

X-ray diffraction profile analysis of martensitic Ti6Al4V (ELI) parts produced by laser powder bed fusion

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Abstract. The quantification of the density of defects in materials through non-destructive methods is of great interest to scientists and engineers. X-ray diffraction (XRD) peak profile analysis is a valuable method that is often used to reveal important microstructural information, such as defects present in crystalline materials as well as crystallite size. In this study, the broadening of XRD peaks of martensitic Ti6Al4V (ELI) produced by laser powder bed fusion (LPBF) was studied following modified Williamson-Hall (MWH) and Warren-Averbach (WA) analytical methods. The level of defects in martensitic LPBF Ti6Al4V (ELI) was found to drastically reduce by at least 73% after exposing the material to stress-relieving heat treatment.

1 Introduction

The ideal X-ray diffraction (XRD) pattern consists of narrow, symmetrical, delta-function-like peaks, positioned into angle 2θ according to a well-defined unit cell plane [1]. The deviations from the ideal diffraction pattern are usually perceived as anisotropic peak broadening [1]. The most common cause of XRD anisotropic peak broadening in crystalline materials is believed to be dislocations and crystallite smallness, with other defects such as stacking and twin faults being less contributory [2]. Manufacturing defects that could manifest in the form of residual stresses, can be desirable or undesirable depending on how they influence the mechanical properties of the final components. For instance, laser peening is used to impart deep beneficial compressive residual stress in metals and alloy components as a method of surface enhancement to improve their fatigue strength and damage tolerance by inhibiting crack initiation and early crack growth [3-5]. Compressive residual stress is also induced to toughen glass to allow for large, thin, crack-and scratch-resistant glass displays on smartphones [4]. On the other hand, tensile residual stresses are detrimental, since they contribute to fatigue damage and stress corrosion cracking (growth of cracks due to the simultaneous act of stress and a reactive environment) [7].

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Laser powder bed fusion (LPBF) is among the most common additive manufacturing technologies currently utilized for the manufacturing of parts for use in industries such as biomedical, aerospace, and military [7–9]. The technology uses a laser heat source that directly fuses powder material during the fabrication process to obtain three-dimensional (3D) parts from digital design data. During this process, steep temperature gradients develop due to localised heating and rapid cooling, causing differential expansion and contraction in different locations of the produced components [9]. In the process, stresses and strains are generated, which remain in the material as residual stresses and strains [10]. These residual stresses in LPBF Ti6Al4V (ELI) have been widely studied and published by players in the field of AM [7–12]. While materials with high levels of residual stresses and strains are often full of defects and flaws, such as dislocations and twins that impact the mechanical properties, details on the intensity of occurrence of these defects in fabricated LPBF Ti6Al4V (ELI) are still scant [11].

Fully martensitic α' microstructure with high dislocation density and stacking faults was observed in [13] using transmission electron microscopy (TEM). The arrangement, type and density of dislocation structures for wrought Ti6Al4V undergoing cyclic tensile straining were studied post-mortem employing TEM in reference [14]. Recently, Yamanaka, *et al.* [15] performed time-of-flight neutron diffraction (TOF-ND) measurements of Ti6Al4V alloys prepared by Electron Beam Powder Bed Fusion (EBPBF) and further examined the dislocation density in as-built states using convoluting multiple whole profile (CMWP) fitting. The study determined the bulky average dislocation density in the as-built α' -matrix microstructure as $6.8 \times 10^{13} \text{ m}^{-2}$. Though the EBPBF process is very similar to the LPBF process, which entails a layer-by-layer build process, it nevertheless has some differences when compared to the LPBF process. In the former, an electron beam is used to melt and fuse the powder particles instead of a laser beam. More so in EBPBF, the powder bed is kept at a high temperature ($>700^\circ \text{C}$) and overnight cooling time is required to cool the powder bed after manufacturing parts [16]. The EBPBF process results in an $\alpha+\beta$ microstructure of Ti6Al4V with lower residual stress, in contrast to LPBF, which results in α' -acicular martensitic microstructure, with high tensile residual stresses [16]. The foregoing discussion suggests that the level of defects in as-built Ti6Al4V samples produced by these two different PBF processes will differ.

In this paper, the dislocation density and crystallite size in as-fabricated and stress-relieved LPBF Ti6Al4V (ELI) are estimated using XRD peak profile analysis following the modified William-Hall and modified Warren-Averbach methods. The analysis of XRD peak profiles to determine the dislocation density in polycrystalline materials by these two methods can be found in references [1, 17-19]. The results obtained in this study were compared for the two types of microstructures, and from these, the effects of stress-relieving heat treatment of as-fabricated LPBF Ti6Al4V (ELI) parts determined. The results were further compared to those obtained in reference [15] for EBPBF parts, to determine the influence of the two types of PBF processes on the level of defects in the materials produced.

