Fatigue Improvement of Additive Manufactured Ti–TiB Material through Shot Peening

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Abstract: In this work, fatigue improvement through shot peening of an additive manufactured Ti–TiB block produced through Plasma Transferred Arc Solid Free-Form Fabrication (PTA-SFFF) was investigated. The microstructure and composition were explored through analytical microscopy techniques such as scanning and transmission electron microscopy (SEM, TEM) and electron backscatter diffraction (EBSD). To investigate the isotropic behavior within the additive manufactured Ti–TiB blocks, tensile tests were conducted in longitudinal, diagonal, and lateral directions. A consistent tensile behavior was observed for all the directions, highlighting a nearly isotropic behavior within samples. Shot peening was introduced as a postmanufacturing treatment to enhance the mechanical properties of AM specimens. Shot peening led to a localized increase in hardness at the near-surface where stress-induced twins are noted within the affected microstructure. The RBF-200 HT rotating-beam fatigue machine was utilized to conduct fatigue testing on untreated and shot-peened samples, starting at approximately 1/2 the ultimate tensile strength of the bulk material and testing within low- (<10^5 cycles) to high-cycle (>10^5 cycles) regimes. Shot-peened samples experienced significant improvement in fatigue life, increasing the fitted endurance limit from 247.8 MPa for the untreated samples to 318.3 MPa, leading to an increase in fatigue resistance of approximately 28%.

Keywords: additive manufacturing; fatigue; Ti–TiB; titanium matrix composite; shot peening

1. Introduction

Titanium (Ti) is of interest in materials research for its complex microstructural properties and alloying capabilities, making it a particularly attractive candidate in light-weighting research due to its high strength, low density, and excellent fatigue strength, particularly for applications requiring corrosion resistance [1,2]. However, limitations on its use due to its relative cost and poor manufacturability relative to other materials [3,4] are drawing manufacturers towards alternate manufacturing methods such as additive manufacturing (AM) to produce Ti parts [5,6]. The capabilities of near-net-shape production through AM processes for Ti parts offers opportunities for machining and reduction in part design lead time, in addition to providing more design freedom than conventional subtractive machining, which leads to cost savings [6]. This is particularly relevant for aerospace [7] and biomedical fields [8] looking to exploit new precise AM methods to produce designs with internal structures and complex shapes that are extremely difficult to manufacture through conventional means.

With flexible powder-based AM strategies becoming more readily available, the production of Ti matrix composites (TMCs) through AM options can be used to fabricate enhanced Ti–TiB materials [9–12]. In Ti alloys, TiB is used as a cost-effective particle-reinforcement agent used to stiffen and strengthen components drastically in low added concentrations [13,14]. As a particle reinforcement additive, TiB makes a strong candidate for its excellent crystallographic compatibility within alpha hexagonal-closed packed Ti structures with TiB’s tendency to form sharp, hexagonal cross-sectioned needles or...
platelets [14,15]. Moreover, in situ AM formation, TiB’s similar coefficient of thermal expansion and density to Ti make it a desirable additive agent [16]. While new AM methods present novel means of producing Ti–TiB parts, more investigation is required to fully understand the material properties and related mechanical characteristics of produced AM parts [12,17,18]. Particularly, the layer-by-layer build strategy used in AM processes has been shown to lead to microstructural anisotropic tendencies in parts produced through laser energy deposition processes [19,20]. Researchers need to better understand how these microstructural anisotropic properties of AM-produced parts influence mechanical properties of samples [21], such as tensile and fatigue strength. For AM samples, build density, quality, and surface roughness are key factors to mechanical success, which can influence fatigue improvement either positively or negatively [22].

This investigation focuses on the tensile and fatigue properties of a unique alpha Ti–TiB material fabricated through the novel Plasma Transferred Arc Solid Free-Form Fabrication (PTA-SFFF) AM process. While the untreated mechanical characteristics of as-produced PTA-SFFF Ti–TiB samples have recently been explored [23], the potential directional mechanical behavior of the AM samples has not been fully characterized. Moreover, further fatigue enhancement through surface treatment such as shot peening has not been probed as an effective surface treatment for these AM specimens, which can result in significant improvements in fatigue behavior in Ti alloys [24,25]. This research provides better insight on the fatigue behavior patterns observed, considering the influence of shot-peening and how microstructurally induced changes influence improved fatigue behavior exhibited in these unique samples. Characterization through scanning electron microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS), electron backscatter diffraction (EBSD), focused ion beam (FIB), and transmission electron microscopy (TEM) is presented in this work to correlate tensile and fatigue testing.

