Development of strength-hardness relationships in additively manufactured titanium alloys

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Abstract

A major concern for additively manufactured (AM) Ti-6Al-4V components is how AM processing parameters and post-process heat treatments impact the resulting mechanical behavior. The applicability of using microhardness measurements as a predictive tool for yield and tensile strengths of AM Ti-6Al-4V would provide a rapid and useful screening mechanism for ensuring that properties meet requirements in complex geometries. However, microhardness measurements on Ti-6Al-4V exhibit high levels of data variability due to the orientational impact of the α phase. In order to overcome this variability in hardness measurements, a methodology

for aggregating microhardness data in individual builds has been developed and validated. By compiling mean microhardness values from various AM components produced by electron beam based directed energy deposition (DED), laser based DED, and laser based powder bed fusion (PBF) processes in the as-deposited and post-process heat treated conditions, strong linear correlations between strength and hardness can be developed in AM materials having a lamellar $\alpha + \beta$ microstructure. With the addition of strain hardening and α phase orientation contributions to the mean microhardness measurement, the strength-hardness correlations of AM Ti-6Al-4V followed empirically derived models, opening the possibility of using these models to predict strengths from AM components regardless of the AM process or post-process state.

Keywords: Additive manufacturing; mechanical characterization; titanium alloys; microhardness; tensile testing

1. Introduction

There are various techniques currently being used to additively manufacture (AM) Ti-6Al-4V components. Differences in the processing conditions and structures produced at different locations result in a wide variation in mechanical properties [1, 2]. For example, tensile strengths in the as-deposited condition of AM Ti-6Al-4V reported in the literature vary from 775 MPa to 1270 MPa [3]. Much of this variation can be attributed to differences within the processing conditions, such as the selection of the energy source (laser, electron beam, and arc), the power input, travel speed, and feedstock form, such as wire or powder. Location and orientation dependent anisotropy in the tensile properties has also been observed in relation to the build

direction [3-11], arising from differing thermal histories experienced at each location [12, 13]. In most cases, post-process heat treatments and hot isostatic pressing (HIP) operations are used and shown to significantly alter the as-deposited Ti-6Al-4V structure as well as the resulting tensile properties [5, 10, 14-19].

In order for AM fabricated components to be used for critical applications, a qualification and certification protocol is needed. A major component of this protocol is to understand how processing conditions, part design, build path planning, and post-process heat treating, among others, will impact the resulting mechanical properties [20, 21]. It is costly and impracticable during production to extract specimens from different locations and orientations in AM components to investigate how processing and design changes will impact the local mechanical properties. Microstructural and phase composition has also been used to predict the mechanical properties [22-24] and toughness [25] for Ti-6Al-4V components, but using these methods requires a detailed microstructural and chemistry composition analysis. In contrast, hardness measurements offer a fast and inexpensive method of measuring material properties at specific locations. A good understanding of the relationship between measured hardness values and the mechanical properties would allow for hardness to be used as a predictive tool for mechanical properties and help streamline the qualification and inspection protocols for AM Ti-6Al-4V components.

Although hardness is widely used as a predictor of strength for steels and other common alloy systems [26, 27], similar relationships are not as widely available for titanium alloys. Currently, the only available strength to hardness correlation for Ti-6Al-4V is an empirical relationship, developed by Hickey [28], based on a linear least square fit of the Vickers hardness, H_V , and tensile strength, σ_{UTS} , for investment cast Ti-6Al-4V components where:

$$\sigma_{UTS}(\text{MPa}) = \frac{H_V(\text{MPa})}{6.33} + 503 \quad (1)$$

A cursory correlation between hardness and build strength was attempted for AM Ti-6Al-4V wall structures produced by a laser based directed energy deposition (DED) process [3]. The tensile strengths were found to decrease with increasing height above the build substrate.

Hardness measurements made across the height of a wall structure produced using the same processing conditions are shown in Fig. 1. These hardness measurements, though, did not show a significant correlation with the build height, but, rather, exhibited a large spread with no observable trend.

The problem with using hardness as a predictive tool for Ti-6Al-4V structures is that the measurement is impacted by the orientation of the underlying hexagonal closed packed (HCP) α phase. With the limited slip systems available in the HCP system, the energy required for slip varies with the crystallographic orientation in relation to the force direction [29]. Slip preferentially occurs on the closest packed planes, and, for HCP systems, the planes exhibiting the highest packing density are governed by the c/a lattice parameter ratio. For titanium alloys, the c/a ratio of the α phase is less than the ideal packing ratio (1.633), and the prism planes ($\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ planes) will exhibit a higher packing density than the basal plane ($\{0001\}$) [30]. Thus, slip is expected to preferentially occur on the prism planes, and this has been observed experimentally for α and $\alpha + \beta$ titanium alloys [29, 31]. Impurity atoms such as oxygen and nitrogen also influence the slip mode and can make the pyramidal planes the preferred slip system [32, 33]. Regardless of whether slip preferentially occurs on the pyramidal or prism planes, the basal planes are the least preferential slip planes. Hardness measurements obtained from α grains oriented with the basal plane normal to the measurement direction for α and $\alpha + \beta$

titanium alloys are significantly higher than those obtained for other crystallographic orientations [29, 34-37]. Viswanathan et al. experimentally found that the hardness obtained by nanoindentation on α grains presenting the basal plane exhibited 50% higher hardness than α grains presenting the prism planes [34].

