Since January 2020 Elsevier has created a COVID-19 resource centre with free information in English and Mandarin on the novel coronavirus COVID-19. The COVID-19 resource centre is hosted on Elsevier Connect, the company's public news and information website.

Elsevier hereby grants permission to make all its COVID-19-related research that is available on the COVID-19 resource centre - including this research content - immediately available in PubMed Central and other publicly funded repositories, such as the WHO COVID database with rights for unrestricted research re-use and analyses in any form or by any means with acknowledgement of the original source. These permissions are granted for free by Elsevier for as long as the COVID-19 resource centre remains active.
Research Paper

Deformations of Ti-6Al-4V additive-manufacturing-induced isotropic and anisotropic columnar structures: \textit{In situ} measurements and underlying mechanisms

Jo-Chi Tseng\textsuperscript{a,b,++}, Wei-Chin Huang\textsuperscript{c}, Wei Chang\textsuperscript{d}, Arno Jeromin\textsuperscript{b}, Thomas F. Keller\textsuperscript{b,e}, Jun Shen\textsuperscript{a}, Andrew Chihpin Chuang\textsuperscript{f}, Chun-Chieh Wang\textsuperscript{g}, Bi-Hsuan Ling, Lia Amalia\textsuperscript{a}, Nien-Ti Tsou\textsuperscript{d}, Shao-Ju Shih\textsuperscript{h}, E-Wen Huang\textsuperscript{d,i,*}

\textsuperscript{a} College of Mechatronics and Control Engineering, Shenzhen University, Shenzhen 518060, China
\textsuperscript{b} Deutsches Elektronen-Synchrotron DESY, D-22603 Hamburg, Germany
\textsuperscript{c} Laser and Additive Manufacturing Technology Center, Industrial Technology Research Institute, Hsinchu, 31040, Taiwan
\textsuperscript{d} Department of Materials Science and Engineering, National Chiao Tung University, Hsinchu, 30013, Taiwan
\textsuperscript{e} Physics Department, Universität Hamburg, D-20355 Hamburg, Germany
\textsuperscript{f} X-ray Science Division, Advanced Photon Source, Argonne National Laboratory, Lemont, 60439, USA
\textsuperscript{g} National Synchrotron Radiation Research Center, Hsinchu, 30076, Taiwan
\textsuperscript{h} Department of Materials Science and Engineering, National Taiwan University of Science and Technology, Taipei City 106, Taiwan
\textsuperscript{i} Research Center for Intelligent Medical Devices, Ming Chi University of Technology, New Taipei City 24301, Taiwan

\textbf{ARTICLE INFO}

Keywords:
Microstructure
Additive manufacturing
Ti-6Al-4V
\textit{In situ} X-ray diffraction
Heterogeneous texture distribution

\textbf{ABSTRACT}

The deformations of isotropic and anisotropic Ti-6Al-4V columnar structures fabricated by additive manufacturing were extensively examined. The distinct texture and microstructure distributions were characterised. \textit{In situ} X-ray diffraction measurements show different lattice activities resulting from the different microstructure distributions. Spatially resolved mapping revealed manufacturing-induced crystallite-orientation distributions that determine the deformation mechanisms. We propose a self-consistent model to correlate the multi-scale characteristics, from the anisotropic-texture-distribution microstructure to the bulk mechanical properties. We determined that basal and pyramidal slip activities were activated by tension deformation. The underlying additive-manufacturing-induced crystal plasticity plays a major role. We find that the texture development of the columnar structures and the distribution of crystallite orientation achieved by different processing conditions during additive manufacturing have important effects on the mechanical properties. The dominant deformation mode for the anisotropic Ti-6Al-4V columnar structure is basal slip, and that for the isotropic Ti-6Al-4V columnar structure is pyramidal slip. The difference may be important for determining the fatigue behaviour.

1. Introduction

The use of additive manufacturing (AM) in various applications has increased significantly in the past few years [1]. This emerging technology has even led to new business models for advanced manufacturing [2,3]. For example, the recent COVID-19 situation may re-shuffle the global supply chain, and AM could provide new solutions [4]. The unique feature of AM is the capability to integrate digitalised data from various sources, from high-throughput examinations to advanced photon sources for integrated computational materials engineering (ICME) [5].

The advantages of AM for metallic and other systems can be found in the review by Tofail et al. [6]. Specifically, Ti-based alloys, in particular Ti-6Al-4V, have been developed for several decades for use in aerospace, automotive, and medical applications. Given its non-toxicity, anti-corrosive nature, and stable properties in the human body, Ti-6Al-4V is an important material for biomedical implants [7]. Furthermore, its mechanical properties are also well adapted to the growth of bones [8]. The high strength-to-weight ratio and fatigue resistance of Ti-6Al-4V also make it attractive for use in the components of turbine engines, gears, and wings in the aerospace industry. These properties make Ti-6Al-4V an in-demand material, with more than 50% of the...
metallurgy industry working towards its production [9]. However, certain bottlenecks have been found in the conventional approach of manufacturing Ti-6Al-4V, such as the processing of samples with complicated geometry, oxidation of the surface, and flexibility of sample sizes [10]. To solve these problems, efforts have been made towards developing coated and/or doped Ti-6Al-4V or using novel processing approaches, such as AM for titanium alloys [11–13]. Hence, additively manufactured Ti-6Al-4V has drawn significant attention [14,15].

Laser powder-bed fusion (LPBF), electron-beam melting, and directed energy deposition are three major AM technologies. The spot-sized beam and quick heating-sintering methodologies make AM an elegant approach for fabricating samples rapidly, manufacturing neck-like shapes, and adding dopants easily to Ti-6Al-4V.