2. Experimental Procedure and Materials

2.1. Material & Microstructural Characterization

As-received nearly commercially pure alpha Ti AM blocks with low amounts of TiB particles (<1 wt% boron) produced with the PTA-SFFF process are studied within this manuscript. Previously reported inductively coupled plasma optical emission spectroscopy (ICP-OES) results highlight that samples have a mass percent of 97.3% Ti, 0.82% Al, 0.63% B, 0.37% Si, and 0.20% Fe [23]. Microstructural analysis of the as-received and as-fractured surfaces was performed using the FEI Quanta 200 field emission gun (FEG) Scanning Electron Microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS), electron backscatter diffraction (EBSD), focused ion beam (FIB), and transmission electron microscopy (TEM) is presented in this work to correlate tensile and fatigue testing.

2.2. Tensile Testing

The tensile mechanical behavior of as-received AM blocks was investigated to evaluate the directional behavior of the layer-by-layer deposited PTA-SFFF Ti–TiB samples. ASTM Test Method E8/E8M [26] was consulted, as suggested by the ASTM F3122-14 Standard Guide [27], where, for evaluation, a 150-kN maximum load MTS Criterion Series Universal Testing Machine equipped with a 25-mm gauge extensometer at a crosshead speed of 0.05 mm/min was used. Due to the lateral dimensional restrictions of the AM blocks, subsize tensile samples were produced with shortened grip ends of 22 mm. This was
found to be suitable given the gripping capability of the tensile testing machine used and the success of a preliminary testing trial. Three tensile sample types were produced through wire electrical discharge machining (EDM) from different directions from AM blocks (Figure 1), which included (1) “longitudinal” samples machined along the AM build or z-direction, (2) “diagonal” samples machined from a 45° angle from the AM build direction, and (3) “lateral” samples machined perpendicularly to the AM build direction. Four samples from each of these directions were considered for testing and were cut from the lower half of the same PTA-SFFF AM block. All samples were ground to a 600 P grit scheme to remove surface impurities and oxides obtained during the wire EDM process.

![Figure 1](image1.jpg)

Figure 1. PTA-SFFF Ti–TiB AM block highlighting sample directions considered for tensile testing. Produced AM blocks were approximately 22 cm by 10 cm by 5 cm in size.

2.3. Fatigue Testing

To prepare fatigue samples, square cross-sectional pieces were cut using a wire EDM from PTA-SFFF Ti–TiB blocks in the longitudinal direction and then milled to the specified dimensions (Figure 2). Untreated PTA-SFFF Ti–TiB samples were ground using an incremental grit scheme and polished using 3 μm and 1 μm diamond polishing solutions to a mirror finish. For the samples treated with the previously described shot-peening treatment, shop-peened surfaces were lightly ground to 600 P and 800 P and polished with 3 μm and 1 μm diamond polishing suspensions to attain a mirror finish. This improved the surface roughness as well as removed most oxides and contaminants from the peening process.

![Figure 2](image2.jpg)

Figure 2. Fatigue sample dimensions used in testing (in mm).

Rotating bending fatigue tests were performed using the rotating beam RBF-200 HT fatigue machine under a fully reversed, pure bending condition (R = −1) at a testing
frequency of 8000 rpm. Testing began at approximately 1/2 the measured ultimate tensile strength. The applied stress was either increased or decreased in intervals of 25 MPa, ensuring that 10–20 samples would fail between the $10^4$ low-cycle regime and the $10^7$ high-cycle regime. Testing was terminated if a sample reached $10^7$ cycles and did not fail, which is considered run-out or infinite life in this scheme. Once this occurred, stress intervals were decreased to 12.5 MPa to get a better insight into the fatigue endurance limit. Testing concluded once three samples were able to hit $10^7$ cycles and not fail. Both the highest stress at run-out and a fitted endurance limit are reported as an approximated value to the fatigue endurance limit of these samples. The stress-life or S–N curves were constructed to evaluate the fatigue behavior of the samples and compare the two sample types.