2 Materials and method

The samples used in the study were produced by direct metal laser sintering (DMLS), a trademarked variant of LPBF. An EOSINT M280 machine was used to fabricate cylindrical samples with a diameter and length of 6 and 80 mm, respectively, using gas-atomised spherical Ti6Al4V (ELI) powder. The longitudinal axis of the samples was aligned parallel to the build direction. The cylindrical samples produced here were to be used in separate research to study the compressive properties of the alloy. The work presented in this study is limited to dislocation analysis and hence details on the effect of build orientation on mechanical properties are not discussed. Some of the produced samples were stress relieved

to alleviate their process-generated residual stresses, at a temperature of 650 °C in an argon gas atmosphere for a soaking time of 3 h, followed by furnace cooling to room temperature. These conditions of heat treatment for this material have been shown to cause negligible microstructural transformation [13]. Stress-relieving heat treatment was executed using an SS12/24-13MDX Super Series™ vacuum furnace system with a horizontal vacuum chamber.

In this study small pieces of samples with a length of 12 mm were cut from the produced samples of length 80 mm for XRD analysis. These 12 mm samples were sectioned into halves along the length (across the diameter) using an electrical discharge machine (EDM-wire cutting). The EDM wire cutting is normally suitable for cutting such small work pieces, creating smooth surfaces without burrs, while maintaining close tolerances. The cut surfaces of the as-built and stress-relieved Ti6Al4V samples were then mounted before undergoing chemical and mechanical polishing and thereafter cleaned under tap water. Kroll’s etchant, consisting of 2mL HF (hydrofluoric acid), 92 mL distilled water and 6 mL HNO3 (nitric acid), was then applied to the surfaces of the polished LPBF Ti6Al4V (ELI) samples to help delineate grain boundaries on them. The microstructure of the samples was then studied using a JOEL JSM-7001 scanning electron microscope (SEM).

Subsequently, XRD analysis of the produced and polished samples was conducted using a Bruker D2 Phaser equipped with a cobalt anode, whose operating set of parameters is shown in Table 1. The common Bragg reflections for the α/α -phase and the {110} plane for the β -phase of Ti6Al4V (ELI) were identified and measured. The information of these peaks was further analysed using the modified Williamson-Hall (MWH) and modified Warren-Averbach methods (MWA).

Table 1. Operating parameters of Bruker D2 Phaser

Bruker D2 Phaser	Parameter	Value
X-ray source Co K α 1/ K α 2	Wavelength	0.1788 nm/ 0.1792 nm
Detector	2 θ angular range	5.638°
Goniometer Radii	Primary radius	70.7 mm
	Secondary radius	70.7 mm
Slits	Primary Söller	2.5°
	Secondary Söller	2.5°

3 Results and discussion

3.1 Microstructure of LPBF Ti6Al4V (ELI)

Figure 1 shows the microstructures of the LPBF Ti6Al4V (ELI) alloy in the as-built and stress relieved states.

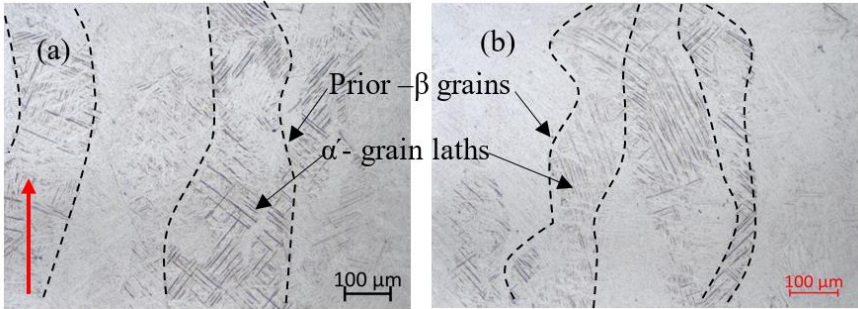


Fig 1. The microstructures of LPBF Ti6Al4V (ELI) in (a) as-built state and (b) stress-relieved state (the red arrows show the build orientation during the DMLS process)

