

INITIAL RESEARCH INTO THE FATIGUE BEHAVIOR OF ADDITIVELY PROCESSED BETA TITANIUM ALLOYS PRODUCED FROM RECYCLED FEEDSTOCK

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Abstract

Beta titanium alloys are promising materials for biomedical and aerospace applications due to their low elastic modulus, high strength, and biocompatibility. For such applications, however, a thorough understanding of their fatigue behavior is essential. This study provides an initial insight into the fatigue properties of two additively processed beta titanium alloys (Ti-13Nb-13Zr and Ti-15Mo) produced from recycled feedstock. Both alloys were manufactured by recycling titanium scrap (offcuts and similar waste), followed by powder preparation for 3D printing of fatigue test specimens. The aim was to evaluate whether the combination of recycling and additive manufacturing can deliver adequate fatigue performance.

Basic fatigue tests were conducted on a limited number of samples, focusing on comparison of the two alloys and complemented by fractographic analysis and evaluation of the results with respect to available literature, which remains scarce for fatigue behavior of the investigated beta titanium alloys produced via additive manufacturing. The integration of additive processing with the aspect of recycling makes this work a unique contribution to the field.

Although the limited number of specimens did not allow the construction of a complete SN curve, the obtained results confirm the potential of recycled beta titanium alloys for applications subjected to cyclic loading and indicate directions for further optimisation of the manufacturing route.

Keywords: Ti-13Nb-13Zr, Ti-15Mo, Recycling, Additive Manufacturing, Fatigue

1. INTRODUCTION

Beta titanium alloys are attractive for biomedical applications primarily due to their low elastic modulus and high biocompatibility [1]. At the same time, owing to the combination of high strength and low density, they also appear to be promising materials for aerospace and space applications [2].

In both of these fields, the use of additive manufacturing has been increasingly expanding. This technology enables improved material utilization, the fabrication of geometrically complex or optimized lightweight structures, and, in the case of biomedicine, the production of patient-specific implants [3,4]. However, for such applications, a thorough evaluation of fatigue properties is essential. These properties have so far been described in detail mainly for additively manufactured $\alpha+\beta$ titanium alloy, Ti-6Al-4V [5], whereas data for promising β -titanium alloys, such as Ti-13Nb-13Zr and Ti-15Mo, remain scarce.

In addition to the limited availability of data on their properties in the additively manufactured state, the wider use of β -titanium alloys is constrained by their high cost, which is attributed not only to the complexity of titanium processing but also to the presence of expensive alloying elements such as Nb and Mo [6]. A possible route to cost reduction is the recycling of titanium scrap, such as support structures generated during additive

manufacturing or machining chips. These scraps are commonly processed into ferrotitanium in the steel industry, resulting in the loss of valuable raw material for direct titanium applications [7]. Therefore, their reutilization could help reduce the cost of β -titanium alloys, even at the expense of a moderate increase in the content of interstitial elements (O, N, H).

The present study provides fatigue properties of additively manufactured β -titanium alloys Ti-13Nb-13Zr and Ti-15Mo, whose feedstock powders were produced from recycled sources.

