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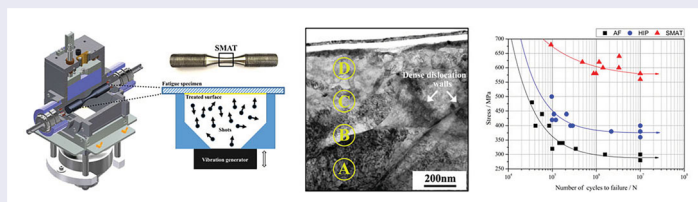
Fatigue strength improvement of selective laser melted Ti6Al4V using ultrasonic surface mechanical attrition

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ABSTRACT

Ultrasonic surface mechanical attrition treatment (SMAT) was employed to modify the surface microstructural layer of SLM Ti6Al4V ELI biomedical material to improve the fatigue performance. The SMAT method can introduce a nanostructured layer in the SLM sample surface by imposing high-strain-rate plastic deformation. The nanostructured layer improves the mechanical strength of the SMAT-affected zone and induces compressive residual stress parallel with the surface which suppress the initiation of cracks. As a result, the specimen after SMAT exhibits significantly higher fatigue strength than the non-treated sample in both low- and high-cycling regime.



IMPACT STATEMENT

This research demonstrates that surface mechanical attrition treatment (SMAT) is a promising post-processing technology for selective laser melting. SMAT improves the fatigue strength of selective laser melted Ti6Al4V by 100%.

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KEYWORDS

Selective laser melting (SLM); additive manufacturing; nanocrystalline; fatigue resistance; TEM characterisation

Selective laser melting (SLM) is an additive manufacturing technology which has been predominantly used for the production of near-net-shape metal-based components with complex geometry and high dimensional accuracy. Ti and Ti alloys have been widely used in biomedical industry due to their excellent mechanical and biological properties. Ti6Al4V, as one of the most used Ti alloys, is a candidate material of high interest for SLM and has been intensively studied in the past decade. Nevertheless, studies have shown that SLM Ti6Al4V has less favourable fatigue performance as compared with conventionally manufactured counterparts due to the inherent defects, tensile residual stress and rough surface, which significantly limits its wide use in the biomedical field where long-term cyclic loading is applied [1].

Currently, the most reliable way to improve the fatigue resistance of SLM Ti6Al4V is hot isostatic pressing (HIP) treatment. The defects and pores in as-fabricated (AF) SLM parts can be significantly reduced after HIP and therefore the fatigue resistance dramatically improves to nearly the same level as the wrought counterparts [2]. In addition to HIP treatment, various post-treatment methods were applied to improve the fatigue resistance of SLM Ti6Al4V in recent years, including electro-polishing [3], shot peening [4], heat treatment [5], ultrasonic nanocrystal surface modification (UNSM) [6]. It has been reported that reducing surface roughness by electro-polishing helps to improve the fatigue resistance of SLM Ti6Al4V due to mitigated notch effect. However, deep surface features on the sample cannot

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be fully removed using electro-polishing, and hence the fatigue performance improvement is limited. Shot-peening improves the fatigue resistance of SLM Ti6Al4V through introducing a compressive residual stress layer to the sample surface [4]. However, shot-peening results in a rough surface which is detrimental to the fatigue resistance. Heat treatment also leads to improved fatigue performance due to the formation of α and β phases and increased ductility [5]. However, heat treatment cannot close the inherent pores inside the sample; thus the fatigue performance improvement is also bounded. UNSM is a technique that can also be used to improve the fatigue strength of additively manufactured materials. The improvement is achieved through severe plastic deformation of a metal surface layer which is induced by a hard tip under ultrasonic frequency vibration [6]. UNSM is powerful in treating simple structures (e.g. flat surface and cylinder) but not quite suitable when dealing with complex structures (e.g. light-weight structure).

Surface mechanical attrition treatment (SMAT) is an emerging post-treatment technology used to introduce a nanostructured layer to metal surface and improve the fatigue resistance of the metal [7]. In this process, small particles repetitively impact on a metal surface using an ultrasonic transducer to accelerate the projectiles. The resultant high-strain-rate deformation causes nanocrystallization in the surface. The nanostructured layer can thus have superior fatigue resistance as compared to the coarse-grained counterparts. As compared with shot-peening and UNSM methods, SMAT provides more precise control on the processing parameters and is suitable for treating complex structures, respectively. Therefore, this research will apply SMAT to SLM parts for the first time with the aim of improving fatigue performance.