2.4. Shot-Peening Surface Treatment

Shot-peening treatment was conducted at an industrial facility, where the total circumference of fatigue samples was shot-peened for 100% coverage from a ground surface condition of 600 P based on work produced by Farokhzadeh et al. [28]. Samples were blasted for 2 min in an air-powered system at 90 psi (~621 Pa or 6.2 bar) with S-550 grade steel shots with an average diameter of 1.05 mm. Surface roughness before and after shot peening was evaluated at three different area locations (area size of $1000 \times 1393.7 \, \mu m^2$) using the Keyence VK-X100 Series 3D Laser Scanning Microscope.

3. Experimental Results
3.1. Microstructure Analysis

The as-received PTA-SFFF blocks feature fine, needlelike TiB particles throughout an alpha Ti structure (Figure 3). The TiB particles appear generally homogeneously distributed throughout the structure, with no observable AM directional dependence. Using the particle analysis function in ImageJ, particle sizes of the TiB structures were analyzed in the longitudinal and lateral directions and are presented in Table 1, supporting this claim. From the micro and macrostructure of the samples, no voids or unmelted regions were noted in the produced AM samples. Overviewing the etched microstructure, AM bead interfaces were observed to be indistinguishable from the rest of the structure and grain boundaries were seen to cross AM bead interfaces. No signs of twins or other deformation features within the structures were evident in the untreated etched microstructure.

![Figure 3. (a) Secondary electron (SE)-SEM image highlighting the typical microstructure of PTA-SFFF samples studied in this research, etched using Kroll’s reagent, which features alpha (\(\alpha\)) grain Ti with needlelike TiB particles. (b) Higher magnification image of the etched microstructure, representatively highlighting the untreated subsurface of the samples.](image-url)
Table 1. Average TiB analysis distribution summary obtained from three SEM micrographs at 200× magnification (regions approximately 685 µm by 590 µm in size).

| Sample Microstructure Orientation | Percent Area Covered by TiB Particles (%) | Aspect Ratio (Height/Width) | TiB Width (µm) | TiB Height (µm) | TiB Size (µm²) |
|----------------------------------|----------------------------------------|---------------------------|---------------|----------------|---------------|
| Longitudinal                     | 9.2 ± 0.7                              | 3.3 ± 1.4                 | 1.6 ± 0.7     | 4.6 ± 2.2      | 6.2 ± 1.6     |
| Lateral                          | 9.2 ± 1.4                              | 3.3 ± 1.5                 | 1.5 ± 0.9     | 4.5 ± 1.7      | 6.6 ± 1.2     |

FIB was used to prepare a TEM lamellae cross-section that intersected TiB particles along their widths (Figure 4). TEM imaging (Figure 5) highlights that no gaps or cracking was observed around TiB particles within the Ti structure. High-resolution TEM imaging shows the interface of the Ti matrix and a TiB particle (Figure 5b), highlighting a coherent interface between the Ti matrix and a TiB particle. Electron energy loss spectroscopy (EELS) was used to confirm that TiB and Ti were observed in the structure and line scans in TEM were further employed to study the Ti–TiB interface. These results showed no noticeable gaps between the two phases (Figure 6), and thus, further confirms the coherent interface as qualitatively seen in Figure 5.

Figure 4. SE-SEM image showing the focused ion beam (FIB) cross-section of the AM Ti–TiB sample.

Figure 5. (a) Transmission electron microscopy (TEM) image taken from the enclosed dashed region highlighted in Figure 4. (b) High-resolution TEM image featuring a Ti–TiB interface in the material shows a coherent Ti–TiB boundary in the material, taken from the enclosed dashed rectangle region highlighted in (a).
Figure 5. (a) Transmission electron microscopy (TEM) image taken from the enclosed dashed region highlighted in Figure 4. (b) High-resolution TEM image featuring a Ti–TiB interface in the material shows a coherent Ti–TiB boundary in the material, taken from the enclosed dashed rectangle region highlighted in (a).

Figure 6. (a) High-angle annular dark-field STEM image highlighting the interface of the Ti matrix and a TiB particle. (b) Corresponding EELS mapping overlay from the region indicated by the Spectrum Image box, with Ti shown in green and B in yellow. (c) Line scan across the Ti–TiB interface highlighting a sharp interface with no voids or discontinuities in the material at the boundary, taken from the line indicated in (a).