However, it has been found that the temperature gradient, build direction, and other AM processing parameters all interact with each other to affect the microstructure of the printed component, changing its mechanical properties [16–20]. The grains, coexistence of α and β phases, and crystallite defects are reported to be crucial factors affecting the mechanical properties of Ti-6Al-4V [21–24]. Moreover, porosity is a vital concern for Ti-6Al-4V [25]. Characterisation methods such as electron microscopy (EM), electron backscatter diffraction (EBSD), and X-ray diffraction (XRD) have been used to identify the homogeneity of phases and analyse the microstructural properties of samples [26–28]. Typically, the microstructural properties are determined by surface analysis (using EM/EBSD) and phase analysis (using XRD) [21,29]. Moreover, by combining these methods, comprehensive details of grain boundaries can be revealed. Raabe et al. demonstrated that 3D EBSD characterisation can be used to correlate the microstructure of alloys with their bulk properties [30–32]. However, EBSD is a destructive analysis approach, and the quality of the data depends on the sampling methodology. Specifically, collecting data from a well-polished specimen surface is essential for a confident reconstruction process from the Kikuchi pattern with all the scattering events included.

Rai et al. noted the importance of the evolution of the grain structure during powder-bed AM via simulation [33]. Later, Liu et al. provided insight into the mechanisms of the columnar-to-equiaxed grain transition during metallic AM [34]. Following their work, in this study, we attempt to reveal the microstructure evolution resulting from deformation, not fabrication, using XRD and tomography.

The use of an advanced photon source, such as a synchrotron X-ray or neutron beam with high penetration capability, combined with in situ tensile experiments, can reveal real-time lattice-to-macroscopic mechanical properties. Accordingly, the correlation of lattice strain with engineering strain can be obtained simultaneously. Studies by Wilkinson et al. have found a strong correlation between lattice strain and engineering strain with texture development during tensile experiments using high-energy synchrotron XRD analysis [35–37]. Huang et al.’s in situ neutron diffraction experiments also successfully captured the phase transformation of the AM-induced transient phase [17,38]. However, the pores induced by the AM process play a crucial role in mechanical performance, as determined by in situ diffraction-related results reported by Qiu et al. [39,40]. These results are important for the potential high-throughput experimental methodologies of the materials genome initiative (MGI) [41] and efficient AM development and production [6]. Therefore, information obtained from X-ray experiments can contribute to a comprehensive correlation between microstructure analysis and the bulk properties of materials.

The fabrication strategies [42,43] associated with AM have been extensively summarized by the AM community for controlling the elongation, strength [44–47], residual stress [48–51], and fatigue [52,53] properties. For example, Galarraga et al. already categorized the microstructure-dependent mechanical properties of the Ti-6Al-4V ELI alloy fabricated by electron beam melting (EBM) [54]. Galarraga et al. have found that equiaxed α + β, columnar α + β, partially α', and fully α' correspond to furnace-cooling, EBM-built, air-cooling, and water-cooling condition, respectively, show different strengths and elongation levels accordingly [54]. For large deformations, Galarraga et al. summarized the thickness of hcp lamel and lamellar structure effects [54]. Wang et al. demonstrated the hierarchically heterogeneous microstructure [55].

However, unlike the aforementioned comprehensive discussion on the overall strengths and elongation comparisons in the archived literature, we focus on small deformations to investigate the onset of plasticity in this work, which is essential for examination of the fatigue mechanisms [53]. For correlation with the bulk mechanical behaviour, we followed the protocols of in situ diffraction measurements because detectable lattice asymmetry can reveal the initiation of fatigue cracks [56]. Once there is a sufficient amount of fatigue pore formation, Considère’s volume conservation for crystal plasticity will not be followed, which can be observed by the in situ lattice deformation [57,58].

In the current study, we applied various high-energy synchrotron X-ray diffraction (SXRD) experimental approaches to investigate LPBF-manufactured Ti-6Al-4V and the effect of the LPBF processing parameters, such as the build direction. Liu et al. [34] and Rai et al. [33] have already concluded that, together with the thermal gradient, the solidification rate can generate different microstructures, such as columnar and equiaxed grains. However, the direction correlation of such a columnar structure with the bulk mechanical properties of a material remains uncertain. In this work, we examined the deformation mechanisms for two additive-manufacturing-induced columnar structures: one has an isotropic texture, and the other has an anisotropic texture.

Moreover, we designed spatially resolved scans to map crystallite orientation distributions. This non-destructive mapping can disclose the local texture and microstructure, while the in situ diffraction analysis can provide kinetic information at the lattice scale and characterise the bulk properties of the material. Meanwhile, the average crystal structure of the prepared Ti-6Al-4V sample was also theoretically simulated (and compared with experimental results) for a quantitative analysis of crystal defects via whole powder pattern modelling (WPPM) [59,60].

In addition, we can also use the synchrotron X-ray to scan the specimen with varying penetration power to gauge information through the thickness of the samples. Subsequently, spatial scans allowed us to map the microstructural distribution for the whole testing area and to quantify the strain by modelling of the X-ray patterns. This makes it possible to build multi-scale character recognition from the lattice microstructure to bulk mechanical properties for LPBF specimens. This work focuses on examining the AM-induced texture and microstructure distribution effects of Ti-6Al-4V, which are important for fatigue characterisation.

2. Methods

2.1. Samples

Two Ti-6Al-4V samples were prepared via LPBF with build directions parallel (vt-anisoTAV) and perpendicular (hz-isotAV) to the axis to form distinct textures for uniaxial tensile experiments (Fig. 1(a) and (b)). The zigzag approach was applied, and the vectors were changed by 67° from layer to layer [61].