2. EXPERIMENTAL METHODS

Recycling of titanium scraps was employed as the primary route for material preparation and was carried out by Advanced Metal Powders s.r.o. Scraps, such as printing supports and offcuts, from Titanium Grade 2 were used as the feedstock and remelted using the Horizontal Plasma Arc Melting (HPAM) method into rods with irregular cross section. During remelting, alloying elements were added in the form of pure molybdenum sheets or a combination of crushed zirconium and niobium sheets to produce Ti-15Mo and Ti-13Nb-13Zr alloys, respectively. The nominal chemical compositions (wt%) complied with ASTM F2066-23 and ASTM F1713-08(2021)e1a standards and were verified using ED-XRF spectrometer (Delta). The produced rods were processed into semi-finished feedstock for atomization with a maximum diameter of 8 mm. The Ti-15Mo alloy was processed by multi-step hot working at 950 °C, following the procedure described previously [8], whereas for Ti-13Nb-13Zr hot working was replaced by water jet cutting to reduce oxygen uptake prior to atomization. Both alloys were ultrasonically atomized using an ATO LAB+ system. This resulted in highly spherical powder particles with a size distribution ranging from 10 to 120 μm and a mean particle size of approximately 60 μm . Prior to printing the final samples, Laser Powder Bed Fusion (LPBF) process parameters were optimized by printing cubic specimens with various parameter sets and evaluating porosity using light microscopy and image analysis, as described in detail in [8]. The selected printing parameters included a laser power of 180 W for both alloys, with scanning speeds of 300 mm/s for Ti-15Mo and 500 mm/s for Ti-13Nb-13Zr. The properties of these alloys in the as-built LPBF condition were thoroughly investigated in [8]. In the present study, microstructures of both alloys in the etched condition were examined using optical microscopy (Neophot 32). For the Ti-13Nb-13Zr alloy, oxygen content was measured using a combustion analyzer (G8 Galileo), and Vickers hardness was evaluated using a Struers Duramin hardness tester (10 kgf) for comparison with the data reported in [8]. Fatigue samples were LPBF-manufactured using a Trumpf TruPrint 1000 system and subsequently machined to the geometry shown in **Figure 1** without any post-processing heat treatment.

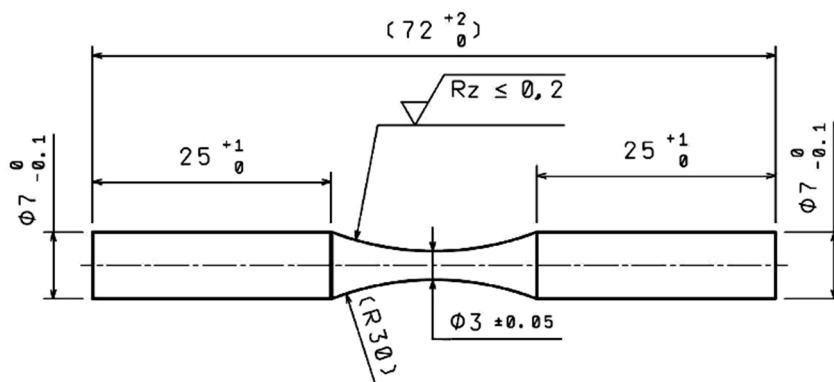


Figure 1 Additive-manufactured and machined sample for the fatigue test, dimensions in mm

Due to the limited amount of available material, only six samples were produced from each alloy. Three stress levels were selected for fatigue testing, with two specimens tested at each level. Fatigue tests were performed at room temperature using an Instron ElectroPuls E10000 system with a sinusoidal waveform at 20 Hz, a load ratio of $R = 0.1$, and a run-out limit of 2×10^5 cycles. After testing, fracture surfaces were examined by scanning electron microscopy (SEM) Tescan MIRA3. Fractographic analysis was performed on one representative sample per alloy and stress level, provided that failure occurred.

3. RESULTS

As noted above, the properties of LPBF-fabricated Ti-15Mo and Ti-13Nb-13Zr alloys produced from recycled feedstock have been described in detail in [8]. For completeness, the microstructure of the Ti-15Mo alloy in the as-built condition is shown in **Figure 2a**. In the case of Ti-13Nb-13Zr, the modified processing route applied in the present work resulted in a significant reduction of oxygen content, from an average value of 7.775 ppm to 2.118 ppm. Due to the reduced oxygen content, a lower α -phase fraction can be expected. Optical microscopy showed indications of a characteristic needle-like microstructure (**Figure 2b**), similar to that previously reported [8]. However, in the present case, the boundaries of individual melt pools are clearly visible. More pronounced differences are expected to be revealed by SEM observations. This assumption is also supported by the measured lower hardness, which decreased from the original 454 ± 15 HV10 [8] to 267 ± 7 HV10.