The flowchart of the SLM sample preparation and SMAT process is provided in Figure 1. Spherical Ti6Al4V ELI (EOS GmbH, Germany) powders with the average diameter of 35 μm were used as the feedstock. EOS M290 SLM system (EOS GmbH, Germany) was used to fabricate the cylinder Ti6Al4V samples. The manufacturing parameters were set as: laser power of 280 W, scanning speed of 1.2 m/s, laser beam focal diameter of 100 μm , layer thickness of 30 μm and hatch distance of 50 μm [2]. All samples were built in a cylindrical shape with a diameter of 14 mm and a height of 85 mm as shown in Figure 1(a). The AF samples were post-treated with HIP at 900°C and 120 MPa for 2 h as shown in Figure 1(b) following manufacture. The HIP samples were then machined into threaded cylindrical fatigue specimens according to ASTM E606 standard as shown in Figure 1(c). SMAT was then carried out on the surface of HIP fatigue specimens as shown in Figure 1(d).

Spherical balls (304 stainless steel + ZrO_2) with a diameter of 3 mm were accelerated under an ultrasonic frequency (20 kHz) for 1 h. The average roughness (R_a) of the surface was measured to be $1.07 \pm 0.53 \mu\text{m}$. The X-ray micro-computed tomography ($\mu\text{-CT}$, X5000, North Star Imaging, USA) indicates that the surface layer of the SMAT fatigue specimen is smooth and intact without any damage as shown in Figure 1(e).

The AF and HIP samples were examined by an X-Ray diffractometer (XRD, Siemens D500, Germany) with the Co ($\lambda = 1.789 \text{ \AA}$) source. Electron backscatter diffraction (EBSD) was used to further characterise the sample grain structure and phase composition. The nanostructure of the SMAT fatigue specimen was characterised by transmission electron microscopy (TEM, FEI Titan Themis 200, US). High frequency (130 Hz) cycle fatigue test at zero mean stress and load ratio of $R = -1$ were carried out at ambient temperature using a resonant test machine (QBG-100, China). The load direction was aligned with the build direction of the SLM samples. Run-out fatigue tests were terminated at 10^7 cycles to minimise the time and cost of the experiment. For comparison, the fatigue properties of the AF and HIP samples were also studied. A total of 15 specimens were tested for each group. The fracture morphologies were examined by scanning electron microscope (SEM, FEI Nova NanoSEM 450, US). The nanohardness of the SMAT-affected zone and non-affected zone were measured by nanoindenter (XP, MTS, USA) based on 10 measurements for each.

Figure 2 shows the XRD spectrum and EBSD characterisation of the AF and HIP samples. The XRD spectra demonstrate that the AF sample is composed of α phase, part of which transforms into β phase after HIP. The inverse pole figures (IPFs) shown in Figure 2(b) and e further indicate that the AF sample is characterised by very fine, low-ductility acicular α' martensitic phase due to the extremely high cooling rate in the molten pool, while the acicular α' martensitic phase transforms into a mixture of uniformly distributed lamellar α and β phases after the HIP treatment. The average grain sizes are $1.03 \pm 0.27 \mu\text{m}$ and $1.63 \pm 0.34 \mu\text{m}$ for the AF and HIP samples, respectively. The EBSD phase maps shown in Figure 2(c) and (f) further reveal the distribution of the hexagonal close-packed α/α' phase (coloured in red) and the body-centred β phase (coloured in green). Due to the high cooling rate experienced by the solidifying powder bed during SLM, tiny β phase is scattered throughout the matrix of the AF sample as shown in Figure 2(c). The β phase in the AF sample is not detected through XRD due to their extremely low content. In the HIP sample, a small amount of β phase exists along the α grain boundaries as shown in Figure 2(d).