Ti-6Al-4V alloy micropowder was purchased from Atomic Craft Corporation. The powder consisted of Ti (balance), Al (5.5–6.75 %), V (3.5–4.5 %), O (< 0.2 %), N (< 0.05 %), C (< 0.08 %), H (< 0.015 %), and Fe (< 0.3 %) in weight per cent. The particle size distribution was from 15 to 45 μm. AM of the manufactured Ti-6Al-4V alloy powders was performed on an Industrial Technology Research Institute (ITRI)-AM 250 machine. Samples were processed with the (ITR)-AM 250 ‘zig-zag’ scan strategy, which was specifically developed for the Ti-6Al-4V alloy material to minimise thermal and residual stress build-up in the equipment. The zig-zag strategy that composes the core of the contouring hatch is 0.1 mm offset, using 170 W laser power (fibre laser
with a wavelength of 1070 nm), 1150 mm/s scan speed, 0.1 mm hatch spacing, and 70 μm laser spot size. Standard machine parameters provided by the (ITRI)-AM 250 for the Ti-6Al-4V alloy were used for all builds. Further details of the parameter values are considered proprietary by ITRI. The 80 mm × 20 mm × 5 mm build plates were machined out of Ti-6Al-4V, and their surfaces were sandblasted. Layers of the build were incremented by 30 μm. Processing was done under a flowing, inert argon atmosphere with oxygen monitoring. All processing was completed at room temperature with no heat applied to the build plate. Samples were removed from the machine and cleaned of extra powder by sonicating in water. Parts were then dried with clean compressed dry air.

Through this process, multilayer-like Ti-6Al-4V samples can be obtained. Specifically, we controlled the fabrication parameters to ensure our samples were mainly in a hexagonal close packed (hcp) phase as α phase with very minor body centered cubic (bcc) phase as β phase. For overall bulk strengths and elongations, our samples have the same trend as Galarraga et al. summarized [54].

2.2. Synchrotron X-ray measurements

To examine the chemical distribution, the specimens were illuminated using the X-ray Nanoprobe beamline of the Taiwan Photon Source, TPS 23A, with 100 nm spatial resolution for complementary elemental distributions. The energy of the incident X-rays was 12.8 keV, well above the k-edge absorption energies of the constituent elements of the specimens. The excited fluorescence was collected simultaneously by a silicon drift detector with an energy resolution of approximately 150 eV [62].

To reveal the pores, synchrotron transmission X-ray microscopy was used to examine beneath the material surface. Transmission X-ray microscopy (TXM) enables the characterisation of high-resolution X-ray radiography in three dimensions (3D). With the aid of a Fresnel zone plate and high-flux synchrotron hard X-ray source, TXM can achieve a spatial resolution of approximately 60 nm. The images of the tomography were reconstructed using Amira software [63]. The number and the geometry of the voids were estimated from the Amira algorithm analysis of the 3D TXM images.

To investigate the deformation mechanisms, in situ uniaxial tensile SXRD experiments were carried out at P02.1, DESY [64]. The samples were mechanically cut to the required shape according to the ASTM subsize of the EBM-04 Standard Test Methods for Tension Testing of Metallic Materials for the in situ uniaxial tensile SXRD experiments [65]. A high-energy X-ray beam of approximately 60 keV (wavelength ~0.207 Å) and a Perkin-Elmer EN1621 2D detector with a 0.2 × 0.2 mm² pixel size were used to collect the 2D XRD images. The sample-to-detector distance was set to 1185 mm. A 1 × 1 mm² X-ray beam size was used for all experiments. For the tensile experiments, a 5 kN tensile/compression module machine (Kammrath-Weiss) was used, with an elongation rate of 20 μm/s applied in continuous mode. The continuous mode enables the correlation of engineering mechanical properties and lattice kinetics without creep. During the uniaxial tensile experiments, both engineering strain and XRD patterns were simultaneously collected every second to study their correlation. The samples were also measured before and after the uniaxial tensile experiment with a 1 × 1 mm² X-ray beam spot over the entire rectangular area of the specimen to examine the local texture development.

2.3. Data reduction & fitting

Prior to the analysis of the 2D XRD images, Cr₂O₃ powder was measured to calibrate the sample-to-detector distance, the beam centre, and the tile of the detector. Based on the instrumental parameters obtained by the Cr₂O₃ measurement, the collected 2D XRD images for samples could be integrated into 1D XRD patterns with the Data Analysis Workbench (DAWN) [66,67]. Two strategies for the integration of 2D XRD images were used. (1) Each 2D XRD image was integrated for 36 distinct orientation regions along the azimuthal angle to the 1D XRD patterns to study the texture properties of samples. Each peak in the 1D XRD patterns was fitted with pseudo-Voigt functions by a Python program, and the peak intensity, peak position, and peak width were obtained for subsequent analysis. (2) The 2D XRD image was integrated using full Debye–Scherrer rings to obtain the average peak positions and lattice parameters. The determination of dₒ for each sample was performed to address the starting parameters for the as-prepared samples with residual stresses induced from the AM process. Subsequently, calculation of the change in lattice strain during the tensile experiment was used to determine the deformation behaviour of samples. In addition to comparing the XRD patterns obtained from the two sampling techniques, the theoretical static strain and growth fault were simulated via the whole powder pattern modelling (WPPM) approach [59,68,69]. The broadening of peaks in an XRD pattern can be regarded as an effect of the microstructure characteristics, such as crystallite size and strain. One of the modelling approaches, WPPM,
allowed us to simulate the amount of growth faults for hcp Ti-6Al-4V. WPPM is implemented in the PM2K v2.1 software package, and an XRD pattern can be generated by the Fourier transform method using a defined crystal model with the desired defects, such as dislocations, twins, or growth faults. By changing the probability of the aforementioned defects, a series of XRD patterns can be obtained to understand the number of defects in correlation with the change in the XRD pattern. Because the size of crystallites in the studied case was not the dominant factor, the parameters controlling crystallite size were fixed during the simulation process.

2.4. Electron microscopy

A high-resolution field-emission scanning electron microscope was used for the EBSD experiments with a 5 keV electron beam [70]. The samples were fixed on copper tape.