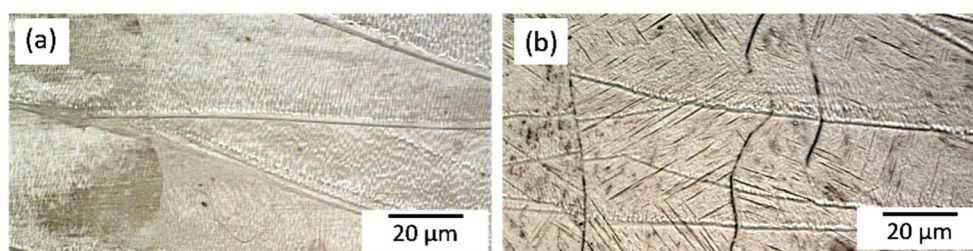


Figure 2 Microstructure of additively manufactured (a) Ti-13Nb-13Zr and (b) Ti-15Mo in the build direction

3.1 Fatigue testing

Figure 3 presents the results of fatigue testing for both investigated alloys. The obtained data indicate significant differences in fatigue behavior between Ti-13Nb-13Zr and Ti-15Mo.

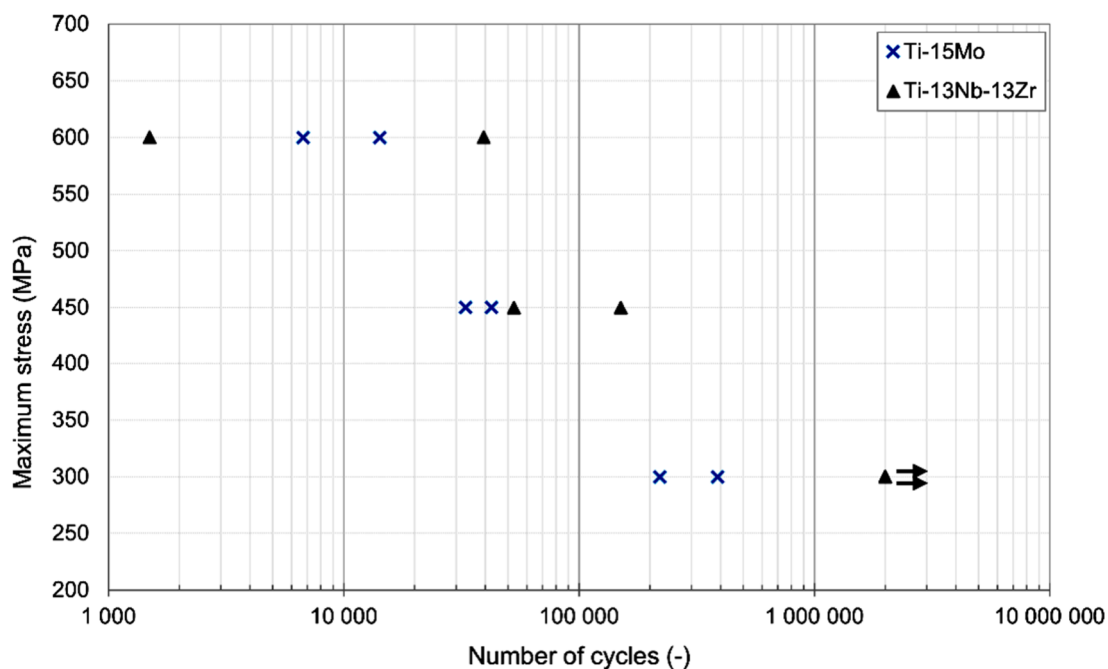


Figure 3 Fatigue results for additively manufactured Ti-13Nb-13Zr and Ti-15Mo

For Ti-13Nb-13Zr, the obtained results were compared with available literature on additively manufactured materials. Study [9] reports fatigue behavior under comparable loading conditions ($R = 0.1$, 20 Hz), although

with lower volumetric energy densities ($69.44\text{--}92.99\text{ J/mm}^3$) than the value used in this study (150 J/mm^3). Overall, a higher fatigue life was achieved in the present work; however, differences in specimen geometry (rectangular cross-section in [9] versus circular in this study) must be considered. At 450 MPa, [9] reports fatigue lives of approximately 50,000 cycles, whereas the present results range from 50,000 to 150,000 cycles. More pronounced differences were observed at lower stress levels: no failure occurred at 300 MPa up to 2×10^5 cycles in this study, while [9] reports failure even at 250 MPa within several hundred cycles. Similar trends were also reported in [10].