The microstructures of LPBF Ti6Al4V (ELI) in the as-built and stress-relieved states shown in Figure 1 consist of columnar prior β -grains (delineated by broken lines) elongated along the build direction. These prior β -grains are filled with α' -laths which have an acicular morphology. This non-equilibrium microstructure results from high cooling rates during the DMLS process, which can reach up to 10^6 °C/s [20]. The stress-relieving heat treatment at 650 °C for 3 hours did not result in noticeable microstructural change as shown in Figure 1 indicating negligible microstructural decomposition. A similar observation was also reported in [13] for this material, where no β -phase was detected using XRD and TEM. However, the partial decomposition of α' -martensitic microstructure is possible for temperatures lower than 650 °C for prolonged soaking periods as reported in [21]. Full decomposition of α' -martensitic microstructure has been realized for heat treatment temperatures of 800 °C and above [22, 23]. Various values for α' martensitic start temperature (M_s) have been reported in the range of 575 °C – 800 °C [24, 25]. Factors such as homogeneity of composition, initial microstructure and the presence and types of impurity elements are suggested in literature to affect the M_s [24, 26, 27].

3.2 X-ray diffraction peak profiles of LPBF parts

Figure 2 shows the XRD peak patterns of α -phase (hcp) in as-built and stress-relieved LPBF Ti6Al4V (ELI) specimens.

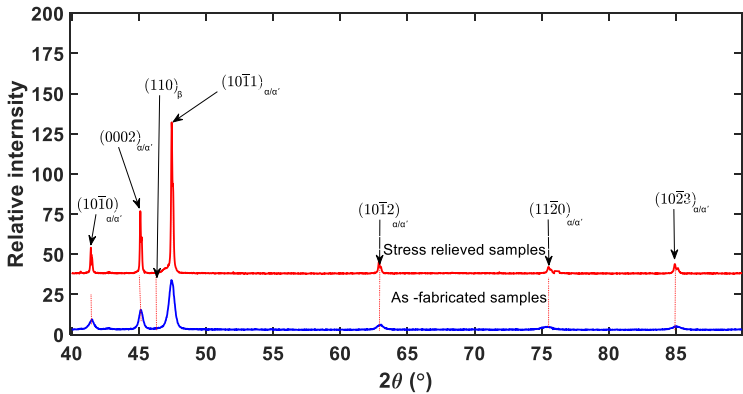


Fig 2. The as-built and stress relieved LPBF Ti6Al4V (ELI) XRD peak profiles

The XRD patterns in Figure 2 indicate Bragg reflections of $\{10\bar{1}0\}$, $\{0002\}$, $\{10\bar{1}1\}$, $\{10\bar{1}2\}$, $\{11\bar{2}0\}$ and $\{10\bar{2}3\}$ planes in the as-built and stress-relieved samples. There is no evidence in the figure that the β -phase was precipitated during rapid cooling of the DMLS process or transformed from the α -phase during stress relieving heat treatment. Similar observations were reported in Krakhmalev *et al.* [13] from XRD and TEM data of the alloy.

The Bragg peak reflections for all the planes shown in Figure 2 demonstrate a larger integral breath of the peaks for as-built samples as compared to the stress-relieved samples. Broadening of the peaks can arise from various factors including instrumental effects, small crystallite sizes and the presence of large defects (lattice strain) in polycrystalline materials [1, 2, 28]. The broadening of the XRD peaks in as-built samples was attributed to the high levels of residual stresses and strains associated with LPBF parts as a result of large thermal gradients and high cooling rates prevailing in their formation. Such residual stresses and strains are relieved upon heat treatment, thus leading to a reduction of the peak integral breaths as shown in Figure 2. It is important to note that only the α -peak reflections were considered in this study as no noticeable β -phase was detected via XRD as shown in Figure 2. The lattice change due elemental diffusion in this study was assumed to be negligible.

The lattice strain (ϵ_l) in polycrystalline materials is directly related to the dislocation density ρ present in such a material by the following expression [29]:

$$\rho \approx \frac{4\pi\langle\epsilon_l^2\rangle}{\ln\frac{D}{b}} \quad (1)$$

where the symbols D , C , L and b are crystallite size, average contrast factor, Fourier length and the Burgers vector of the dislocations, respectively. Even though this model shows how dislocation density in a material is related to lattice strain and crystallite size, the parameter $\langle\epsilon_l^2\rangle$ has been shown in [30] to diverge logarithmically with increasing crystallite size. Therefore, due to the long-range decay of strain fields created by dislocations, this model is not ideal for use in calculating the dislocation density [29]. If strain in materials is due to the presence of dislocations, the analysis of XRD peak profile broadening by MWH and MWA methods has been shown to yield positive results in the evaluation of the density of these dislocations [1, 2, 15, 19, 39].