3.2. Tensile Testing

Table 2 summarizes average tensile testing results obtained in each direction tested, where values reported are similar and within range to each other, highlighting nearly isotropic behavior between sample orientations. Figure 7 shows graphically the engineering stress versus strain testing results of all twelve tensile tests conducted, with four samples tested in the longitudinal, lateral, and diagonal directions. All testing conducted showed consistency within the elastic region as well as within the plastic region, with an outlier test that can be identified for both the longitudinal and diagonal directions (Figure 7). This further supports the claim of near-isotropic behavior in the AM-produced block samples.

Similar tensile fracture behavior was noted for all samples through SEM. Thus, representatively, Figure 8 highlights the fracture surface analysis of a longitudinal sample. Overall, the surfaces were found to exhibit intermixed ductile–brittle fracture mechanisms, where ductile behavior prevailed dominantly. Two common fracture characteristics are noted, where Figure 8b highlights a largely dimpled surface morphology while Figure 8c features an area that is observed to be more cleaved in pattern, and thus, more brittle.
Table 2. Tensile result summary for longitudinal, lateral, and diagonally oriented AM Ti–TiB samples.

| Sample Orientation | Ultimate Tensile Strength (MPa) | Elastic Modulus (GPa) | Percent Elongation (%) | Percent Reduced Area (%) | 0.2% Offset Yield Strength (MPa) | Fracture Stress (MPa) |
|--------------------|--------------------------------|-----------------------|------------------------|--------------------------|---------------------------------|----------------------|
| Longitudinal       | 614.1 ± 20.3                   | 98.3 ± 7.1            | 9.5 ± 1.3              | 8.9 ± 1.0                | 480.7 ± 15.5                   | 605.3 ± 17.4         |
| Lateral            | 609.2 ± 7.4                    | 97.4 ± 3.9            | 8.1 ± 0.8              | 7.1 ± 1.4                | 480.0 ± 5.3                    | 603.4 ± 7.7          |
| Diagonal           | 593.0 ± 18.8                   | 99.9 ± 2.8            | 9.3 ± 0.5              | 8.0 ± 0.4                | 460.0 ± 20.9                   | 581.7 ± 22.8         |
| Overall Testing    |                                |                       |                        |                          |                                 |                      |
| Average            | 605.4 ± 17.7                   | 98.5 ± 4.6            | 8.9 ± 1.1              | 8.0 ± 1.2                | 473.6 ± 17.1                   | 596.8 ± 19.1         |

Figure 7. Directionally oriented tensile testing results for the AM Ti–TiB material, presented in the form of engineering stress versus percent strain.

Figure 8. (a) SE-SEM image showing representative tensile fracture surface of a longitudinal tensile fracture sample, highlighting the typical fracture features observed. Higher magnification regions highlighted from enclosed areas in (a) show signs of (b) dimpled morphology and (c) a less-ductile, more-cleaved region.
3.3. Surface Study of the Influence of Shot Peening

The severe plastic deformation induced to the surface of samples from shot peening led to a change in surface roughness. The arithmetic mean height surface roughness (Ra) and the root mean square surface roughness (Rq) increased from Ra = 2.75 ± 0.64 μm and Rq = 3.46 ± 0.81 μm at the 600-P untreated condition to Ra = 6.03 ± 0.71 μm and Rq = 6.21 ± 1.06 μm at the shot-peened condition. The post-treatment mechanical grinding and polishing step introduced, described previously, led to a decrease in roughness of Ra = 3.20 ± 1.11 μm and Rq = 4.85 ± 1.60 μm for the test samples. As a comparison basis, untreated samples polished similarly up until a 1-μm diamond polishing suspension had roughness values of Ra = 2.55 ± 0.39 μm and Rq = 3.11 ± 0.45 μm.

A localized hardening effect as a result of shot peening was observed at the treated surface, with hardening effects experienced up to 1 mm in depth from the surface of coupons prepared in the same manner [29]. In our recent work, we note that the peak hardness measured at the near-surface was found to be 400.4 HV, which decreased until a depth of 1 mm was reached where the measured hardness plateaued at an average value of 286.6 ± 6.3 HV [29].

Shot peening was noted to result in hatchlike microstructural features and stress-induced microstructural twinning in the subsurface structure (Figure 9), which was not present in the untreated subsurface of samples (Figure 3). Figure 10a shows the results of the EBSD analysis on the shot-peened sample. The scanned area (~80 μm × 50 μm) was captured 100 μm away from the shot-peened top surface. The difference in the microstructure between the alpha matrix and TiB particles is clearly shown in the inverse pole figure (IPF) map (Figure 10b), where the alpha Ti (hcp) phase is colored in green and the TiB (cubic) phase in red. It is worth noting that the TiB particles distributed in the alpha Ti matrix have a distinct needlelike morphology.