3. Results

3.1. Physical properties of as-prepared samples

Prior to the uniaxial tensile experiments, the samples were mechanically polished and characterised to investigate the physical properties related to mechanics, such as the concentration of pores, the distribution of elements, and the homogeneity of phases. We followed the protocols reported by Stef et al. [71] to examine the pores in our samples. The 3D transmission X-ray tomography (TXM) high-spatial-resolution images are shown in SI 1(a) and (b), respectively. Although the pore volume fractions of our samples are much lower, Stef et al. have confirmed the influence of voids on the mechanical properties of ultimate tensile stress (UTS) and ultimate elongation for a volume fraction as low as 0.48 vol.%. Hence, in this study, we limited our scope to only small deformations within 3.8%, which is much lower than the UTS and the ultimate elongation. For small deformations, Ashby et al.’s construction shows that in deformed crystalline metals, volume conservation occurs during plastic deformation subjected to slip and dislocation activities without void nucleation [72]. Specifically, the onset of void effects can be revealed by the deviation of the Poisson’s ratio [56,58], which describes the expansion of a material in directions perpendicular to the direction of compression. We applied angle-resolved diffraction to monitor the two orthogonal diffractions to examine the evolution of the diffraction pattern in both the tension and transverse directions simultaneously.

X-ray fluorescence mappings (SI 1(c)) show the homogenous distribution of elements in LPBF-manufactured Ti-6Al-4V. In addition, only the hcp phase of Ti-6Al-4V was identified from the SXRD analysis for both samples (SI 2).

The residual strains present in the as-prepared samples after removal from the LPBF bed were analysed via scanning XRD, as shown in SI 3. The range of the residual strain was similar for both as-prepared samples, between approximately $10^{-2}$ and $10^{-3}$ %.

3.2. Crystallite stacking models and preferred orientations

Although only the hcp phase was identified for both samples, the peak intensities in both XRD patterns are slightly different, in particular for the (0002) and (10 1 0) planes, as shown in SI 2. The intensity difference in a 1D XRD pattern may be influenced by the microstructure and texture of a sample, and beam instability of the instrument, among other factors. In the case of highly polycrystalline materials, such as the as-prepared samples, texture formation is most likely the cause. Similar evidence was reported by Dye et al., where a strong texture was observed in a mixture of α and β Ti-6Al-4V systems [24]. Kanitpanyacharoen et al. have also shown different deformation modes and their associated orientation-dependent diffraction-intensity patterns with azimuthal angles of 2D XRD patterns for texture development in other hcp systems [73]. Therefore, to resolve the convoluted information in the XRD patterns from both samples, the raw 2D XRD images of as-prepared vt-anisoTAV and hz-isoTAV were analysed (Fig. 2(a) and (c)). The azimuthally resolved Debye–Scherrer rings in Fig. 2(a) show that (0002), the second ring away from the beam centre, is partially discontinuous for vt-anisoTAV. The intensities of the (0002) Debye–Scherrer ring are much higher at the azimuthal angles of 50°, 140°, 180°, 230°, and 320°. In contrast, no regularity of the intensity changes

\[
\text{azoB}
\]
was found for the (0002) ring of hz-isoTAV (Fig. 2(c)). Considering that the thermal history of the build layers in AM is the same for both samples, the stacking structure along with the X-ray viewing direction and tensile direction can be distinct if the build direction is different (Fig. 2(b) and (d)). The yellow cubes represent the X-ray transmitted volume with a 1 × 1 mm² beam size, which corresponds to the measuring area for each resulting XRD pattern. It is clear that vt-anisoTAV, with the build direction perpendicular to the tensile direction, has a layer-like structure with highly preferred orientated crystallites (Fig. 2(b)). When X-rays are transmitted through such a sample, the regular changes in diffraction intensities can be illustrated. In contrast, the crystallite structure of the hz-isoTAV is randomly stacked along the X-ray path if its build direction is parallel to the tensile direction (Fig. 2(d)). Consequently, the plot of the (0002) Debye–Scherrer ring appears to have no intensive regularity with the change in intensity along the azimuthal angles.

To exclude the randomness from measuring with a 1 × 1 mm² X-ray beam, spatially resolved scans over the entire rectangular area of both samples were performed (Fig. 3(a) and (b)). The normalised intensity of the (0002) Debye–Scherrer ring along the azimuthal angles is used to exclude the experimental error caused by the beam instability.

As shown from the static plots in Fig. 3(a) and (b), six strong peaks were obtained for vt-anisoTAV, at azimuthal angles of 40°, 130°, 170°, 220°, 310°, and ∼0°, whilst no strong peak was found for hz-isoTAV. It is expected that the peaks can be paired as 40°/220°, 170°/310°, and 130°/0° from the perspective of the multiplicity of (0002). Interestingly, in the non-normalised data set (SI 4), the same trend of strong peaks was found in the third Debye–Scherrer ring, assigned to (10 T 1) (Figs. 2 and 3). The two conditions discussed above are only satisfied when (10 T 1) is almost parallel to the build direction and (0002) is at an angle of approximately 30° to the sample surface (Fig. 4(a)).

Accordingly, the spatial mapping of the distribution of (0002) preferred orientations over the entire sample with respect to transmitted X-ray cross-sections is plotted in Fig. 4(b) and (c). In the vt-anisoTAV, Fig. 4(b), more than 80 % of the area was in the same orientation. Conversely, the hz-isoTAV sample (Fig. 4(c)) had less than 35 % of crystallites in the same orientation.

Thermodynamically, such preferred orientations of (0002) in the Ti-6Al-4V system could be caused by the texture formation of an hcp structure. When the crystallites grow rapidly, the growth of an hcp structure along the c axis is likely preferable, leading to (10 T 1) facets lying on the substrate [47,75–79]. The map of the microstructure from columnar to equiaxed grains subjected to both various thermal gradients and solidification rates has been reported by Liu et al. [34]. With different building strategies, the crystallites of our samples have different effective times and regions for grain growth during the manufacturing process. Furthermore, successive build layers can be regarded as substrates for the following layers in the AM approach. Accordingly, our samples manufactured via LPBF show different partial preferred orientations for the crystallites. For more details of how and why the

Fig. 3. Collection of 2D XRD raw images over the whole testing area via the scanning X-ray method for (a) vt-anisoTAV and (b) hz-isoTAV samples and the static plots of the (0002) Debye–Scherrer ring along the azimuthal angles for all collected images.
hcp texture is formed, please see the following representative references [80–82].