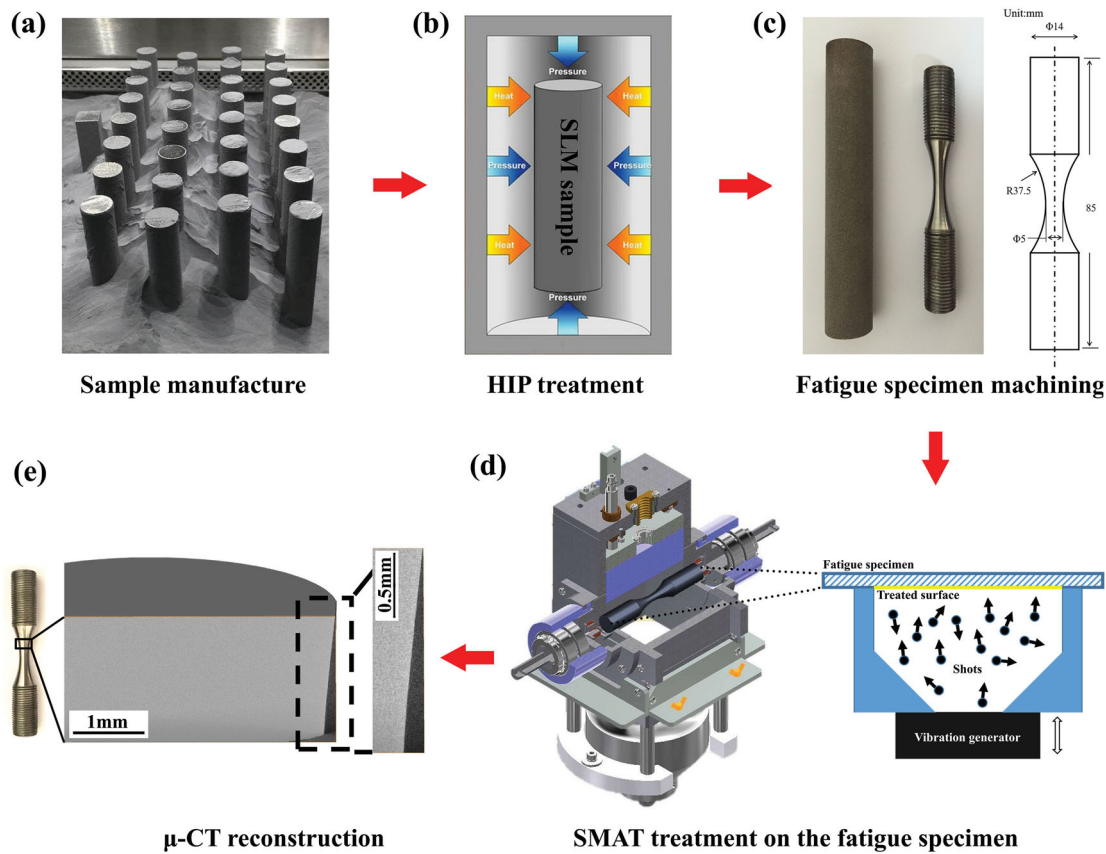


Figure 1. Flowchart of the SLM Ti6Al4V fatigue specimen preparation and SMAT processes. (a) photograph of the AF Ti6Al4V samples, (b) schematic of HIP treatment, (c) photograph of a fatigue specimen, (d) schematic of SMAT process, and (e) μ -CT reconstruction result showing the specimen surface after SMAT.