3.2.1 LPBF Ti6Al4V (ELI) XRD peak profile analyses by the modified Williamson–Hall method

It is important to note that each observed peak profile is a convolution of the profiles of two main factors. These factors that are attributed to the observed peak profiles include instrumental broadening and sample broadening. The sample factors that contribute to broadening are crystallite size and lattice strain (micro-strain), previously mentioned in the introduction section. Therefore, at first, the instrumental broadening must be deconvoluted from sample broadening.

The backgrounds of the LPBF Ti6Al4V (ELI) XRD profiles presented in Figure 2 were subtracted and each peak reflection was normalised using the expression:

$$\int_{-\infty}^{\infty} I(2\theta)d2\theta = 1 \quad (2)$$

where the symbol I is the peak intensity.

It should be noted that due to the two different wavelengths of the X-ray source (cobalt) shown in Table 1, most diffraction data consist of $K\alpha_1$ and $K\alpha_2$ peak doublets rather than just a single peak according to Bragg's law. Such a peak doublet for a typical $(10\bar{1}1)$ reflection is shown in Figure 3.

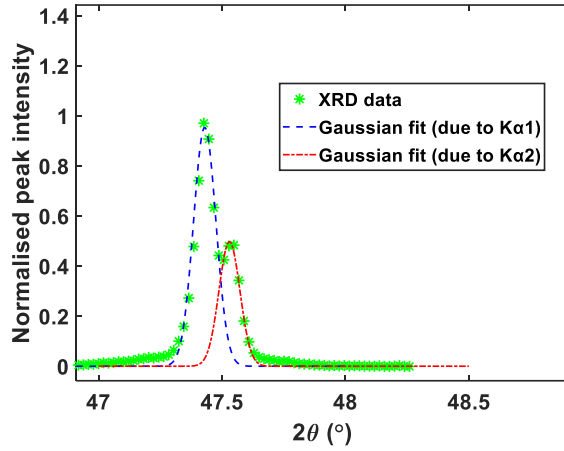


Fig. 3. A classical deconvoluted cobalt $K\alpha_1$ and $K\alpha_2$ peak doublets for $(10\bar{1}1)$ reflection using a Gaussian function.

To evaluate for strain and size broadening in this study, only the Co $K\alpha_1$ radiation was required and therefore, the individual peaks were deconvoluted into $K\alpha_1$ and $K\alpha_2$ using Gaussian and Lorentzian functions.

The MWH method is of the form [19]:

$$(\Delta K)^2 \cong \left(\frac{0.9}{D}\right)^2 + \left(\frac{\pi M^2 b^2}{2}\right) \rho K^2 \overline{C_{hkl}} \quad (3)$$

where K is the diffraction vector expressed as $K = 2\sin\theta/n\lambda$ in nm^{-1} and ΔK is expressed as $\Delta K = 2\cos\theta(\Delta\theta)/n\lambda$. In this case, the parameters θ and $\Delta\theta$ stand for the diffraction angle and the Full Width at Half Maximum (FWHM) in radians of the XRD peaks, respectively, while λ and n are the wavelength of the X-rays and order of reflection (taken as 1 in this study), respectively. The parameter M denotes a dimensionless constant that depends on the effective outer cut-off radius of a dislocation, while parameters D , C and b are as explained below Equation 1. The average contrast of dislocations in this equation is denoted as $\overline{C_{hkl}}$ and is expressed as [31]:

$$\overline{C_{hkl}} = \overline{C_{hk.0}}(1 + q_1x + q_2x^2), x = \frac{2}{3}\left(\frac{l}{Ka}\right)^2 \quad (4)$$

where the parameters, q_1 and q_2 are curve fitting parameters that depend on the elastic properties of materials, while a and l are lattice constants in the basal plane (taken as 0.295 nm for Ti-Hex) and the last index of the (hkl) peak reflection, respectively. The parameter $\overline{C_{hk.0}}$ is the average contrast factor corresponding to $(hk.0)$ reflections, which occurs in the planes where the last lattice index, l , is zero. The $\overline{C_{hk.0}}$ and q values for the common slip systems in titanium can be found in [2]. To evaluate the parameters q_1 and q_2 and therefore, the parameter $\overline{C_{hkl}}$, Equations (3) and (4) are combined to give a quadratic function of the form:

$$((\Delta K)^2 - \omega)/K^2 \cong X\overline{C_{hk.0}}(1 + q_1x + q_2x^2) \quad (5)$$

where, the parameters X and ω are defined as, $X = \pi M^2 b^2 \rho / 2$ and $\omega = \left(\frac{0.9}{D}\right)^2$. As Equation (5) is a quadratic function of x , the values of the parameters, q_1 and q_2 in it can be determined from the coefficient of the curve of $((\Delta K)^2 - \omega)/K^2$ against x .