The variability introduced by the underlying α grain orientation on the hardness measurement is a major contributor to the wide measurement spread observed in AM Ti-6Al-4V components. In Ti-6Al-4V, the structure consists primarily of α phase ($> 85\%$ for wrought [38, 39] and $> 90\%$ for AM Ti-6Al-4V [18, 40, 41]), and this phase would be expected to dominate the hardness measurements. Fig. 2(a) shows a Vickers microhardness indent made with a 1 kg_f load within an AM Ti-6Al-4V microstructure that was fabricated using an electron beam based DED process. Even though this single hardness indent encompasses a number of α lathes, the crystallographic orientation of the underlying α phase could vary significantly from one hardness measurement location to another resulting in significantly different hardness measurements within the same area. For example, in Fig. 2(b), the hardness varied from 2.85 to 3.41 GPa within a 1.5 mm² area for an AM electron beam based DED Ti-6Al-4V microstructure where the orientational differences of the α phase are highlighted using polarized light.

The unknown impact of the underlying microstructure on the resulting hardness makes correlating the measured hardness and strength problematic. In order to obtain a hardness to strength relationship over a wide range of processing and post-processing conditions, this investigation compiled room temperature microhardness and strength measurements from AM Ti-6Al-4V components fabricated by laser based DED with a powder feedstock, electron beam based DED with a wire feedstock, and laser based powder bed fusion (PBF) AM processes in the as-deposited and post-process heat treated conditions. A methodology for aggregating hardness

data obtained for each processing condition was developed. The mean microhardness was then correlated to the strengths for analyzing the strength to hardness relationships. As part of the methodology for aggregating the hardness data, a specific number of Vickers microhardness measurements were made at locations corresponding to those where static mechanical testing specimens were extracted. It is shown that when these statistical variations are taken into account, hardness measurements can be used to predict strength in AM Ti-6Al-4V regardless of the fabrication technology or post-processing conditions.

2. Background

Hardness measurements have been widely used as a predictive tool to estimate the yield and tensile strengths of Ni, Fe, Cu, and Al based alloy systems [26, 27, 42] as well as for nanocrystalline metal systems and metallic glasses [43, 44]. Even across these different metallic systems, a general relationship for correlating yields strength, σ_y , and the hardness, H , follows:

$$\sigma_y \approx \frac{H}{3} \quad (2)$$

This correlation, however, is only valid for metallic materials that exhibit minimal strain hardening. If the material exhibits strain hardening, then the hardness measurement itself induces strain hardening, and the resulting hardness measurement is representative of the strain hardened material and not the material prior to the measurement [27].

For metals that strain harden, theoretical equations have been derived for equating the tensile and yield strengths to the hardness for steels, nickel, aluminum, and copper alloys. Tabor [27] observed that the strain induced by a Vickers indenter is between 8 and 10%, and the equivalent stress at this strain was approximately $H_V/2.9$ for steels and $H_V/3$ for copper alloys. If a metal

exhibits power-law strain hardening, the true stress, σ_t , as a function of true strain, ε , can be expressed as:

$$\sigma_t = K\varepsilon^n \quad (3)$$

where K is the strength coefficient of the material and n is the strain hardening exponent. Using the approximate stress observed for steels and copper alloys at a strain of 8%, Tabor derived a relationship equating the tensile stress, σ_{UTS} , to the Vickers hardness. This relationship was further improved and simplified by Cahoon as follows [45]:

$$\sigma_{UTS} = \left(\frac{H_V}{2.9}\right) \left(\frac{n}{0.217}\right)^n \quad (4)$$

Cahoon et al. [26] also derived a relationship between the 0.2% offset yield strength, σ_y , and the Vickers hardness for a metal that exhibits power-law strain hardening behavior. From Eq. (3), K can be solved by assuming that $H_V/3$ is the stress at a strain of 8%, and the yield strength to Vickers hardness relationship can be described as follows:

$$\sigma_y = \left(\frac{H_V}{3}\right) \left(\frac{\varepsilon_y}{0.08}\right)^n \quad (5)$$

where ε_y is the true strain at the 0.2% offset yield strength. Cahoon et al. empirically observed that ε_y was approximately 0.008 for both aluminum and steel samples [26]. Assuming that ε_y can be treated as a constant for all metals, the yield strength can be equated to the Vickers hardness using the following relationship [26]:

$$\sigma_y = \left(\frac{H_V}{3}\right) (0.1)^n \quad (6)$$

However, the strain hardening exponent, n , may not be known for a particular material and obtaining n typically requires direct measurement through tensile testing. Thus, using these

relationships as a predictive tool may not be practicable for AM processes where the strain hardening behavior is not well characterized. Instead of relying on relationships empirically derived using n , a linear correlation between the strength and hardness can also be used as a predictive tool when the strain hardening behavior is unknown. Pavlina and Van Tyne [46] show that, despite varying strain hardening behaviors for various types of steels, the Vickers hardness still exhibits a strong linear correlation with the resulting tensile and yield strengths. However, the strain hardening does have an impact on the strength to hardness relationships, and the predicted strengths tend to be lower than the observed strengths for steels exhibiting a large amount of strain hardening [46].