![Figure 9](image)

Figure 9. SE-SEM image showing (a) the typical subsurface cross-section microstructure of a PTA-SFFF Ti–TiB coupon subjected to shot peening and polishing treatment, as described in Section 3.3, etched in Kroll’s etchant. (b) The higher magnification image from the enclosed box region showing microstructural hatched features due to shot peening.

To confirm the existence of deformation twinning in the shot-peened sample, the TiB phase was subtracted from the EBSD map because of the similarity in the morphology of long needlelike TiB particles and twin boundaries. When the TiB phase is subtracted from the microstructure, the mechanical twins in the alpha Ti matrix become evident in area A (Figure 10c). Two grains (labeled Area 1 and Area 2 in Figure 10c) were closely examined to confirm the existence of deformation twins. It can be seen from the IPF map as well as the image quality (IQ) map (Figure 10d) that the mechanical twins have a different crystal orientation (different color) and morphology to their surrounding matrix.
To further characterize the twins observed in the shot-peened sample, the [0001] pole figure was plotted for each of the twin/matrix sets (Area 1 and Area 2), as shown in Figure 11a,b. The misorientation between the twins and the surrounding matrix in area 1 was calculated to be $85.0 \pm 1.4^{\circ}$ $<11\overline{2}0>$ using at least 125 individual points along different regions (Figure 11c). On the other hand, the misorientation between twins and the surrounding matrix in area 2 was $83.5 \pm 1.4^{\circ}$ $<11\overline{2}0>$ (Figure 11d). This misorientation is almost identical to the tensile twin, type 1 (TT1), that is closely related to CSL $\Sigma_{11}b$ (which has an exact value of $84.8^{\circ}$ $<11\overline{2}0>$ [30–33].

![Figure 10. EBSD maps of the shot-peened Ti–TiB sample. (a) Inverse pole figure (IPF) map, (b) unique color phase map showing the difference between the alpha Ti (hcp) and TiB (cubic) phases, (c) IPF of area A showing the existence of twins, and (d) image quality (IQ) map.](image)

### 3.4. Fatigue Testing

Figure 12 features the fatigue life S–N diagram compiled from testing both untreated and shot-peened samples. Overall, the shot-peened samples performed significantly better than untreated samples, being able to sustain higher stress amplitudes at similar sample life cycles in comparison to the untreated samples. For the untreated samples, the highest stress at run-out was 282.5 MPa, whereas the fitted endurance limit was 247.8 MPa. For shot-peened samples, the highest stress at run-out was 332.5 MPa and had a fitted endurance limit of 318.3 MPa. Based on the fatigue machine used for data curation, a measurement-related standard error of $\pm 5.4$ MPa is reported with these values.

The SEM images in Figures 13–16 are used to present key patterns observed in the fracture surfaces of samples. Overall, three distinct fracture regions were identified as follows: Region I, Region II, and Final Rupture. Region I was defined as the area in which fatigue crack initiation and the initial, slower crack propagation occurred. For both untreated and shot-peened samples, Region I was found to be flat and smooth. While striations were noted within Region I, cleavage steps and facets were observed in higher presence, and thus, Region I was dominated by brittle fracture mechanisms throughout all samples. The Final Rupture region is the largest region by area for all samples studied and is where the fast fracture occurred. Generally, this area was rougher than Region I and prominently featured a dimpled morphology, and thus, was more ductile in nature. Last of all, Region II was considered the transitional region between Region I and Final
Rupture and had both brittle and ductile dominating fracture behavior patterns. For both untreated and shot-peened samples, two figures are depicted, which are chosen from the low- (<10⁵) (Figures 13 and 15) and high-cycle (>10⁵) (Figures 14 and 16) fracture life regimes from testing.

**Figure 11.** The misorientation between the deformation twins and the surrounding matrix (shown in Figure 10): (a,b) [0001] pole figures and (c,d) inverse pole figures illustrating the misorientation angle and axis pair for area 1 and area 2.