The XRD peak broadening due to lattice strain and crystallite size is normally represented by Gaussian and Lorentzian distribution functions, respectively. Furthermore, the

instrumental broadening was measured from the diffraction pattern of a strain free sample that displays no broadening of its own. The National Institute of Standards and Technology (NIST) silicon Si-640d standard sample was used to measure the instrumental broadening.

The ΔK and K for each XRD peak of the as-built and stress relieved samples of LPBF Ti6Al4V (ELI) as well as that of the NIST Si640d standard sample were obtained while fitted separately as Lorentzian and Gaussian functions. Figure 4 shows the ΔK against K plots of the obtained results.

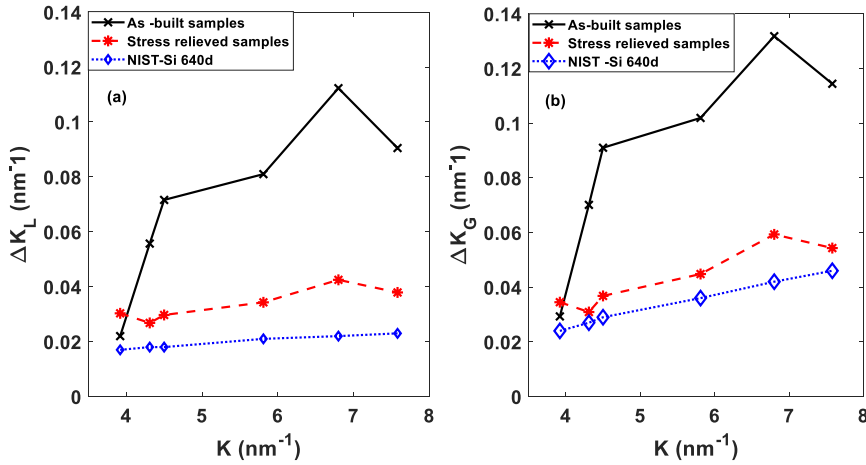


Fig. 4. The FWHM (ΔK) against the diffraction vector (K) for the as-built and stress-relieved LPBF Ti6Al4V (ELI) referenced against strain-free NIST Si-640d samples obtained from XRD data fitted as (a) a Lorentzian and (b) a Gaussian function.

The instrumental broadening was removed from each peak to obtain the true broadening at every diffraction vector using the following expressions [31]:

$$\Delta K_L^t = \Delta K_L^m - \Delta K_L^s \quad (6)$$

$$(\Delta K_G^t)^2 = (\Delta K_G^m)^2 - (\Delta K_G^s)^2 \quad (7)$$

where ΔK_G^t and ΔK_L^t are calculated true Gaussian and Lorentzian values of FWHM for the LPBF Ti6Al4V (ELI) samples. The parameters ΔK_L^m and ΔK_L^s denote the FWHM values of the LPBF Ti6Al4V (ELI) samples and NIST Si-640d reference in Lorentzian function presented in Figure 4(a). Similarly, parameters ΔK_G^m and ΔK_G^s denote the FWHM values of the LPBF Ti6Al4V (ELI) samples and NIST Si-640d reference in Gaussian function presented in Figure 4(b).

The convolution of Lorentzian and Gaussian functions using the true values obtained from Equations 6 & 7 to obtain resultant FWHM (ΔK_R) due to crystallite size and lattice strain can be evaluated from the following expression [32, 33]:

$$\Delta K_R = \frac{1}{2} \left\{ 1.0692 \Delta K_L^t + \sqrt{0.86639 (\Delta K_L^t)^2 + 4 (\Delta K_G^t)^2} \right\} \quad (8)$$

The results obtained from this expression against the diffraction vector are shown in Figure 5.