3. Experimental

Strength and hardness data were collected from AM Ti-6Al-4V components processed by electron beam based DED, laser based DED with two different conditions, and laser based PBF. Components produced by the electron beam based DED process were fabricated using an electron beam AM (EBAM) system (Sciaky, Inc.) in a vacuum environment at pressures below 16 mPa. The feedstock was a 1.6 mm diameter prealloyed Ti-6Al-4V wire (Astrolite Alloys) that was fed into the molten pool at a rate of 95 mm s^{-1} . The working distance between the gun and the workpiece was 248 mm, and the beam was oscillating in a circular pattern at a rate of 1 kHz. Two sets of components were fabricated using laser based DED with different processing conditions. The first set of laser DED builds (2 kW laser based DED) were fabricated using an IPG Photonics® YLR-12000 laser system operating at a laser power of 2 kW. Deposition was conducted within an atmospherically controlled chamber purged with ultra-high purity argon gas, and oxygen levels were maintained below 100 ppm. The laser was delivered through a 200 μm diameter fiber to a Precitec® YW-50 laser optic that uses a 200 mm collimator and a 200 mm

focal length lens. Grade 5, Ti-6Al-4V powder (TIMET Powder Metals, LLC) with a mean diameter of 100 μm was fed through a Precitec® YC 50 cladding head positioned 10 mm above the substrate. Further details on the processing parameters are elsewhere [3].

L-shape wall structures were fabricated for both the electron beam and 2 kW laser based DED builds. The number of passes used to form the wall structures was varied depending on the desired final thickness of the wall structures. A single pass was used for thin wall structures, and three passes were used for thick wall structures. To maintain a consistent wall height during fabrication, the laser was moved in the opposite direction for each successive layer. Fig. 3 shows a representative schematic of an L-shape wall structure fabricated by the electron beam and 2 kW laser based DED processes. Round tensile samples extracted from the DED wall structures were proportionally sized to meet the requirements set forth in ASTM E8 [47]. Tensile samples extracted from the 2 kW laser based DED builds and the thin wall electron beam based DED builds were machined to a gage diameter of 1.54 ± 0.04 mm with a gage length of 6.35 mm. Tensile samples extracted from the thick wall electron beam based DED builds were machined to a gage diameter of 4.0 ± 0.01 mm with a gage length of 16.0 mm. Microhardness measurements were made on the metallographic samples at locations that corresponded to the general locations of the tensile testing specimens. The geometrical orientation of the surface of the metallographic samples that was polished and used for microhardness measurements in relation to the build is highlighted in the inset figure of Fig. 3.

Additional simple wall structures were fabricated using a 0.5 kW laser based DED on a LENS® MR-7 system (Optomec) with a ytterbium-doped fiber laser (IPG YLR-500-SM). Details on the system can be found elsewhere [48]. The processing chamber was purged with high purity argon gas, and oxygen levels were maintained below 20 ppm. The substrate was positioned 9.37 mm

below the powder delivery nozzles. The powder used for deposition was Grade 5, Ti-6Al-4V (Phelly Materials, Inc.), with a mean particle size of 126.8 μm . The measured output of the laser was $460 \pm 25\text{W}$ with a processing speed of 10.6 mm s^{-1} . Wall structures fabricated using a single pass and the laser moved in the opposite direction for each successive deposition layer to help maintain a consistent wall height. The wall structures were built to a length of $50 \pm 0.3\text{ mm}$, a height of $51 \pm 0.5\text{ mm}$, and exhibited a wall thickness of $2.5 \pm 0.3\text{ mm}$. Rectangular tensile samples were extracted from the wall structures and proportionally sized to meet the requirements set forth in ASTM E8 [47] with a gage length of 8.4 mm, a thickness of $1.23 \pm 0.02\text{ mm}$, and a width of $2.12 \pm 0.02\text{ mm}$. Microhardness measurements were made on metallographic samples that were extracted near the grip ends of the tensile samples.

Test coupons were also fabricated using a laser based PBF process on an EOSINT M 280 machine (EOS GmbH Electro Optical Systems). The Ti-6Al-4V powder (EOS Titanium Ti64) had a mean diameter of 45 μm , and components were built in an argon atmosphere with a nominal beam power of 340 W and a scanning speed of 1250 mm s^{-1} . Round geometry tensile samples were machined from the test coupons to the requirements set forth in the ASTM E8 [47] with a gage diameter of 4.0 mm and a gage length of 16.0 mm. Microhardness measurements were made on metallographic samples that were obtained adjacent to the grip ends of the tensile samples.