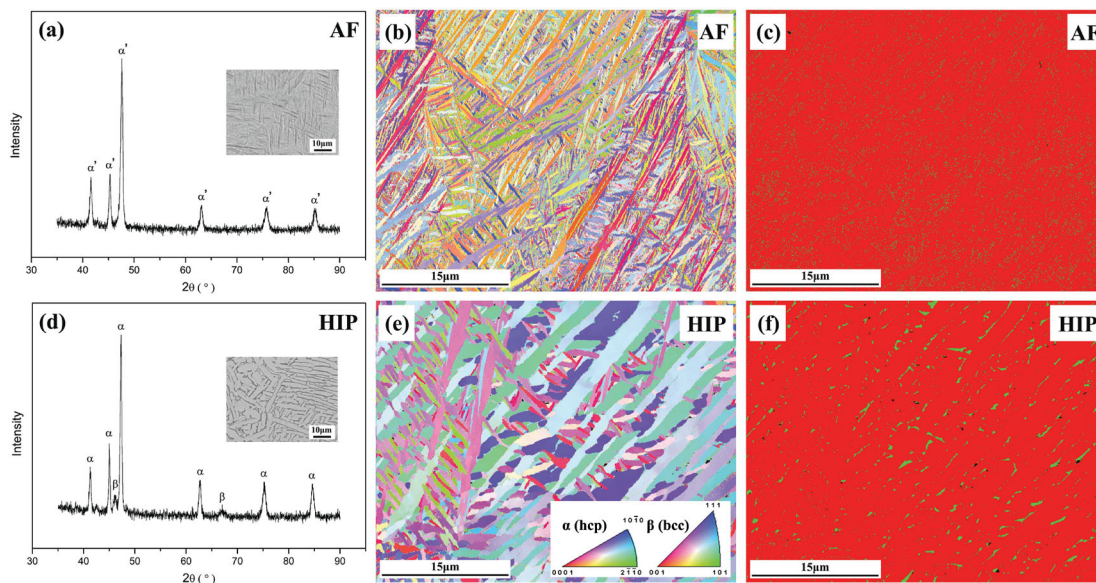


Figure 2. XRD spectrum and EBSD mapping of the SLM Ti6Al4V. (a) AF XRD spectrum, (b) AF IPFs, (c) AF phase mapping, (d) HIP spectrum, (e) HIP IPFs, and (f) HIP phase mapping. In the phase mapping, red colour indicates the α/α' phase and green colour indicates the β phase.

Figure 3 shows the cross-sectional TEM images of the SLM Ti6Al4V sample after SMAT. In the bright field TEM (BF-TEM) image as shown in Figure 3(a), the SMAT

sample can be divided into two regions: SMAT affected zone and non-affected zone. In the non-affected zone (i.e. approximately 1.5 μm below the surface), grains remain

coarse with the grain size of larger than $1\text{ }\mu\text{m}$, which is comparable to the grain size of the HIP sample. This phenomenon indicates that the microstructure in the non-affected zone has not been affected after SMAT but is in consistent with that in the HIP sample. However, in the SMAT-affected zone, grains experienced significant refinement due to the SMAT-induced plastic deformation. The high-magnification BF-TEM image of the selected region in Figure 2(b) shows dense dislocation walls (DDWs) in the SMAT affected zone, which is clear evidence of severe plastic deformation. To investigate the nanostructure in the SMAT-affected zone further, four characteristic regions from 'A' to 'D' as marked by yellow circles in Figure 2(b) are chosen for high-resolution TEM (HRTEM) characterisation. Note that the markers 'A' to 'D' were only applied to demonstrate the depth of different characteristic regions below the surface; the HRTEM images were taken within these regions but not in the exact place as marked in the figure.

In the 'A' region (i.e. 660 nm below the surface), the HRTEM image shows the formation of hexagonal close-packed (hcp) α phase. The well-defined SAED pattern indicates a coarse-grain structure, which means that the 'A' region is not significantly affected by SMAT and experienced only slight plastic deformation. In the 'B' region (i.e. 460 nm below the surface), the HRTEM image shows the formation of disordered atoms in some areas, and the SAED pattern confirms the co-existence of both coarse grains and fine subgrains with the size of a few hundred nanometres. This suggests that the 'B' region has a finer grain structure than that in the 'A' region. In the 'C' region (i.e. 270 nm below the surface), SMAT causes more severe plastic deformation than in the 'B' region. The formation of the DDWs as shown in Figure 3(b) indicates localisation of severe plastic deformation, which normally appears at large plastic deformation stage [8]. In the 'C' region, the HRTEM image shows an even higher dislocation density than in the 'B' region, and the inserted dark-field TEM (DF-TEM) image shows the formation of subgrains with size of approximately 100 nm. The SAED pattern suggests that the grain structure in the 'C' region is further refined as compared with the 'B' region. In the 'D' region (i.e. 130 nm below the surface) where the material experienced the most intensive plastic deformation, high-density dislocation tangles are observed. Equiaxed nanocrystalline grains (less than 100 nm) with high-angle boundaries were formed as can be seen from the inserted DF-TEM image due to further enhanced dislocation accumulation [9]. The full-ring SAED pattern confirms the formation of highly refined nanoscale grains distributed in random crystallographic orientation. Based on the TEM characterisation, it can be concluded that the SMAT method can result in

the nanocrystallization of the surface layer of the HIP SLM Ti6Al4V. A grain structure transition of 660 nm thick from the microcrystalline to nanocrystalline and increased dislocation density can be found along the direction towards to the surface (from the 'A' region to the 'D' region) due to the gradually enhanced plastic deformation.