The nature of the growth direction of an hcp structure leads to the assumed stacking structure for the two samples with respect to the LPBF build strategy, as shown in Fig. 2(b) and (d). The crystallites were rotated 67° layer-by-layer along the build direction (for ease of understanding, the crystallites are shown with a 3D structure). The 3D crystal structure with face (0002) facets and side (1011) facets is shown based on an hcp structure configuration.

Therefore, the crystallites were arranged in the same way for both samples. However, depending on the viewing direction of the X-ray beam, the scattering events differed according to the orientation of crystallites in the area measured by the 1 × 1 mm² transmitted X-ray beam. For the vt-anisoTAV sample, the (0002) was facing or at an angle of less than 30° to the surface of the specimens (Fig. 2(b)). Consequently, a highly preferred orientation of (0002) could be expected and observed in the X-ray pattern; the (0002) was parallel to the tensile direction in this configuration. In contrast, for the hz-isoTAV sample, a highly random crystallite orientation was evident in the X-ray transmitted area. However, crystallites with a growth direction along (1011) were mostly observed on the surfaces of the samples, which was parallel to the tensile direction, as shown in Fig. 2(d). Based on the two presumed directional-distinct stacking models, the X-ray scattering events of d(0002) were expected to be azimuthally dependent in both cases [36].

By calculating the diffraction angles of the aforementioned crystallite orientations, the strong peaks, from three grouped φ angles, obtained from experimental data (Fig. 3(a)) were matched to the proposed stacking crystallite model shown in Fig. 2(b). It should be noted that for the case assuming (0002) to be almost perpendicular to the X-ray beam, the scattering events on (0002) are difficult to detect because of the use of a planar detector. Conversely, no preferred orientation was found from the 2D XRD images for hz-isoTAV, as shown in Fig. 3(b). This can be explained by the highly random orientations of the crystallites across the transmitted X-ray beam in the horizontally built sample. Here, the definition of crystallite orientation should be further specified. The preferred orientation of a crystallite can be different from the texture properties of crystallites for a material. The preferred orientation of crystallites refers to the specific direction that most of the crystallites in particles are oriented towards, whilst the texture is regarded as the growth direction of crystallites in particles along a specific direction. Sometimes, the preferred orientation of crystallites is not significantly related to the texture formation of crystallites, and vice versa. In this particular case, strong preferred orientations of crystallites were observed, based on the scattering spots shown in the 2D XRD images.

The stacking structure of the crystallites for vt-anisoTAV could be considered a layer-like structure with respect to the direction of the uniaxial tensile experiments, and the (0002) directions were oriented at an angle of less than 30°. In contrast, for the hz-isoTAV sample, no specific (0002) stacking structure was observed; however, the (1011) is in-plane with the applied tension direction.

Interestingly, the presumed stacking models also agree with the results of EBSD tests (Fig. 5). The crystallites in the vt-anisoTAV sample have a layer-like arrangement along the build direction, as shown in Fig. 5(a). Looking at the X-ray viewing direction in Fig. 5(c), the crystallites are regularly oriented, which leads to the discontinuity in the Debye-Scherrer rings in the 2D XRD image shown in Fig. 2(a). Such texture properties are specific to the tensile direction for vt-anisoTAV. Alternatively, on the surface of the hz-isoTAV, an in-plane stacking structure along (1011) was observed for the majority of the sample test area (Fig. 5(b)). Also, the crystallite orientation along the X-ray viewing direction was highly random, resulting in the good continuity of the Debye-Scherrer rings in the 2D XRD image shown in Fig. 2(c). In the other direction, the EBSD measurements for the hz-isoTAV sample show a columnar microstructure similar to that of the vt-anisoTAV sample along the X-ray viewing direction, where partial layer-like arrangements of crystallites are illustrated (Fig. 5(d)). Although the testing area in the EBSD analysis is rather small, the observations support the presumed models. By combining the results of the designed spatial scan method, a comprehensive picture of the crystallite stacking structure in both as-prepared samples can be defined. It is important to understand such distinctive stacking crystallite structures and their effect on the mechanical properties for LPBF-manufactured Ti-6Al-4V.

4. Discussion

The correlation between engineering mechanical properties and the proposed stacking structure models is discussed below focusing on small deformation. Both samples were examined using in situ XRD uniaxial experiments under continuous mode at the high-energy Synchrotron X-ray beamline, P02.1, DESY. This allowed us to collect the 2D XRD images rapidly (1 frame/s) and illustrate the dynamic changes in engineering strain and lattice strain simultaneously, without creep. By integrating the 2D XRD images to the 1D XRD patterns and subsequently fitting the diffraction peaks, the lattice strain on different crystal planes can be calculated using:

$$\Delta \varepsilon = (d_0 - d_\varepsilon)/d_0,$$

where $d$ is the d-spacing between two lattice planes. The XRD patterns were collected for both samples before the tensile experiment to define the $d_0$.

The results in Fig. 6 show that the lattice strain averaged over all of the azimuthal angles and engineering strain are highly correlated in both the elastic and plastic deformation regions during the uniaxial tensile experiments [37]. However, the individual lattice strain
evolution on different crystal planes was very different between the two cases. The lattice strains calculated from (1010), (0002), and (1011) are similar for vt-anisoTAV, although the lattice strain of the (0002) crystallite plane slightly decreases in the plastic deformation region (Fig. 6(a)). In contrast, the lattice strain of (1011) is much more pronounced in both the elastic and plastic deformation regions for the hz-isoTAV sample (Fig. 6(b)). Such distinctive directionally related deformation can be correlated with the proposed stacking structure.