The components analyzed in this investigation were tested in the as-deposited condition and after post-process heat treating that included stress relieving, annealing, and hot isostatic pressing (HIP) using the heat treating schedules defined in the AMS 4999A specification [49]. Annealing was performed at 920°C for 4 h within an argon atmosphere, HIP was performed at 900°C for 2

h at 100 MPa within an argon atmosphere, and stress relieving was performed in a vacuum furnace at 650°C for 4 h.

Uniaxial tensile testing was conducted according to the ASTM E8 standard [47] at a certified testing laboratory (Westmoreland Mechanical Testing & Research, Inc., Youngstown, PA). All tensile samples were tested at a strain rate of 0.005 min^{-1} . The 0.2% offset yield strength and tensile strength were obtained from the resulting stress-strain curves. The strain hardening exponent, n , was also estimated from the stress-strain curves following the recommendations outlined in ASTM E646 [50]. There was a large uncertainty for the measured n from the DED tensile tests since the extensometer was removed after the 0.2% offset yield strength value was obtained and the remaining plastic deformation region in the engineering stress-strain curves was estimated from the crosshead position of the tensile testing machine. Using the crosshead position instead of an extensometer increased the strain measurement uncertainty and this uncertainty was accounted for within the reported standard deviation. During testing of the PBF samples, the extensometer remained on the sample through failure and a complete stress-strain curve was obtained.

Vickers microhardness measurements were made on polished metallographic samples that were extracted from each of the AM builds. All microhardness measurements were made using a LECO-M-400-G1 hardness tester with a 1 kg_f load that was applied for 10 s. Calibration was conducted with a steel Vickers reference hardness block with a certified mean Vickers microhardness value of 354.7 HV with an uncertainty of 1.77 HV. Although Vickers microhardness values are typically reported as a Vickers pyramid number (HV) with units of $\text{kg}_f \text{ mm}^{-2}$, hardness values here were reported in units of MPa to help develop the strength-hardness correlations (note: $1 \text{ MPa} = 0.10197 \text{ kg}_f \text{ mm}^{-2}$). In addition, all microhardness measurements on

the extracted metallographic samples were made on a surface corresponding to the x- and z-axes or the y- and z-axes for a coordinate system defined for AM in ASTM 52921 [51].

The microstructures of the AM components were characterized from selected metallographic samples at the same locations where hardness measurements were taken. Preparation of these specimens was completed first by polishing using SiC grinding paper up to P2000 ISO grit size with final polishing conducted using a two-step process with a 3 μm diamond slurry followed by 0.05 μm colloidal silica suspension. The polished surface was etched with Kroll's reagent (2% hydrofluoric acid and 3% nitric acid in water) in order to expose the microstructures in the Ti-6Al-4V samples extracted from each build. The microstructures were captured on a Nikon Epiphot optical microscope.

4. Results and discussion

4.1. Identification of strength to hardness relationships

It has been shown in previous studies that changes in wall thickness as well as the addition of a post-processing HIP cycle have a significant impact on the resulting tensile and yield strengths of wall structures fabricated using a laser based DED process [3, 52]. Fig. 4 shows a categorical plot of the yield and tensile strengths obtained at locations across the build heights of the thin and thick wall structures fabricated by a 2 kW laser based DED process. The as-deposited thick wall build exhibited slightly higher strengths than the as-deposited thin wall build [3]. The tensile properties obtained from the thick wall builds also exhibited location dependencies where the tensile strength decreased with height [3] resulting in a higher data variability than observed for the thin wall build. After the addition of a post processing HIP cycle, the strengths dropped significantly for both thick and thin wall structures [52]. In contrast to the as-deposited builds,

the thick wall builds that underwent HIP exhibited lower strengths than the thin wall builds that underwent HIP.

The lower tensile properties of the thick wall structures after HIP may be the result of significant changes in the dominant strengthening mechanisms compared to the as-deposited condition. In the as-deposited condition, the thick wall builds may have exhibited a higher level of solid solution strengthening than compared to the thin wall builds [3]. After HIP, both the thick and thin walls appeared to exhibit a similar microstructure which implied that they both exhibited a similar level of solid solution strengthening. The thin wall builds, however, exhibited significantly higher amount of prior β grain intercepts than the thick wall builds [3] and the size and morphology of the prior β grains is expected to remain the same after post processing heat treating below the β transus temperature ($\approx 995^{\circ}\text{C}$) [18]. Therefore, boundary strengthening (Hall-Petch strengthening) may have become a more dominate strengthening factor for the builds that underwent HIP resulting in the higher strengths observed in the thin wall structures as compared to the thick wall structures.

A series of Vickers microhardness measurements were also made at locations that generally corresponded to where mechanical properties were obtained across the height and length of these same wall structures. Histograms of the microhardness measurements obtained from each wall structure are shown in Fig. 5. There was a large spread in the hardness data with significant overlap between the wall structures. The data also demonstrated differing means between the wall structures, indicating that there were potential relationships between the mean hardness values and the different processing and design conditions. For example, the as-deposited thick wall structure exhibited a higher mean hardness than the other wall structures, and this higher hardness mirrored the higher strengths observed in this structure. In addition, the microhardness

data sets generally followed a normal distribution making it possible to analyze these distributions in more detail.