The fatigue properties of the SMAT specimen are provided in Figure 4. Figure 4(a) shows the S-N curves of the SLM Ti6Al4V fatigue specimens under AF, HIP and SMAT conditions. Note that only less than 15 data can be seen from Figure 4(a) because some data overlapped with each other at the fatigue limit. In the LCF regime, the ultimate fatigue strength of the SMAT specimens is approximately 675 MPa at the cycling number of 10^5 , which is 93% higher than that of the AF specimens (350 MPa) and 50% higher than that of the HIP specimens (450 MPa). In the HCF regime, the ultimate fatigue strength of the SMAT specimens reaches 580 MPa, which is twice of that of the AF specimens (290 MPa) and 57% higher than that of the HIP specimens (370 MPa). The figure strength obtained in this work is also higher than those obtained through other surface treatment technologies under comparable testing conditions such as mechanical polishing (350 MPa) and shot-peening (370 MPa) [4]. The test results clearly indicate that SMAT significantly improves the fatigue resistance of the SLM Ti6Al4V in both LCF and HCF regimes.

It is known that reduced porosity and enhanced ductility will tend to result in improved fatigue resistance [4]. For the AF specimen, internal pores are inevitable due to the presence of unmelted powders and the balling phenomenon, which occur during SLM processing. Cracks tend to initiate from internal pores due to the stress concentrations from these defects [1], which results in lower fatigue strength. For the HIP specimen, the internal defects are mostly removed, and the material ductility is improved [2] due to the formation of the α and β phases as discussed in Figure 2. In this work, the pore size and porosity of the AF specimens were found to be $34.31\text{ }\mu\text{m}$ and 0.024% using μ -CT scanning, respectively. Note that the resolution of the CT machine is $4\text{ }\mu\text{m/voxel}$, which means that any pores with the size larger than $4\text{ }\mu\text{m}$ are taken into account for porosity analysis. After HIP treatment, porosity was effectively removed. Hence, the fatigue strength of the HIP specimen is also higher than that of the AF specimen. For the SMAT specimen, a significant improvement of the fatigue strength can be attributed to three reasons. Firstly, nanocrystallization in the surface layer results in improved hardness ($4.6 \pm 0.21\text{ GPa}$ in the SMAT-affected zone against 3.60 ± 0.17 in the non-affected zone) and mechanical strength due to the increased grain boundaries and