For hz-isoTAV, (1011) was presumably in-plane with the uniaxial tensile experiment, which played a crucial role during the deformation, as observed from the highest strain accumulated [22]. In contrast, the lattice strain seems to have developed nearly equally for the vt-anisoTAV. However, by evaluating the lattice strain in two orthogonal dimensions, considering $\varepsilon_{11}$ and $\varepsilon_{22}$, a distinct change in the lattice strain of the (0002) crystallite planes can be observed (Fig. 7). At the beginning of plastic deformation, a decrease in $\varepsilon_{11}$-left and an increase in $\varepsilon_{11}$-up were observed. Until $\varepsilon_{11}$-up reached zero, only $\varepsilon_{11}$-left decreased. It seems that the deformation of (0002) was non-uniform along the tensile direction during the tests. Based on the calculated values of the $\varepsilon_{11}$ and $\varepsilon_{22}$ and lattice strains on d_{0002}, a deformation mechanism of (0002) can be proposed (Fig. 7(d)). The crystallites are deformed elastically with a twist-like deformation mechanism in the plastic region. As $\varepsilon_{11}$-up approached zero, the decrease in $\varepsilon_{11}$-left led to the sliding of the crystallite planes normal to (0002) in one direction. In contrast, ordinary deformation was obtained for the other crystallite facets during the tensile test. This could indicate that the basal slip system, or (0002), was dominant in the case of vt-anisoTAV. In addition, the accompanying distortion of (1011) was observed in the plastic deformation region, showing a strong correlation with (0002).

To reconcile the different lattice strain evaluations of the two samples and the relationship among the three crystallite facets, a hypothesis schematic is shown in Fig. 8(a) and (b), demonstrating the multiple crystallites before and after the uniaxial tensile experiments. It is clearly shown that the elongation and deformation of the crystallite
Fig. 7. (a) Schematic showing how the 2D lattice strains are calculated. Correlation between the lattice strain and strain on different crystal planes is shown for (b) vt-anisoTAV and (c) hz-isoTAV samples. (d) Demonstration of the deformation of d_{0002} in the 2D lattice strain configuration.

Fig. 8. Schematic of multiple deformed crystallites during the uniaxial tensile experiment for the (a) vt-anisoTAV and (b) hz-isoTAV samples.
planes behave differently for the two samples, and this corresponds well with the presumed stacking structures and associated experimental lattice strain behaviour.

For the vt-anisoTAV, the more layer-like stacking structure caused by its columnar texture led to presumably distinct layers perpendicular to the experiment’s tensile axis. Most of the (0002) facets were at an angle of less than 30° to the tensile direction. Such a stacking structure resulted in a strong correlation between the (0002) and (1011) planes and the major deformation of (0002) along the tensile direction. In addition, between distinct layers or within one build layer, a twist-like distortion of the crystallites occurred (Fig. 7(d)). Not until the lattice strain of εz became zero and the sliding of the crystallites occurred did the rupture of the sample, either in the pore-joint area or in the crystallite-twisted area, take place. It has been reported that larger amounts of plastic strain in the basal planes, or the (0002) facets, can lead to less critical stress normal to the (0002) facet and result in a quicker occurrence of rupture [22,83]. This could explain the weaker mechanical properties in the vt-anisoTAV sample.

Alternatively, for the hz-isotAV, the (1011) facets are in-plane with the tensile experiments and seem to be the dominant planes in plastic deformation. The residual strain and plastic strain were accumulated in the (1011) facets subjected to tensile deformation. This observation is commonly reported in hcp Ti-6Al-4V, and the development of (1011) growth faults or a higher yielding strength could be expected. To understand the correlation of the development of the pyramidal slip system with changes in the XRD pattern, theoretical patterns with different probabilities of the (1011) growth faults were simulated using the Warren model in whole powder pattern modelling (WPPM) [38,68]. Based on a hcp Ti-6Al-4V crystal model implemented in WPPM, the XRD pattern was obtained by the Fourier transform method governed by the pre-calculated instrumental profile and all crystallographic parameters given, including the multiplicity, probability of growth fault, and negligible presence of twin faults. As a result, the decreased intensity and broadening of the (1011) peak can be seen, while the other peaks do not change. Thus, by calculating the intensity of the (1011) peak in the XRD patterns collected during the uniaxial tensile experiment, the development of the (1011) growth fault can be quantified. For the vt-anisoTAV sample, the intensity of the (1011) peak decreases slightly, while for hz-isotAV, it decreases by a factor of one-third (SI 5(b) and (c)). Comparing the results with the theoretical calculation, it can be deduced that more than 5% of the (1011) growth faults were developed during the uniaxial tensile experiments for the hz-isotAV. In contrast, the (1011) growth faults did not significantly develop during the tensile experiments in the vt-anisoTAV sample.

Based on the proposed model depicted in Fig. 2(b) and (d) and the discussion above, the slip systems between two samples were found to be distinct because of the different microstructures resulting from the different orientations of the columnar texture. Basal and pyramidal slip are the dominant systems for the vt-aniso- and hz-isotAV samples, respectively. In addition, the slip system evolution could lead to the different mechanical behaviour. In summary, the stacking structures caused by the isotropic and anisotropic columnar structures subjected to different build strategies led to distinct underlying deformation mechanisms.

5. Conclusion

Ti-6Al-4V was manufactured using LPBF, and the characteristics at different scales, including the microstructure, texture, and engineering mechanical properties, were investigated.