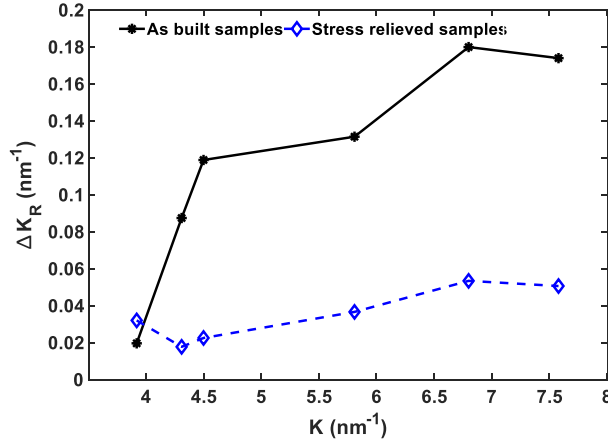


Fig. 5. The resultant ΔK_R obtained after convolution of Gaussian and Lorentzian functions against diffraction vector for as built and stress relieved Ti6Al4V(ELI) samples

From Equation 5 the values of curve fitting parameters q_1 and q_2 for the LPBF Ti6Al4V (ELI) samples were determined from a plot of the function $((\Delta K)^2 - \omega)/K^2$ against x shown in Figure 6. From the best fit of these curves in this figure, the obtained values of parameters q_1 and q_2 were -0.34 and 0.04, respectively, for the as-built samples while those of the stress-relieved samples were -0.37 and 0.05, respectively. The hcp crystal system of Ti6Al4V consists of eleven sub-slip systems while considering the three slip planes (basal, prismatic and pyramidal) and different slip directions in each plane. The values of q_1 and q_2 for each sub-slip system were numerically calculated in Dragomir and Ungar [19] and these values are well outlined in [18].

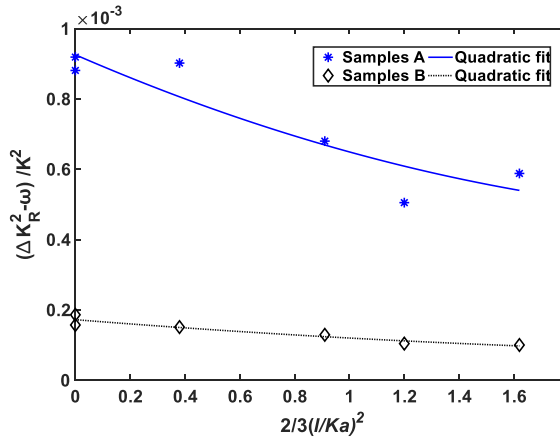


Fig. 6. A plot of $((\Delta K)^2 - \omega)/K^2$ against x for the as-built and stress-relieved LPBF Ti6Al4V (ELI) samples.

It is important to note that the MWH method assumes that all different slip systems can be activated, unless the q values obtained match the numerically calculated values for 11 different slip systems in hcp-titanium as published in [2]. The method to evaluate portions of different slip systems is summarised in [18]. The obtained values of q from Figure 6 do not match the values of the pair presented in [18, 19]. This suggests that more than one slip

system in this material is activated during deformation of these two different forms of LPBF Ti6Al4V (ELI) alloy. Thus, the method of calculating the average Burgers vector and average contrast factor used in [26, 30, 32] was adopted in this research. The method is well summarised in the authors’ work published in Muiruri *et al.* [18].

Using this method and the values of q_1 and q_2 obtained for LPBF Ti6Al4V (ELI), the fraction of type <a> dislocations, type <c> dislocations and type <c + a> dislocations, abbreviated as h_a , h_c and h_{c+a} for the two types of LPBF Ti6Al4V (ELI) samples were obtained. These fractions of dislocation population were then used to evaluate the product of the average Burgers vector and contrast factor over the three different slip systems using the following expression:

$$\overline{b^2C_{hk.0}}^{(m)}=\sum_{i=1}^3 h_i \bar{C}^{(i)} b_i^2 \tag{9}$$

where the parameters h_i represent the fractions of the dislocation population, h_a , h_c and h_{c+a} , in the material with the same Burgers vectors and $\bar{C}^{(i)}$ are averaged dislocation contrast factors over the sub-slip systems, each corresponding to the same Burgers vector type. The results obtained are presented in Table 2.

Table 2. Dislocation population and the average contrast factor for LPBF Ti6Al4V (ELI) alloy.

Samples	Dislocation population			Average Contrast factor
	<a> (h_a)	<c> (h_c)	<c+a> (h_{c+a})	$(\overline{b^2C_{hk.0}}^{(m)})$
As-built	0.8800	0	0.1200	0.0253 nm ²
Stress-relieved	0.9088	0	0.0912	0.0245 nm ²

The dislocation population were predominately <a> type with <c+a> type of dislocation being relatively low, while <c> type of dislocation were absent in the as-built and stress relieved samples of Ti6Al4V (ELI) alloy. The evaluated values of $\overline{b^2C_{hk.0}}^{(m)}$ are close to the value obtained in [30] of 0.0222 nm². The mean value obtained for $\overline{b^2C_{hk.0}}^{(m)}$ using the values obtained in Table 2 for this material is 0.0249±0.0006 nm². This mean value was used to determine the dislocation densities in as –built and stress relieved Ti6Al4V (ELI) samples using the MWA method.

