriau, S., Taylor, A., Hafok, M., Hohenwarter, A. and Bachmaier, A., "Saturation
605 of Fragmentation During Severe Plastic Deformation," *Annual Review of Materials Research*,
606 2011, 40, 2322-2329.
- 607 [25] Lekoadi, P., Tlotleng, M., Maledi, N. and Masina, B.N., "Improving the Microstructure of High
608 Speed Selective Laser Melted Ti6Al4V Components by Varying residence time during Heat
609 Treatment," *University of the Witwatersrand, School of Chemical & Metallurgical Engineering*,
610 Johannesburg, 2012.
- 611 [26] Madikizela, C., Cornish, L.A., Chown, L.H., and Moller, H., "Microstructure and mechanical
612 properties of selective laser melted Ti-3Al8V-6Cr-4Zr-4Mo compared to Ti-6Al-4V," *Materials
613 Science & Engineering*, 2019, 747, 225-231.
- 614 [27] Agius, D., Kourousis, K.I. and Wallbrink, C., "A Review of the As-built SLM Ti-6Al-4V
615 Mechanical Properties towards Achieving Fatigue Resistant Designs," *Metal*, 2018, 8, 1-25.
- 616 [28] Phutela, C., Aboulkhair, N.T., Tuck, C.J. and Ashcroft, I., "The Effects of Feature Sizes in
617 Selectively Laser Melted Ti-6Al-4V Parts on the Validity of Optimised Process Parameters,"
618 *Materials*, 2019, 13.
- 619 [29] Li, Y., Song, L., Xie, P., Cheng, M. and Xiao, H., "Enhancing Hardness and Wear Performance
620 of Laser Additive Manufactured Ti6Al4V Alloy Through Achieving Ultrafine Microstructure,"
621 *Materials*, 2020, 13.
- 622 [30] Jazdzewska, M., Kwidzińska, D.B., Seyda, W., Fydrych, D. and Zieliński, A., "Mechanical
623 Properties and Residual Stress Measurement of Grade IV Titanium and Ti-6Al-4V and Ti-13Nb-
624 13Zr Titanium Alloys after Laser Treatment," *Materials*, 2021, 14.
- 625 [31] Malefane, L.B., "Determination of the Fatigue Properties of Ti6Al4V(ELI) Parts Built By a
626 Direct Metal Laser Sintering System with Standard Process Parameters Followed By Post-
627 Processing Treatments," *Central University of Technology Free State, Bloemfontein*, 2019.
- 628 [32] Jovanovic', M.T., Tadić, S., Zec, S., Mišković, Z. and Bobić, I., "The effect of annealing
629 temperatures and cooling rates," *Materials and Design*, 2006, 27, 192-199.
- 630 [33] Zhao, J., Wang, Z., Tian, L., Wan, J., Zong, X., Zhang, S., Zhao, H., Zhang, S. Zhao, H., "Study
631 on mechanical properties of Ti-6Al-4 V titanium alloy with different microstructures under
632 combined tension-bending load," *Journal of Alloys and Compounds*, 2022, 936.
- 633 [34] Zhang, H., Wang, C., Zhou, G., Zhang, S. Chen, L., "Dependence of strength and ductility on
634 secondary α phase in a novel metastable- β titanium alloy," *Journal of Material Research and
635 Technology*, 2022, 18, 5257-5266.
- 636 [35] Nalla, R., Ritchie, R.O., Boyce, B.L., Campbell, J.P. and Peters, J.O., "Influence of Microstructure
637 on high cycle fatigue of Ti6Al4V: Bimodal vs. Lamellar structures," *Metallurgy and Material
638 Transactions*, 2002, 33, 899-918.
- 639 [36] Benedetti, M. and Fontánari, V. "The Role of Bimodal and Lamellar Microstructures of Ti-6Al-
640 4V on the Behaviour of Fatigue Cracks Emanating from Edge-Notches," *Department of
641 Materials Engineering and Industrial Technologies*, University of Trento, Trento, 2004.
- 642 [37] Zha, M., Zhang, H., Yu, Z., Zhang, X., Meng, X., Wan, H. and Jiang, Q., "Bimodal microstructure
643 - A feasible strategy for high-strength and ductility materials," *Journal of Materials Science &
644 Technology*, 2018, 34, 257-264.
- 645 [38] Xing, L., Zhang, W., Zhao, C., Gao, W., Shen, Z. and Liu, W., "Influence of Powder Bed
646 Temperature on the Microstructure and Mechanical Properties of Ti6Al4V," *Materials*, 2021, 14,
647 2278.