**Figure 12.** Fatigue stress amplitude versus number of cycles, S–N diagram of untreated and shot-peened PTA-SFFF Ti–TiB samples. Shot-peened samples performed significantly better than untreated samples, being able to sustain higher stress amplitudes at similar sample life cycles in comparison with the untreated samples.
Figure 13. (a) Backscattered electron (BSE)-SEM image highlighting the overall fracture surface of a PTA-SFFF Ti–TiB fatigue sample, which was tested at a stress amplitude of 307.5 MPa and failed at 105,300 cycles. (b) BSE-SEM image highlighting the fatigue crack origin and crack propagation, taken from the enclosed area in Region I highlighted in (a). (c) SE SEM image showing a dimpled fracture surface with TiB particles, taken from the enclosed area in the Final Rupture region highlighted in (a).

Figure 14. (a) BSE-SEM image highlighting the overall fracture surface of a PTA-SFFF Ti–TiB fatigue sample, which was tested at a stress amplitude of 432.5 MPa and failed at 24,600 cycles. (b) SE-SEM image highlighting the fatigue crack origin and crack propagation, taken from the enclosed area in Region I highlighted in (a). (c) SE-SEM image showing a cracked fracture surface with TiB particles, taken from the enclosed area in the Final Rupture region highlighted in (a).
Figure 15. (a) BSE-SEM image highlighting the overall fracture surface of a shot-peened PTA-SFFF Ti–TiB fatigue sample, which was tested at a stress amplitude of 382.5 MPa and failed at 506,700 cycles. (b) BSE-SEM image highlighting the fatigue crack origin area, taken from the enclosed area in Region I highlighted in (a). (c) SE-SEM image showing a cross-hatched, cracked fracture surface, taken from the enclosed area in the Final Rupture region highlighted in (a).

Figure 16. (a) BSE-SEM image highlighting the overall fracture surface of a shot-peened PTA-SFFF Ti–TiB fatigue sample, which was tested at a stress amplitude of 507.5 MPa and failed at 48,000 cycles. (b) BSE-SEM image highlighting the fatigue crack origin area, taken from the enclosed area in Region I highlighted in (a). (c) SE-SEM image showing a dimpled structure with TiB particles within dimples, taken from the enclosed area in the Final Rupture region highlighted in (a).
For untreated samples, fatigue cracks were found to originate at the surface of the tested samples, with most cracks observed to start at localized surface defects for both the low- and high-cycle fatigue regimes. Initial crack propagation was noted to occur radially from the crack initiation point (Figure 13). In Figure 14b, tested at a stress amplitude of 432.5 MPa and failing at 24,600 cycles, the initial crack propagation area was observed at the surface, where localized TiB particles were somewhat agglomerated. In the Final Rupture zone of the untreated samples, dimples with some cleavage planes are noted (Figures 13c and 14c). Figure 13c has TiB particles situated around dimpled areas and shows TiB needles, which either are seen to be whole and coherent within the Ti matrix despite fatigue failure or have broken but remain coherent to the Ti matrix, with no particle pullout observed. Figure 14c further illustrates this in a nondimpled area in the Final Rupture region, showing that TiB particles within are intact or broken, which both remain coherent in the Ti structure.

In contrast, all shot-peened AM Ti–TiB samples observed contained crack initiation sites at the subsurface level and exhibited void nucleation at these regions for both the low- and higher-cycle fatigue regimes. Initial crack propagation occurred concentrically to the crack origin regions and crack propagation occurred radially from the region observed using BSE-SEM. Both Figures 15 and 16 show this behavior in samples, where typically the crack initiation site was roughly situated between 0.7 to 1.5 mm in the subsurface of samples, with no observable dependency of its location on the applied load and cycle life. Similar to the untreated samples, the shot-peened samples contained TiB particles, which were observed to stay intact or break but both remained coherent in the structure with no particle pullout within the Final Rupture zone. This is highlighted in Figure 16c, a shot-peened sample, where small TiB particles are shown to stay coherent to the structure and are observed within dimples that are present. One key distinguishing factor between the fracture surfaces of the shot-peened samples and the untreated samples was the presence of a cross-hatched fracture surface, shown in Figure 15c. This cross-hatched fracture surface pattern was observed in all shot-peened samples, found only at the near-subsurface of the Final Fracture area.

In the testing of both sample types, it was noted that with increasing stress amplitude, going from high- to low-cycle regimes, the size of Region I and II decreased while the Final Rupture zone increased. Apart from that observation, negligible differences in the fracture behavior between the low- and high-cycle fatigue regimes were observed.