3.2.2 Evaluation of dislocation density and effective cut-off radius of dislocation using the MWA method

The MWA method is of the form [33, 34]:

$$\ln A(L) = \ln A^D(L) + \ln A^S(L) \tag{10}$$

where the functions $A(L)$, $A^D(L)$ and $A^S(L)$ are the real, size and strain parts of the Fourier coefficients of the XRD peaks, respectively. When the strain Fourier coefficients are caused by dislocation and for small values of Fourier length (L), Ungar *et al.* [35] expanded Equation 10 and expressed the Fourier transform of the XRD profiles as:

$$\ln A_L = \ln A_L^D - \rho \frac{\pi}{2} L^2 \ln \left(\frac{R_e}{L} \right) K^2 \overline{b^2C} \tag{11}$$

where $\overline{b^2C}$ is the value obtained using the MWH method presented in section 3.2.1, while R_e and ρ denote the effective cut-off radius of dislocation and the dislocation density, respectively. From Equation 11 the dislocation density can be determined from a plot of $\ln A_L$ vs. $K^2 \overline{b^2C}$ such that the gradient of the resulting curves is $Y = \rho \frac{\pi}{2} L^2 \ln \left(\frac{R_e}{L} \right)$. The formulation of the gradient can further be expressed as:

$$\frac{Y}{L^2} = \rho \frac{\pi}{2} \ln R_e - \rho \frac{\pi}{2} \ln L \quad (12)$$

The real part of Fourier coefficient (A_L) of the XRD peak profiles can be evaluated from the FWHM of Gaussian and Lorentzian fitting using the following equation [32,34]:

$$A_L = \exp - \left\{ \left(\frac{1}{2} L^2 (\Delta K_G^t)^2 \right) + L \Delta K_L^t \right\} \quad (13)$$

A plot of $\ln A_L$ against $K^2 \overline{b^2C}$ of the as-built and stress relieved LPBF Ti6Al4V (ELI) samples for a range of values of Fourier length is shown in Figure 7.

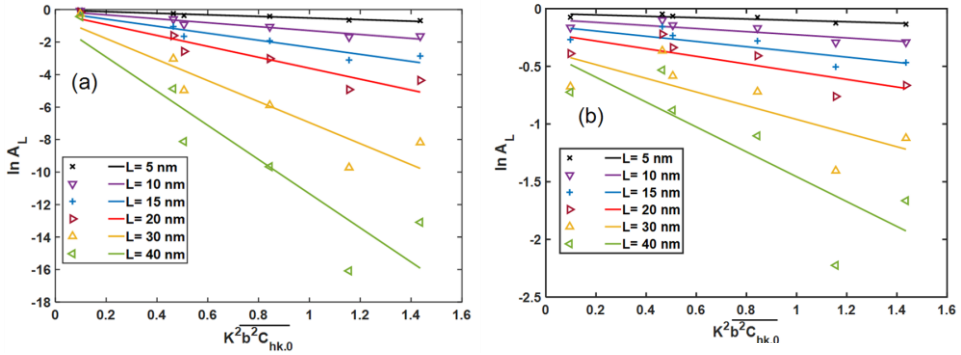


Fig. 7. The plot of $\ln A_L$ against $K^2 \overline{b^2C}$ of (a) as-built and (b) stress relieved LPBF Ti6Al4V (ELI) samples

The plot of Equation 12 from the data obtained from Figure 7 is shown in Figure 8.

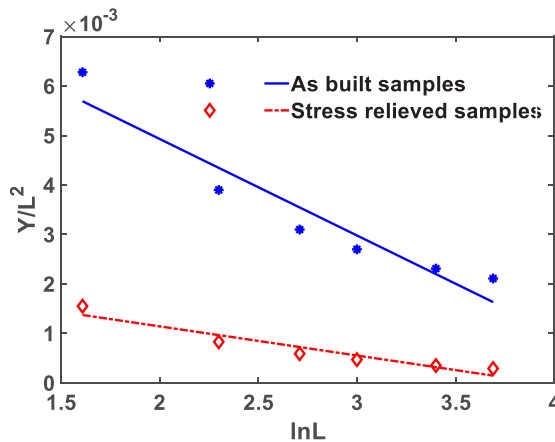


Fig. 8. The curves of $\frac{Y}{L^2}$ against $\ln L$ of as-built and stress relieved LPBF Ti6Al4V(ELI) samples

The dislocation densities and effective cut-off radius of dislocations in the as-built and stress relieved samples of LPBF Ti6Al4V (ELI) were obtained from the gradient and the y-intercept of the curves in Figure 8, respectively, and are presented in Table 3.