A one-way analysis of variance (ANOVA) was conducted at the 0.05 level of significance to determine if the means of the microhardness measurements between the wall structures were statistically different. The results of the ANOVA are listed in Table 1. The mean microhardness values were statistically different for the wall structures, as confirmed by the calculated p-value of less than 0.001. Although the ANOVA results showed a statistically significant difference among the means, an ANOVA does not identify which of the builds exhibited a statistically different mean microhardness. Therefore, a Tukey pairwise mean comparison was conducted to determine which wall structures exhibited statistically different means to the other wall structures by analyzing the 95% confidence interval of the means. The Tukey pairwise comparison is listed in Table 2. In this analysis, the confidence intervals of the means did not overlap, indicating that each wall structure exhibited a statistically different mean microhardness.

Since there were statistically significant differences between the mean microhardness measurements for each wall structure, the measured microhardness means were further correlated with the tensile and yield strengths. Fig. 6 shows the resulting mean yield and tensile strengths plotted as a function of the mean microhardness measurements for these four wall structures. The as-deposited thick wall build exhibited the highest strengths and the highest mean microhardness values among the builds. With the addition of a post-process HIP cycle, the thick wall build exhibited the lowest strengths and lowest mean microhardness values. Overall, a strong correlation between both the yield and tensile strengths and the mean microhardness measurements were observed across all of these wall structures.

Although there appeared to be a strong correlation between hardness and strength, the impact of the variability of the microhardness measurements compared to the variability observed from the tensile testing was analyzed to confirm that these relationships were statistically significant. This measurement variability impacts the capability of hardness measurements to predict strengths. To analyze how the hardness measurement variability compared to that obtained from tensile testing, the coefficients of variation were compared. The coefficient of variation is a normalized value describing data variability and it allows direct comparison of data variability across different measurement techniques. The coefficient of variation is calculated by dividing the standard deviation by the mean. Fig. 7 compares the coefficients of variation obtained from the four wall structures for the Vickers microhardness, yield strength, and tensile strength data. The coefficients of variation obtained from the microhardness data was similar to both the tensile and yield strength data, indicating that microhardness measurements exhibited a similar capability as tensile testing for identifying significant mechanical behavior differences between AM Ti-6Al-4V components.

The similar coefficients of variations highlighted the advantage of the relatively simple microhardness measurement procedure compared to the complexity of tensile testing. The material volume impacting the measurement is much smaller for the microhardness tests compared to tensile testing thus making the microhardness tests more susceptible to measurement variation from the impact of the α grains. The sources of measurement error for microhardness measurements, however, is much smaller than for tensile testing. Microhardness measurement errors may come from measurement of the indentation and from the applied load. Measurement errors from tensile testing can arise from various sources including the load cell, extensometer, calculation of the 0.2% offset, measurement of the sample dimensions, and the

dimensional accuracy of the machined tensile specimens. In addition, AM components may exhibit undetected internal defects (gas entrapment or lack-of-fusion porosity) that could also impact the resulting data variations observed for the strengths [17, 53, 54].

From the correlations of the microhardness with the yield and tensile strengths, the hardness measurements appeared to show general relationships between both component design (thin and thick wall) and processing conditions (as-deposited and HIP). The strong observed correlation of the mean microhardness measurements to the strengths opened the possibility of using microhardness measurements as a predictive tool for understanding how processing and design changes would impact the resulting strengths for AM Ti-6Al-4V components. This analysis was then extended to other AM components produced using both electron beam and other laser based DED processing techniques with different processing conditions in both the as-deposited and post-processed conditions.

4.2. Development of strength-hardness relationships

As part of extending the strength to hardness correlation to other AM processes and post process conditions, an analysis was conducted on the variance of the microhardness measurements. The resulting measurement variability of the microhardness measurements is not only impacted by general measurement errors, but also by the underlying orientation of the α phase that changes from one measurement location to the next. This measurement variability impacts the confidence in the measured mean for a given number of measurements. As detailed in Supplementary A, the microhardness measurements on AM Ti-6Al-4V exhibited a relatively high variability resulting in a large margin of error for the mean microhardness value obtained from just a few measurements. By taking 25 individual microhardness measurements within a region of interest, however, the margin of error was reduced to below 40 MPa ($4.1 \text{ kg}_f \text{ mm}^{-2}$) with a 95%

confidence level. To develop strength to hardness relationships for AM Ti-6Al-4V components, the mean microhardness value obtained from at least 25 individual measurements within a selected metallographic regions were compiled and correlated to the yield and tensile strengths obtained from tensile samples extracted at corresponding locations.

4.2.1. Strength-hardness correlation for DED components

The AM DED components analyzed in this investigation were made from various AM processing techniques and tested in the as-deposited and post-processed conditions. These different processing and post-process conditions were expected to produce varying microstructures and a wide range of mechanical properties. Fig. 8 shows representative optical micrographs obtained from builds produced by both laser and electron beam based DED in the as-deposited and HIP conditions. The microstructures exhibited a lamellar $\alpha + \beta$ structure where the α phase (light phase) was outlined by the more darkly etched β phase. Generally, all the builds exhibited similar microstructure features but the scale of these microstructure features varied depending on the AM process and the post-process conditions. Builds produced by the electron beam based DED process exhibited a coarser microstructure compared to builds produced by laser based DED with different power levels. After post-process heat treating by either HIP or annealing, all of the builds, regardless of the AM process, exhibited a coarser lamellar microstructure compared to the as-deposited condition.