Synchrotron 2D XRD analysis suggests that the orientations of the stacking crystallites differed with respect to the local columnar texture distributions, which resulted from control of the thermal gradient and solidification rate, as a result of the nature of the preferred growth direction of the hcp structure. For the vertically built Ti-6Al-4V (vt-anisoTAV), the crystallites were highly preferentially oriented in an anisotropic manner, where, in most of the grains, the (0002) planes were distributed with orientations less than 30° from the direction of the tensile experiments. In contrast, the crystallites in the horizontally built Ti-6Al-4V (hz-isotAV) were randomly oriented. Similar evidence was obtained from the complementary EBSD analysis. The distinctive columnar texture from the crystallite stacking structures resulted in different deformation mechanisms.

During the uniaxial tensile experiment, a (1011) strain accumulation in the hz-isotAV sample was observed, leading to the development of over 5% (1011) growth faults. Twist-like (0002) slip faulting (a basal slip system) occurred in the vt-anisoTAV, which was caused by the strong preferential orientation illustrated in the stacking structure model.

Based on the use of multi-scale characterisation, the distinctive stacking structures originating from the different microstructures were successfully defined using a non-destructive in situ approach and were correlated with the tensile properties for LPBF-manufactured Ti-6Al-4V.

Contributions

EWH outlined, organised, supervised, and conducted the research. JCT and EWH prepared the manuscript. EWH coordinated the revision. JCT, WC, AJ, and TFK performed, collected, and analysed the EBSD data. JCT, performed, collected, and analysed the data from the high-energy synchrotron X-ray diffraction measurements and EBSD. LA carried out the complementary EBSD for samples at different surfaces for the revision. ACC performed and analysed transmission X-ray microscopy (TXM). CCW and LA reconstructed the data from the synchrotron-based TXM experiments. BHL and LA performed and analysed the X-ray Nanoprobe. WCH, SJS, and NTN prepared the samples. All authors approved the final version.

Funding sources

This work was supported by the Ministry of Science and Technology (MOST) Programs (grant numbers 107-2628-E-009-001-MY3, 107-2218-E-009-003, 108-2218-E-007-056, 108-3017-F-009-003, 109-2634-F-009-029 and 108-2221-E-009-131-MY4); and “Center for Semiconductor Technology Research” from the Featured Areas Research Center Program within the framework of the Higher Education Sprout Project by the Ministry of Education (MOE) in Taiwan. This work was financially supported by the “High Entropy Materials Center” from The Featured Areas Research Center Program within the framework of the Higher Education Sprout Project by the Ministry of Education (MOE) in Taiwan and Industrial Technology Research Institute (ITRI) Program 109A502. This work was financially supported by the “Center for the Semiconductor Technology Research” from The Featured Areas Research Center Program within the framework of the Higher Education Sprout Project by the Ministry of Education (MOE) in Taiwan. Also supported in part by the Ministry of Science and Technology, Taiwan, under Grant MOST 109-2634-F-009-029.

Declaration of Competing Interest

The authors declare no competing financial interests.

Acknowledgements

We are deeply grateful to Dr. Jozef Bednarick (DESY, P02.1), Dr. Tim Schoof (DESY, P02.1), Mr. Hsu-Hsuan Chin (NCTU), and Ms. Wen-Chi Yang (NCTU) for the helpful discussions. We also thank Kuan-Yin Tseng for comprehensive help with the instrumentation and experiments. We acknowledge DESY (Hamburg, Germany), a member of the Helmholtz Association HGF, for the provision of experimental facilities.
Parts of this research were carried out at P02.1, PETRA III, DESY and at the DESY Nanolab. This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357. This research used resources of the Taiwan Photon Source. The authors would like to thank Research Center for Intelligent Medical Devices at Ming Chi university of technology to support reactive ion etching instrument for sample surface preparations.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:10.1016/j.adma.2020.101322.

References

[1] M. Attaran, The rise of 3-D printing: the advantages of additive manufacturing over traditional manufacturing, Bus. Horiz. 60 (5) (2017) 677.

[2] J. Savolainen, M. Collan, How additive manufacturing technology changes business models? – review of literature, Addit. Manuf. 32 (2020) 101707.

[3] A. Paalini, S. Kollmannsberger, E. Rank, Additive manufacturing in construction: a review on processes, applications, and digital planning methods, Addit. Manuf. 30 (2020) 100894.

[4] S. Zanoni, M. Ashourpour, A. Baccetti, M. Zanardini, M. Perona, Supply chain implications of additive manufacturing: a holistic synopsis through a collection of case studies, Int. J. Adv. Manuf. Technol. 102 (9) (2019) 3325.

[5] M. Seif, A. Salem, J. O. Haryussen, J. van der Putten, Overview of metal qualification needs for new additive manufacturing, JOM 68 (3) (2016) 747.

[6] S.A.M. Tofigh, E.P. Koumoundouros, A. Bandopadhyay, S. Bose, L. O'Donoghue, C. Chaturvedi, Additive manufacturing: scientific and technological challenges, market uptake and opportunities, Mater. Today 21 (1) (2017) 22.

[7] L.E. Murr, S.A. Quinones, S.M. Gaytan, M.I. Lopez, A. Rodela, E.Y. Martinez, Mechanical behavior of Ti-6Al-4V produced by rapid-layer manufacturing, for biomedical applications, J. Mech. Behav. Biomater. 2 (2) (2009) 20.

[8] Y. Li, C. Yang, H. Zhao, S. Su, X. Li, Y. Li, New developments of Ti-based alloys for biomedical applications, Materials (Basel) 7 (3) (2014) 1709.

[9] M.J. Donachion, Titanium: A Technical Guide, 2nd edition, ASM International, 2000.

[10] R. Huang, M. Riddle, D. Graziano, J. Warren, S. Das, S. Nimbalkar, J. Cresko, E.B. Sirota, A.B. Herhold, Transient phase-induced nucleation, Science 283 (5401) (1999) 100884.

[11] E.W. Huang, S.Y. Lee, J. Jain, Y. Tong, K. An, N.-T. Tsou, T.-N. Lam, D. Yu, H. Chae, J.S. Kim, T. LaGrange, B.W. Reed, M.L. Taheri, M.R. Armstrong, W.E. King, H. Abdolvand, J. Wright, A.J. Wilkinson, Strong grain neighbour effect of texture and microstructure of Ti-6Al-4V sheets, Mater. Sci. Eng. A 760 (2019) 431.