4. Discussion

In this study, the microstructural and tensile mechanical properties of as-received PTA-SFFF Ti–TiB blocks were evaluated and the fatigue behavior of samples before and after shot peening was investigated in greater detail. From the microstructural review of the as-received PTA-SFFF Ti–TiB material, the TiB particles were observed to have no directional dependence on orientation based on AM deposition. High-magnification observations made through FIB and TEM in observing the microstructure and distribution of TiB particles showed that the Ti and TiB interfaces were coherently formed (Figures 4–6). Moreover, no pores or gaps between Ti and TiB were observed in the structure, and voids from AM processing went unobserved throughout the microstructural investigation.

From the directionally oriented tensile testing results obtained summarized in Table 2 and shown graphically in Figure 7, a near-isotropic behavior within the as-received AM block structure was observed despite AM-layering strategies used to produce parts. The core tensile results shown in Table 2 feature low standard deviation values for obtained results in the three directions considered. This near-isotropic behavior in samples can be partially attributed to the fully dense build of the PTA-SFFF blocks as well as the indistinguishable AM bead boundary interface within the structure, which shows good fusion between layers. Furthermore, from fractographic studies of the tensile fracture surfaces (Figure 8), samples exhibited intermixed ductile–brittle behavior, primarily dominated by ductile fracture mechanisms despite TiB additions. Small TiB whisker additions can lead
to selective crack propagation within particles, which can inhibit fracture in samples [34]. Here, we observed TiB within these exposed surfaces appear generally intact and particle pullout was not observed. Under tensile loadings, it is likely that TiB particles helped mitigate failure, though the Ti matrix still had a more dominant role in fracture due to the ductile dimples observed.

Tensile testing results highlighted that samples had sustained ductility in all directions measured through percent elongation and percent reduction in area, with average values of $8.9 \pm 1.1\%$ and $8.0 \pm 1.2\%$, respectively, as presented in Table 2. This compares well with similar additive manufacturing processes involving commercially pure Ti [35,36], where a slight improvement in tensile strength is noted in this work with conserved ductility. Directionality of TiB particles can play a role in the strengthening effect of these additions within TMCs, where TiB behaves as discontinuous fibres within Ti alloys [15,34]. Given the near-nondirectional behavior observed in microstructure and low concentration of boron, the influence of the TiB additions was likely more subtle within the structure, where higher concentrations would expect to lead to further strengthening and ductility loss.

In fatigue testing, the shot-peened PTA-SFFF Ti–TiB fatigue samples outperformed the untreated samples, showing a much higher $10^7$ high-cycle fatigue strength with a highest value of $332.5 \pm 5.4$ MPa versus $282.5 \pm 5.4$ MPa for the untreated samples (Figure 12). The improved fatigue behavior from shot peening is attributed to the introduced compressive residual stresses, which led to localized increases of hardness at the surface of samples [29] and stress-induced cold worked microstructural changes such as deformation twins (Figures 9–11). This induced cold work resulted in the formation and growth of tensile deformation twins, which, in turn, resulted in increasing the hardness of the material even though the exact mechanism is still being contested in the scientific literature [37]. The localized increase in hardness and related microstructural cold-work changes made it more difficult for crack initiation to occur at the surface of shot-peened parts, as it occurred in untreated samples, despite their slightly rougher surface finish even after polishing in comparison with untreated fatigue samples.