Fig. 9(a) and Fig. 10(a) shows plots of the yield and tensile strengths as a function of the mean microhardness where the DED process and post process condition are represented by various markers. Hardness increased with both yield and tensile strengths regardless of the AM process or the post-process condition. Using a wide range of DED processes and post-processing

conditions allowed the strength-hardness relationships to be analyzed over a relatively large microhardness range from 2960 to 3790 MPa (302 to 386 kg_f mm⁻²).

To help analyze the strength-hardness relationships, a linear regression following the least squares criterion was conducted. In this analysis, the mean hardness measurements were considered as the predictor variable, and both the yield and tensile strengths were regarded as the response variable. A least-square linear regression of the data gave a correlation between the yield strength, σ_y , and the tensile strength, σ_{UTS} , to the mean Vickers microhardness, H_V , as:

$$\sigma_y(\text{MPa}) = \frac{H_V(\text{MPa})}{3.60} - 90 \quad (7)$$

$$\sigma_{UTS}(\text{MPa}) = \frac{H_V(\text{MPa})}{3.34} - 56 \quad (8)$$

From the linear regression analysis, there was a statistically significant relationship between the mean Vickers microhardness measurements and the yield strength ($F(1,190) = 652.9$, $p < 0.001$) and tensile strength ($F(1,190) = 846.2$, $p < 0.001$). In addition, the R^2 statistics indicated that 77% and 82% of the data variations could be explained by the regression equations for the yield and tensile strength correlations, respectively. The linear regression equations are plotted as solid lines in Fig. 9(a) and Fig. 10(a), and the dashed lines represent the standard deviation range for the regression. The corresponding residuals for the yield strength and tensile strength regressions, defined as the difference between the predicted strength value from the regression equation and the observed strength value, are plotted in Fig. 9(b) and Fig. 10(b). These residual values appeared to be normally distributed further indicating that the strength-hardness correlations followed a linear trend throughout the hardness range analyzed.

4.2.2. Comparison with empirically derived models

The empirically derived strength to Vickers hardness correlations listed in Eq. (4) and (6) exhibit good agreement with Ni, Fe, Cu, and Al alloys [26, 27, 42]. These correlations, however, require an understanding of the strain hardening behavior for the alloy. For the AM DED Ti-6Al-4V components, the strain hardening exponent, n , was estimated from selected stress to cross-head position curves obtained from tensile testing of samples extracted from components produced by both laser based and electron beam based DED in the as-deposited state and after post-process heat treating. Regardless of the AM process or post-process condition, n remained relatively consistent, with an estimated value of 0.07 ± 0.02 . The estimated value indicated that the AM DED Ti-6Al-4V exhibited minimal strain hardening that is expected for Ti-6Al-4V [32, 33].

Fig. 11(a) shows the yield strengths and Fig. 11(b) shows the tensile strengths plotted against the corresponding mean Vickers microhardness from the DED samples along with the empirically derived models of Eq. (4) and (6) [26, 27, 45] for $n = 0, 0.05, 0.07$ and 0.1 . For a given yield or tensile strength, the hardness values measured were higher than predicted by Eq. (4) and (6) with $n < 0.1$. The data trend, however, seemed to follow a similar slope expected from the empirical models with n between 0.05 and 0.1 .

The higher than expected hardness values could be the direct result of the limited slip systems inherent within the α phase of Ti-6Al-4V. The energy required for slip on the basal planes is higher than that required for slip on either the pyramidal and prism planes [29, 34-37]. Hardness indents made at locations dominated by basal slip would result in a higher hardness measurement than those made at locations dominated by pyramidal or prism slip. The higher hardness measurements at basal dominated locations would effectively increase the overall mean hardness value. Therefore, the empirical models listed in Eq. (4) and (6) may still apply to AM Ti-6Al-4V

if the orientational contribution of the α phase on the hardness is taken into account by dividing the measured hardness by an orientational impact factor, defined here as A , as follows:

$$\sigma_y = \left(\frac{H_V}{A \cdot 3} \right) (0.1)^n \quad (9)$$

$$\sigma_{UTS} = \left(\frac{H_V}{A \cdot 2.9} \right) \left(\frac{n}{0.217} \right)^n \quad (10)$$

The orientational impact factor, A , for the DED AM samples was estimated at 1.12 from a linear least square fit of the data to Eq. (9) and (10) using n obtained from tensile testing ($n = 0.07$).

This value implied that the hardness of the AM Ti-6Al-4V was roughly 12% higher than predicted following the empirical models listed in Eq. (4) and (6). Furthermore, the calculated R^2 statistics from for the linear least square fits indicated that 81% and 76% of the data variations could be explained by Eq. (9) and (10), respectively. These R^2 statistics were similar to that obtained from the linear regression analysis. The high R^2 statistics indicated that both the orientational impact of the α phase and the strain hardening behavior appeared to capture the main variable factors for the strength to hardness correlations for AM DED Ti-6Al-4V.