[12] A.M. Stapleton, S.L. Collart, J. Haryussen, J.H. Stone, T.C. Lindley, D. Dye, Evolution of lattice strain in Ti–6Al–4V during tensile loading at room temperature, Acta Mater. 56 (2008) 6186.

[13] J.W. Warwick, J. Coakley, S.L. Rahghunauth, R.J. Talling, D. Dye, Effect of texture on load partitioning in Ti-6Al-4V, Acta Mater. 50 (2012) 4217.

[14] A. da Plessis, P. Rossouw, Investigation of porosity changes in cast Ti6Al4V rods after hot isostatic pressing, J. Mater. Eng. Perform. 24 (2015) 3137.
strength and ductility, Nat. Mater. 17 (2018) 63.

[56] E.W. Huang, R.I. Barabash, B. Clausen, P.K. Liaw, Cyclic-loading induced lattice-strain asymmetry in loading and transverse directions, Metall. Mater. Trans. A 43 (2012) 1454.

[57] E.W. Huang, R.I. Barabash, B. Clausen, Y.-L. Liu, J.-J. Kai, G.E. Ice, K.P. Woods, P.K. Liaw, Fatigue-induced reversible/irreversible structural-transformations in a Ni-based superalloy, Int. J. Plast. 26 (2010) 1124.

[58] E.W. Huang, C.-K. Chang, P.K. Liaw, T.-R. Suei, Fatigue induced deformation and thermodynamics evolution in a nano particle strengthened nickel base superalloy, Fatigue Fract. Eng. Mater. Struct. 39 (2016) 675.

[59] M. Leoni, T. Confente, P. Scardi, PM2K: a flexible program implementing whole powder pattern modelling, Z. Krist. Suppl. 23 (2006) 249.

[60] P. Scardi, Whole powder pattern modelling, Acta Crystallogr., A, Found. Crystallogr. 58 (2002) 190.

[61] Marc Dümter, Ralph Mayer, Ludger Hümmler, Rainer Salzbeger, Jüha Kotila, Tatju Syyvola, Method and Device for Manufacturing a Three-dimensional Object, US8034279B2, United States, 2011.

[62] E.W. Huang, H.-S. Chou, K.N. Tu, W.-S. Hung, T.-N. Lam, C.-W. Tsai, C.-Y. Chiang, B.-H. Lin, A.-C. Yeh, S.-H. Chang, Y.-J. Chang, J.-J. Yang, X.-Y. Li, C.-S. Ku, K. An, Y.-W. Chang, Y.-L. Jao, Element effects on high-entropy alloy vacancy and heterogeneous lattice distortion subjected to quasi-equilibrium heating, Sci. Rep. 9 (2019) 14788.

[63] T.-N. Lam, C.-H. Wu, S.-H. Huang, W.-C. Ko, Y.-L. Huang, C.-Y. Ma, C.-C. Wang, E.-W. Huang, Multi-scale microstructure investigation for a PM2.5 air-filter efficiency study of non-woven polypropylene, Quantum Beam Sci. 3 (2019) 20.

[64] A.C. Dippel, H.-P. Liersch, J.T. Delitz, P. Walter, H. Schulte-Schrepping, O.H. Seeck, H. Franz, Beamline P02.1 at PETRA III for high-resolution and high-energy powder diffraction, J. Synchrotron Radiat. 22 (2015) 675.

[65] A.A. Benzerga, T. Pardoen, Failure of metals I, A.C. Pineau, A.A. Benzerga, T. Pardoen, Failure of metals I, Springer, Berlin, Heidelberg, 2013, pp. 362–369.

[66] T. Ahmed, H.J. Rack, Phase transformations during cooling in α+β titanium alloys, Mater. Sci. Eng. A 243 (1998) 206.

[67] C.M. Cepeda-Jiménez, F. Potenza, E. Magaliní, V. Luchín, A. Molinari, M.T. Pérez-Prado, Effect of energy density on the microstructure and texture evolution of Ti-6Al-4V manufactured by laser powder bed fusion, Mater. Charact. 163 (2020) 110238.

[68] R.S. Qin, H.K.D.H. Bhadeshia, Phase-field model study of the crystal morphological evolution of hcp metals, Acta Mater. 57 (2009) 3392.

[69] Y.-M. Kim, B.-J. Lee, M.I. Baskes, Modified embedded-atom method interatomic potentials for Ti and Zr, Phys. Rev. B 74 (2006) 14101.

[70] K. Manuda-Jindo, S.R. Nishitani, V. Van Huneg, Hcp-bcc structural phase transformation of titanium: analytic model calculations, Phys. Rev. B 70 (2004) 184122.

[71] R.G. Hennig, T.J. Lenosky, D.R. Trinkle, S.P. Rudin, J.W. Wilkins, Classical potential describes martensitic phase transformations between the α, β, and ω titanium phases, Phys. Rev. B 78 (2008) 144122.

[72] R.G. Hennig, T.J. Lenosky, D.R. Trinkle, S.P. Rudin, J.W. Wilkins, Classical potential describes martensitic phase transformations between the α, β, and ω titanium phases, Phys. Rev. B 78 (2008) 144122.

[73] R.G. Hennig, T.J. Lenosky, D.R. Trinkle, S.P. Rudin, J.W. Wilkins, Classical potential describes martensitic phase transformations between the α, β, and ω titanium phases, Phys. Rev. B 78 (2008) 144122.

[74] R.G. Hennig, T.J. Lenosky, D.R. Trinkle, S.P. Rudin, J.W. Wilkins, Classical potential describes martensitic phase transformations between the α, β, and ω titanium phases, Phys. Rev. B 78 (2008) 144122.