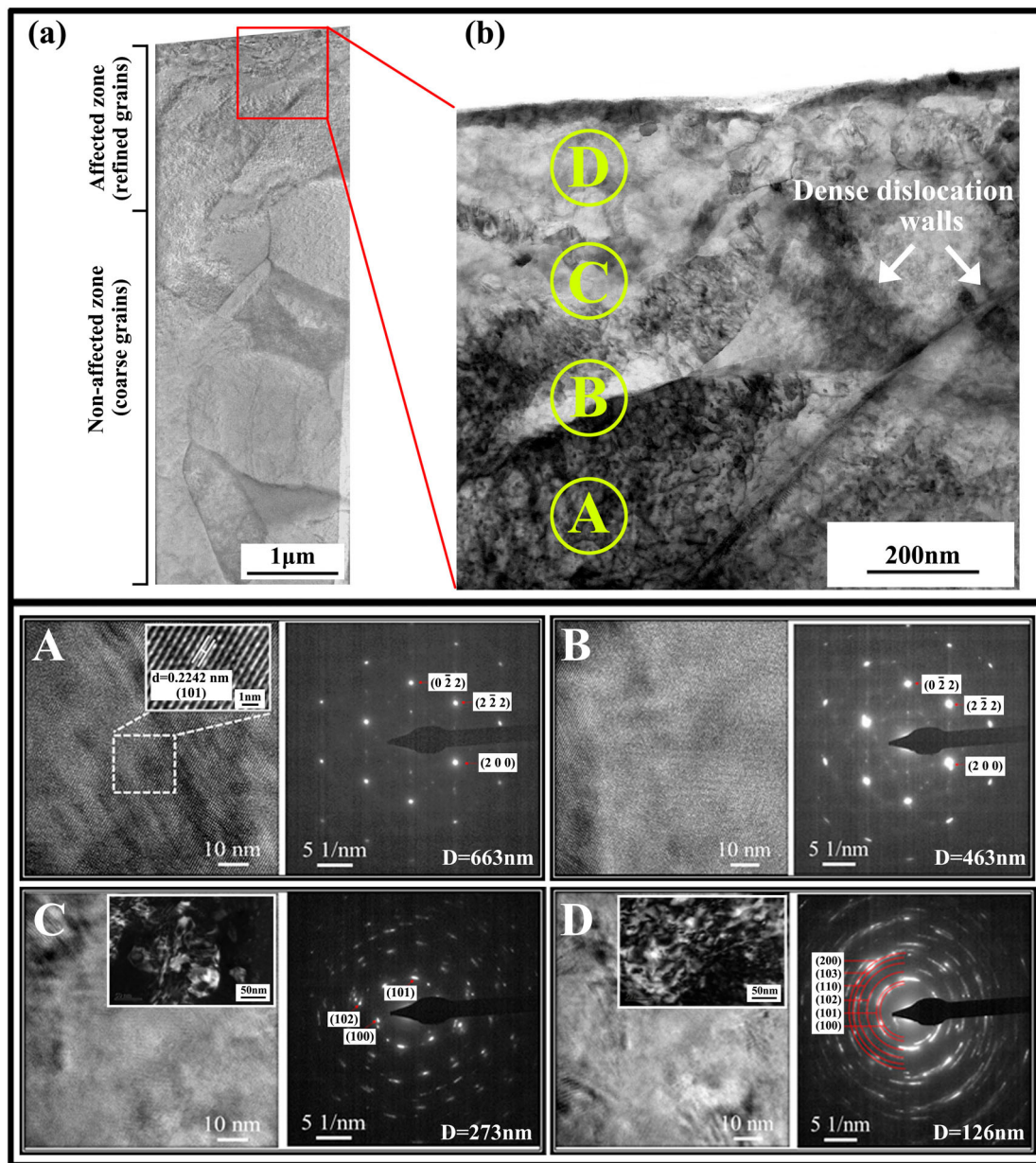


Figure 3. TEM characterisation of the HIP-SMAT sample: (a) overall TEM image of the HIP-SMAT fatigue specimen; (b) BF-TEM image of the selected surface area.

dislocation densities as addressed by Hall-Patch law [10]. Secondly, the increased amount of grains due to nanocrystallization leads to more randomly distributed crystallographic texture in the SMAT-affected layer as shown in Figure 3(d), which helps to relieve the stress concentration [11]. Thirdly, similar to shot peening processes, SMAT also results in a compressive residual stress in the surface layer, which can suppress the initiation of crack and thus partly contributes to the improvement of fatigue strength [12,13]. The combination of the three aspects leads to the delay of fatigue crack initiation which accounts for 90% of the entire fatigue life and thus improves the fatigue strength.

To study the fracture mechanism of the SMAT specimen, Figure 4(b–d) shows the fractography of a SMAT specimen fractured at the stress $\sigma = 600$ MPa and $N_f = 1.12 \times 10^6$ cycles. The fracture surface can be divided into three regions: crack initiation region (Region I), crack propagation region (Region II), and instantaneous rupture region (Region III). In the Region I, it can be observed that the fatigue crack initiates from the inside of the specimen (at the subsurface layer of 470 μm below the surface) as shown in Figure 4(b). This may primarily due to the relief of stress concentration in the nanostructured layer as a result of the randomly distributed crystallographic texture. Additionally, higher hardness