Furthermore, the use of the modified empirically derived models in Eq. (9) and (10) allows for the analysis of AM Ti-6Al-4V components that may fall outside the valid range for the linear regression equations.

4.3. Strength to hardness for PBF

Mean hardness measurements were also correlated to the strengths for samples produced by a laser based AM PBF process after stress relieving and HIP post-processing. Fig. 12 shows yield and tensile strengths from the individual PBF samples plotted against the corresponding mean microhardness values alongside the DED data. For both the tensile and yield strengths, the stress relieved PBF exhibited a significantly different behavior compared to the DED samples. In this

plot, the observed strengths for the PBF samples were higher than expected from the empirically derived correlations for the DED process. The HIP samples, however, exhibited a strength to hardness correlation that appeared to be similar to that observed for the DED samples.

The different behavior of the PBF samples that underwent stress relieving may be the result of different microstructure and strain hardening behavior. Fig. 13 shows representative microstructures of the PBF samples after stress relieving and after HIP. The rapid cooling of the laser based PBF process typically results in a martensitic microstructure consisting of a fine acicular lath structure [55-57] and these laths are considerably finer than the lamellar structure observed in the DED builds. The microstructure of the PBF samples still exhibited a martensitic structure after stress relieving. The martensitic structure may exhibit a significantly different orientational impact on the resulting hardness than compared to the lamellar $\alpha + \beta$ microstructure. After HIP, the microstructure of the PBF samples exhibits a similar lamellar $\alpha + \beta$ structure to that observed in the as-deposited and post-process heat treated DED samples.

The strain hardening exponent, n , was measured from the PBF samples. For the stress relieved PBF samples, n was measured at 0.032 ± 0.001 . The samples that underwent HIP, n measured higher at 0.052 ± 0.001 . This difference in strain hardening behavior can be seen in the resulting stress-strain curves obtained from selected tensile samples shown in Fig. 14. In this figure, the stress relieved PBF samples followed more closely to a material that exhibits no strain hardening where the yield strength (averaging 1050 ± 17 MPa) was relatively close at $94.0 \pm 0.01\%$ of the tensile strength (averaging 1116 ± 19 MPa). The samples that underwent HIP exhibited a larger difference with the yield strength (846 ± 12 MPa) at $89.1 \pm 0.01\%$ of the tensile strength (949 ± 15 MPa) which followed similar behavior as was found for the DED samples where the yield strengths also averaged $89 \pm 2\%$ of the tensile strengths. The difference in strain hardening

behavior can be connected to the martensitic microstructure exhibited within the stress relieved PBF samples. Strengthening mechanisms within the martensitic microstructure may have pushed the yield strength to a level that was approaching the tensile strength, resulting in significantly lower strain hardening than observed for the other AM components that exhibited lamellar $\alpha + \beta$ microstructures.

The data set for the PBF samples was small and could not provide a precise strength to hardness correlation. However, the orientational impact of the α phase and martensite, α' , could be investigated from Eq. (9) and (10). The PBF samples that underwent HIP exhibited a similar lamellar $\alpha + \beta$ microstructure as the DED samples that underwent HIP. A linear least square fit of the data to Eq. (9) and (10) using $n = 0.052$ resulted in an orientational impact factor, A , at 1.14 for the yield strength data and 1.10 for the tensile strength data that implied that the α phase increased the hardness by roughly 10 to 14% that was similar to that obtained from the DED data.

The stress relieved PBF samples exhibited a α' microstructure. From a linear least square fit using Eq. (9) and (10) and with $n = 0.032$, the orientational impact factor, A , for the stress relieved PBF samples was 1.08 and 1.06 for the yield and tensile strength data, respectively, that implied that the orientational impact of the α' structure increased the hardness by roughly 6 to 8%. Based on the results, it appeared that the α' microstructure still impacted the resulting strength to hardness correlations albeit at a slightly lesser amount than samples exhibiting a lamellar $\alpha + \beta$ microstructure.

5. Conclusion

Relationships between microhardness and the corresponding yield and tensile strengths for AM fabricated Ti-6Al-4V components were investigated and developed. It was shown that the microhardness measurements were impacted by the underlying microstructure and the local crystallographic orientation of the α phase, resulting in a large measurement variability. This variability was overcome by developing a methodology for aggregating sufficient microhardness data for each region of interest. Once a sufficient number of measurements were made to minimize the measurement error, microhardness measurements could be used to predict tensile and yield strengths. In addition, microhardness measurements were statistically as capable of resolving significant differences in material properties as tensile testing. The main conclusions are as follows:

- The mean microhardness measurements obtained from AM DED Ti-6Al-4V samples exhibited a linear correlation with the resulting yield and tensile strengths regardless of the process type or the post-process heat treated state.
- Empirically derived strength to hardness models that are widely used for Ni, Fe, Cu and Al based alloys can also be applied to AM Ti-6Al-4V samples if the added contribution of the limited slip system of the underlying α phase was taken into account.
- The stress relieved PBF samples exhibited a significantly different strength to hardness correlation than both the AM DED and PBF HIP samples. This difference was attributed to the martensitic microstructure observed within these samples. This martensitic microstructure resulted in a lower strain hardening exponent and a lower limited slip contribution to the microhardness measurements compared to the AM samples that exhibited a lamellar $\alpha + \beta$ structure.