Table 3. The dislocation density and effective cut-off radius of dislocations in LPBF Ti6Al4V samples

	Effective cut-off radius of dislocation (R_e) (nm)	Dislocation density (m^{-2})
As-built samples	93.06	3.82×10^{15}
Stress-relieved samples	70.02	1.02×10^{15}

The as-built LPBF Ti6Al4V (ELI) parts possess a high level of dislocation density, as seen in this table, which is thought to be due to lattice distortion and a high level of residual stress as a result of rapid cooling during the manufacturing process. Upon stress-relieving heat treatment, the level of dislocation density drastically reduces by almost 73%, clearly indicating the positive response of the heat treatment process in alleviating the residual stresses and attendant distortions. A comparison of the dislocation density in LPBF Ti6Al4V (ELI) obtained from this study and that of the same alloy produced by the EBPBF process is shown in Table 4.

Table 4. A comparison of dislocation density in LPBF and EBPBF Ti6Al4V (ELI) parts

State	PBF process	Diffraction source	Dislocation density (m^{-2})	Reference
As-built	EBPBF	Neutron	6.8×10^{13}	[15]
As-built	EBPBF	Synchrotron radiation	6.1×10^{13}	[15]
As-built	EBPBF	X-ray	7.5×10^{13}	[15]
Annealed (480 °C for 30 h	LPBF	X-ray	0.558×10^{14}	[36]
As-built	LPBF	X-ray	3.82×10^{15}	This study
Stress relieved	LPBF	X-ray	1.02×10^{15}	This study

The results presented in Table 4 show that the dislocation density in as-built and stress-relieved LPBF Ti6Al4V (ELI) parts is relatively higher than that in a similar as-built alloy produced by EBPBF. This suggests that dislocation strengthening is evidently higher for parts produced by LPBF as compared to those manufactured by EBPBF. The observations made in Table 4 were attributed to the difference in level of residual stress and microstructure for parts produced by these two technologies. As previously described in the introduction section, while LPBF is associated with rapid cooling from a temperature way above the β -transus temperature to room conditions, the EBPBF parts are kept at a temperature of about 700 °C before being cooled to room temperature. Therefore, a high level of dislocation density is anticipated in LPBF parts, as compared to EBPBF parts as is observed in Table 4. Furthermore, the LPBF process leads to the formation of a fine acicular (α') martensitic microstructure, while the EBPBF process leads to the formation of a bi-phasic ($\alpha + \beta$) microstructure, of the Ti6Al4V alloy. The fineness of the α' martensitic microstructure suggests a large network of grain boundaries, which are dislocations in nature, and therefore a high density of dislocations, as observed in Table 4.

As shown in Table 4, Ter Haar [36] obtained lower values of dislocation density for LPBF parts annealed at a temperature of 480 °C for 30 hours as compared to stress relieved samples in this study that were annealed at a temperature of 650 °C for 3 hours. The possible reason

for the different results in these two studies is the different methods used to evaluate dislocation densities in LPBF parts. Ter Haar [36] applied the simple Williamson and Smallman (WS) [37] method to evaluate dislocation density using the micro lattice strain and the average crystal size while in this study the MWH and MWA methods were used. As discussed in [1,2,19,37] the WS method is problematic in quantitative peak profile analysis for determining broadening anisotropy and dislocation contrast factors that are particularly important in calculating dislocation density in a polycrystalline material.

4 Conclusions

The XRD peak profiles of as-built and stress-relieved LPBF Ti6Al4V (ELI) were analysed in this study to evaluate the dislocation densities in these two forms of the alloy. The following conclusions were deduced from this study:

- There was no evidence of β -phase in both as-built and stress-relieved samples of LPBF Ti6Al4V (ELI) from the XRD data obtained in this study.
- The Bragg peak reflections of the as-built samples had a larger integral breadth as compared to those of the stress-relieved samples.
- The recorded values of dislocation density in as-built and stress-relieved LPBF Ti6Al4V(ELI) were $3.82 \times 10^{15} \text{ m}^{-2}$ and $1.02 \times 10^{15} \text{ m}^{-2}$, respectively.
- The dislocation densities in LPBF parts are higher compared to those of EBPBF.

The future research aims at analysing the dislocation densities in pre-strained LPBF parts using XRD and other advanced techniques such as transmission electron microscopy (TEM).

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