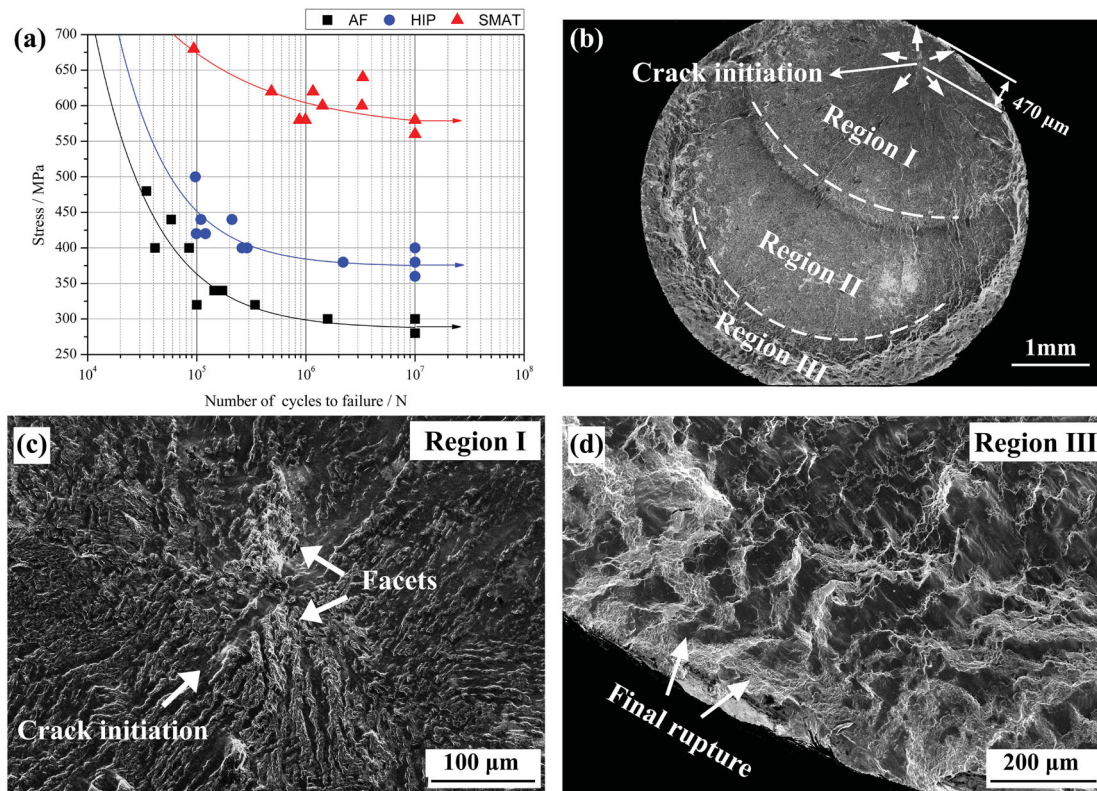


Figure 4. Fatigue properties of the SMAT specimen. (a). S-N curves of the fatigue specimens under AF, HIP and SMAT conditions, (b) morphology of the fractured surface of a SMAT specimen, (c) high-magnification view of Region I, and (d) high-magnification view of Region III.

enhances the crack initiation resistance, which partially contributes to the subsurface crack initiation as well [14]. Compressive stress parallel to the surface can also prevent the crack initiation in the nanostructured layer [15]. The combined action of the three factors surpasses the crack initiation in the nanostructured layer thus pushes the crack source to the subsurface layer. Similar subsurface crack initiation has also been reported in previous works where different surface modification methods were employed [14,16–18]. The magnification of Region I shown in Figure 4(c) reveals that the crack propagates toward the surface and the centre of the specimen with an apparent radiolitic morphologic texture. Facets are observed at the crack initiation point, which arise from the cleavage of the α phase [14]. In the Region II, river-like ridges and a flat area covered with fine fatigue striations can be observed, showing a quasi-cleavage fracture feature [19]. As the crack further propagates from the Region II to the Region III, the roughness of the fracture surface increases due to the increased density of grain boundaries and compressive stress introduced by SMAT, and eventually an instant rupture occurs as shown in Figure 4(d). In contrast to the SMAT sample

investigated in this work, our previous study has shown that crack initiated at the surface layer of AF and HIP samples rather than subsurface surface layer due to the absence of nanostructured layer and then radially propagated towards the central area due to the formation of slip bands by a mixed mechanism of intrusion/extrusion during continuous cyclic loading [2].

In summary, a nanostructured layer consisting of a nanocrystalline grain layer, a subgrain layer and a co-existence layer of fine and coarse grains is generated on the surface of the SLM Ti6Al4V fatigue specimen after SMAT. This nanostructured layer can improve the sample fatigue resistance in comparison with the AF counterpart in both LCF (675 MPa vs 350 MPa) and HCF regime (580 MPa vs 290 MPa) due to the relief of stress concentrations, improved mechanical strength and compressive residual stress in the surface layer. As compared with other surface treatment technologies, SMAT demonstrates superior fatigue improvement, provides better surface quality and is suitable for processing parts with complex structure. Taken overall, SMAT is a promising surface treatment process for fatigue strength improvement of SLM materials.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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