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Figure Captions

Fig. 1. Vickers microhardness measured as a function of the height above the substrate for an AM Ti-6Al-4V wall structure. The mean is represented by the solid line and the standard deviation range is represented by dashed lines.

Fig. 2. A representative Vickers microhardness indent (a) made with a 1 kg_f load on an etched AM Ti-6Al-4V microstructure produced by an electron beam based DED process; and (b) a microstructural area listing the measured hardness values obtained from microhardness measurements where polarized light highlighted orientational differences of the α phase.

Fig. 3. Schematic of an L-shape wall structure fabricated by a 2 kW laser based DED process showing extraction locations for the tensile bars and metallographic samples. Vickers microhardness measurements were made on the y/z surface of the metallographic samples shown in the insert figure.

Fig. 4. Categorical scatter plot of the tensile and yield strength for the thin and thick wall structures fabricated by a 2 kW laser based DED process in the as-deposited and after HIP conditions. The horizontal bars represent the mean and standard deviation range.

Fig. 5. Histogram plots and the normal distribution curves of the Vickers microhardness measurements made across the height and length of wall structures fabricated from laser based

DED with different wall thicknesses and post-processed conditions. The measured mean, standard deviation, and the number of measurements, N , is also noted within the plots.

Fig. 6. The mean tensile and yield strengths as a function of the mean Vickers microhardness for thin and thick wall structures fabricated by laser based DED in the as-deposited condition and after post-process HIP. The dash lines represents a linear least square fit for the data. The error bars represent the standard deviation range for the strengths and a 95% confidence interval range for the hardness.

Fig. 7. The coefficient of variation of the Vickers microhardness, tensile strength, and yield strength measurements obtained from wall structures fabricated from laser based DED with different wall thicknesses and post-processed conditions.

Fig. 8. Representative optical micrographs after etching of Ti-6Al-4V AM components in the as-deposition condition for (a) electron beam based DED, and for (b) laser based DED; and after HIP for the (c) electron beam based DED, and for (d) laser based DED.

Fig. 9. (a) Yield strength plotted against the mean Vickers microhardness of AM DED samples fabricated by various processing techniques and undergoing various post-process heat treating. The solid line represents the least-square linear regression for the data and the dashed lines

represent the residual standard deviation for the regression. (b) Residual plot showing the difference of the observed valued and the predicted value for the linear regression.

Fig. 10. (a) Tensile strength plotted against the mean Vickers microhardness of AM DED samples fabricated by various processing techniques and undergoing various post-process heat treating. The solid line represents the least-square linear regression for the data and the dashed lines represent the residual standard deviation for the regression. (b) Residual plot showing the difference of the observed valued and the predicted value for the linear regression.

Fig. 11. Mean Vickers microhardness as a function of the yield strength (a) and tensile strength (b) measured from the DED samples. Included are the empirical hardness to yield strength correlations for given strain hardening exponents, n , using models of Cahoon et al. [26], Eq. (4), and Cahoon [45], Eq. (6).

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Fig. 13. Representative optical micrographs after etching of Ti-6Al-4V AM for PBF (a) after stress relieving and (b) after HIP.

Fig. 14. Representative stress-strain curves for the Ti-6Al-4V tensile samples produced by laser based PBF after post-process heat treating. The average strain hardening exponent, n , is also noted in the figure.

Fig. 1 - Single Column

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Fig. 4 - Single Column

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Fig. 13. Representative optical micrographs after etching of Ti-6Al-4V AM for PBF (a) after stress relieving and (b) after HIP.

Fig. 14 - Single Column

Fig. 14. Representative stress-strain curves for the Ti-6Al-4V tensile samples produced by laser based PBF after post-process heat treating. The average strain hardening exponent, n , is also noted in the figure.

Tables

Table 1. Analysis of the variance (ANOVA) for the microhardness measurements with a significance level of 0.05.

Source	Adj. sum of squares	Degrees of freedom	Adj. mean square	F value
Between builds	3.37×10^6	3	1.12×10^6	104.2
Within builds	4.96×10^6	460	1.08×10^4	
Total	8.34×10^6	463		

Table 2. Results from Tukey pairwise comparison of the microhardness measurements with a 95% confidence interval (95% CI) on the mean.

Build	N^a	Mean H_V (MPa)	Std. Dev. (MPa)	95% CI
Thin, As-Deposited	116	3585	93.9	3566, 3604
Thick, As-Deposited	116	3722	117	3703, 3741
Thin, HIP	116	3546	113	3527, 3565
Thick, HIP	116	3491	89.5	3472, 3509

^a N designates the number of hardness measurements