In contrast, Ti-15Mo exhibited significantly lower fatigue performance compared to Ti-13Nb-13Zr. A direct comparison with additively manufactured Ti-15Mo is not available in the literature. Study [11] reports fatigue behavior of conventionally produced Ti-15Mo under different microstructural conditions ($R = -1$, 30 Hz), with the lowest fatigue resistance observed for a coarse-grained β structure. Even compared to this least resistant condition, the fatigue life obtained in the present study is substantially lower, particularly in the high-cycle fatigue regime. While the results are comparable at higher stress levels (~ 600 MPa), a marked difference is observed at 450 MPa, where approximately 35,000 cycles were reached in this study, compared to several hundred thousand cycles reported in [11].

The observed discrepancy in fatigue behavior between the investigated alloys is somewhat unexpected. In the present work, the ultimate tensile strength of LPBF-fabricated Ti-15Mo was previously determined to be 1211 ± 8 MPa [8], whereas that of Ti-13Nb-13Zr could not be determined; available literature reports tensile strengths of approximately 1000 MPa for this alloy in the as-built additively manufactured condition [9,10]. Given the general correlation between fatigue and tensile strength, closer fatigue performance would be expected. The observed differences can therefore be attributed primarily to variations in microstructure and to the presence and characteristics of defects introduced during the additive manufacturing process.

3.2 Fractography analysis

Figure 4 presents selected SEM images of fracture surfaces of Ti-13Nb-13Zr specimens. In **Figures 4a** and **4b**, the crack initiation site is indicated by a white arrow, while the boundary between the fatigue fracture region and the final fracture zone is marked by a white dashed line. The individual fracture surfaces differ mainly in the size of the fatigue region, which, however, occupies the majority of the fracture surface in both cases. The fatigue region is characterized by a typical river marks. Higher magnification observations (**Figure 4c**) reveal traces of the additive manufacturing process, as well as a needle-like microstructure characteristic of this alloy in the additively manufactured condition [12].

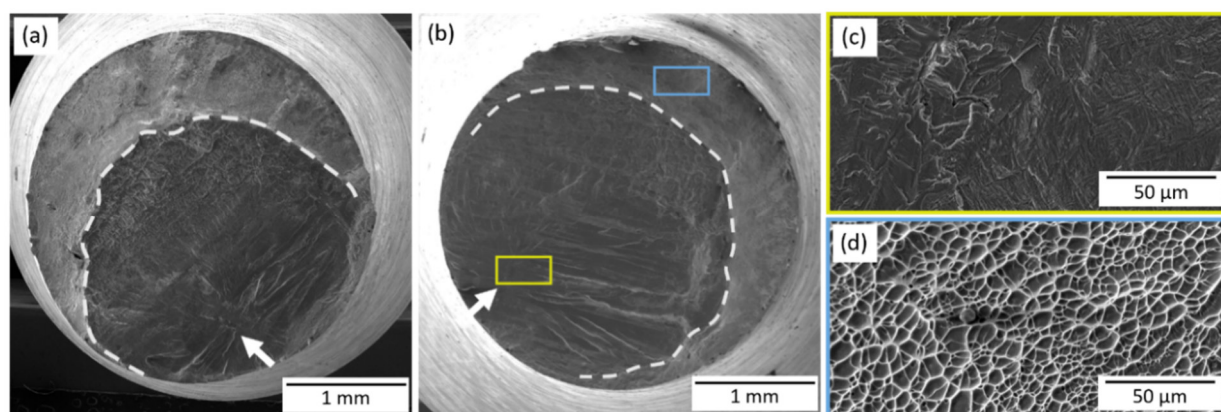


Figure 4 SEM fractography of Ti-13Nb-13Zr alloy fatigue specimens: (a) overview of the fracture surface at 600 MPa ($N_f = 39,251$ cycles); (b) overview of the fracture surface at 450 MPa ($N_f = 52,953$ cycles); (c) detail of the fatigue crack initiation and propagation region; (d) detail of the final fracture zone

In all analysed cases, fatigue crack initiation occurred at defects corresponding to lack-of-fusion (LOF) defects. However, these defects do not appear to be present in significant amounts. Nevertheless, their presence can be expected to have a detrimental effect on the overall fatigue resistance of the material. The final fracture region exhibits predominantly ductile behavior with a typical dimple morphology (**Figure 4d**).