For shot-peened samples, crack initiation sites were found to be approximately situated between 0.7 to 1.5 mm in the subsurface of samples (Figures 15 and 16). This is directly relevant to the influence of the shot-peening process, as untreated samples experienced crack initiation sites starting at the direct surface, likely initiated at regions with small surface defects (Figures 13 and 14). The works of Petit-Renaud highlight that shot peening introduces high amounts of compressive residual stress at or just below a shot-peened surface, which is balanced by smaller amounts of tensile stress below this compressive stress layer [38]. Similarly, Wagner and Luetjering note in their work that the observed depth of crack initiation sites could be correlated to the depth of residual tensile peak stress, where they observed that residual stresses significantly delayed crack propagation [24]. Hardening effects from the shot-peening process are reported to have an influence up to a subsurface depth of approximately 1 mm [29] (Figure 17)—as such, it is noted that fatigue crack initiation sites occurred within regions where hardening influences are minimal. Thus, crack initiation sites are believed to have occurred outside of the compressive residual stress layer introduced in samples, in the tensile-stress-balanced region. The formation of voids around crack initiation sites could also be explained by the presence of tensile stresses in the structure, as numerous voids were not observed at surface-situated crack initiation sites of untreated fatigue samples. Wagner and Luetjering note that shot-peened Ti samples with crack initiation sites at the surface due to high dislocation density would further slow crack propagation and improve fatigue life, whereas increased surface roughness would have an opposite effect [24]. This points towards a need to further explore shot-peening parameters to optimize and further improve the results obtained.
Untreated and shot-peened samples exhibited similar fracture failure mechanisms in Region I, Region II, and the Final Rupture region, apart from fatigue crack initiation sites between the two sample types and the cross-hatched microcracking fracture surface features observed specifically at the near-subsurface of shot-peened samples. This cross-hatched microcracking fracture surface behavior, exemplified in Figure 15c, can be explained due to the microstructural changes in shot-peened samples from the introduced surface treatment process. Shot peening was noted to result in microstructural twinning due to deformation confirmed from EBSD results and cross-hatched features, resembling the formation of stress-induced martensite in the near-subsurface of the samples, featured in Figures 9–11. This cross-hatched microstructural feature resembles the hatched microcracking cleaved fracture sites described (Figure 15c) and is associated with a brittlelike, transgranular fracture mechanism within the residual compressive stress layers introduced through shot peening. For both shot-peened and untreated samples, the Final Rupture regions were ductile in nature, with dimples presented throughout the region, while crack initiation and initial propagation zones were flat with observable cleavage steps, and thus, dominated by brittle fracture mechanisms. Similar features were observed in the low- and high-cycle fatigue regimes as well and both showed trends where, with decreasing stress amplitude, the size of Regions I and II were observed to increase in area while the Final Rupture zone decreased.

Overall, this work found that shot peening led to significant improvements in fatigue behavior, leading to an improvement in the endurance limit achieved. This is explained by localized hardening and microstructurally induced changes experienced at the near-surface of the PTA-SFFF Ti–TiB samples, which mitigated failure and, thus, crack propagation arising at the surface. Moreover, from discussions related to microstructural and mechanical observations, TiB particles additions had a positive influence on sample mechanical behavior, showing an excellent interfacial connection within the Ti matrix and coherence within the structure, even within fracture surfaces. From tensile testing and microstructural observation, it was noted that the PTA-SFFF Ti–TiB samples exhibited nearly isotropic behavior, despite the AM strategies used to produce the bulk material, highlighting the quality of the novel manufacturing method.

5. Conclusions

In this study, the microstructural, tensile, and fatigue properties of a Ti–TiB material produced through the AM PTA-SFFF process were investigated. The effect of microstructural changes induced by shot peening on fatigue behavior was further investigated. The following conclusions can be made from these investigations:

![Figure 17. Average microhardness profile versus depth into the sample of a shot-peened PTA-SFFF Ti–TiB coupon (red triangle) in comparison with an untreated PTA-SFFF Ti–TiB coupon (black circle). Image reproduced with permission from DiCecco et al. [29] (Copyright© 2021 SpringerNature).]
Directionally aimed tensile testing of AM PTA-SFFF blocks exhibited a near-isotropic behavior within the structure, despite AM-layering strategies used to produce samples. This was attributed to the fully dense AM-produced samples (or blocks) and the proper fusion of AM layers.

Overall, the fatigue performance of shot-peened samples was superior to untreated samples in both the low- and high-cycle fatigue regimes. The $10^7$ high-cycle fitted fatigue endurance of shot-peened samples was found to be 318.3 MPa versus 247.8 MPa for the untreated samples, leading to an increase in fatigue resistance of approximately 28%. This improvement in mechanical behavior is attributed to the localized surface hardening and related microstructural changes from shot peening, which helped mitigate and impede crack initiation and initial crack propagation.

For untreated AM Ti–TiB samples, fatigue cracks were found to originate at the surface of fatigue samples, where surface defects such as microscratches were the likely cause of crack initiation.

Shot-peened AM Ti–TiB samples contained crack initiation sites at the subsurface and exhibited void nucleation at these regions, which were approximately situated 0.7 to 1.5 mm in depth from the surface of the samples. Based on similar observations in literature [24,38], this is attributed to tensile residual stresses in the area below the compressive residual stresses introduced through shot peening.

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