Figures 5a-c present SEM images of fracture surfaces of Ti-15Mo specimens. Similarly to the previous case, the crack initiation site is indicated by an arrow, while the boundary between the fatigue region and the final overload region is marked by a dashed line. In contrast to Ti-13Nb-13Zr, a significantly higher amount of defects originating from the LPBF process is observed on the fracture surfaces. Despite prior optimization of the process parameters, the selected conditions proved to be unsuitable for the fabrication of larger fatigue specimens. A considerable number of LOF defects are present, in some cases accompanied by partially unmelted powder particles. The high concentration of these defects, particularly those located near the surface, led to a significant reduction in fatigue resistance and accelerated both crack initiation and propagation. An example of a pronounced LOF defect acting as a fatigue crack initiation site, together with unmelted powder particles, is shown in **Figure 5d**. The fatigue region exhibits a characteristic river marks. Higher magnification observations (**Figure 5e**) further reveal individual melt pools formed during the additive manufacturing process, with their boundaries highlighted by a white dotted line.

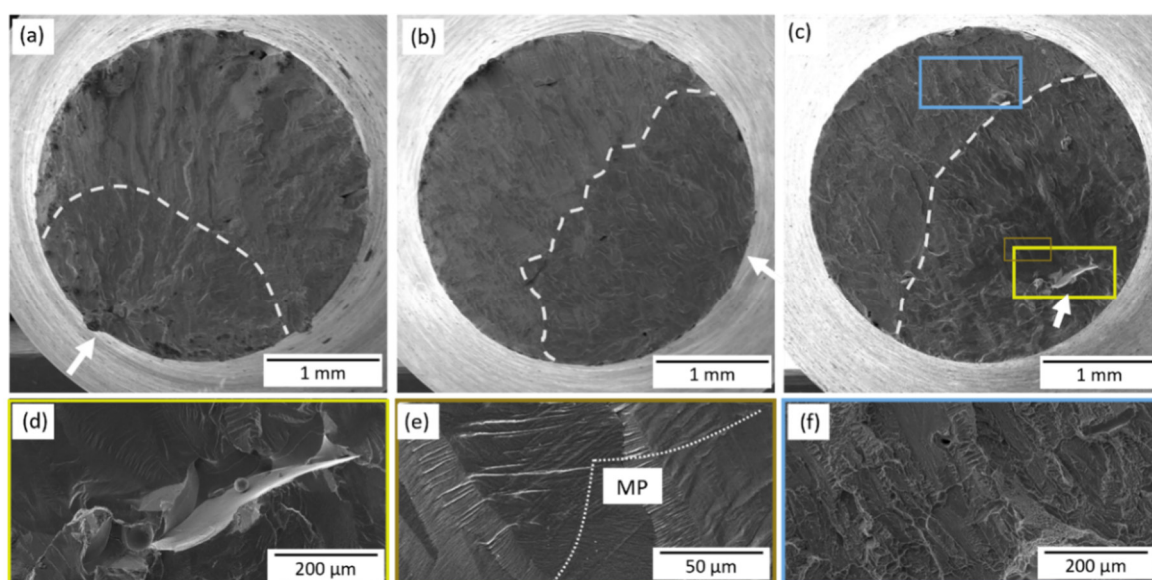


Figure 5 SEM fractography of Ti-15Mo fatigue specimens: (a) 600 MPa ($N_f = 6,733$ cycles); (b) 450 MPa ($N_f = 33,012$ cycles); (c) 300 MPa ($N_f = 388,017$ cycles); (d) fatigue crack initiation at a LOF defect with unmelted powder; (e) detail of fatigue crack propagation region; (f) detail of final fracture zone

The final fracture region (**Figure 5f**) suggests a mixed fracture mode with a tendency towards quasi-brittle behavior. The fracture surface is characterized by an irregular and relatively rough morphology, with limited evidence of typical ductile dimple formation.

4. CONCLUSION

Based on the obtained fatigue and fractographic results, the additively manufactured Ti-13Nb-13Zr alloy exhibits very good fatigue performance compared to previously reported data for additively manufactured counterparts. The results therefore highlight the significant potential of this recycled alloy as a cost-effective material with favorable mechanical properties. In contrast, the results for Ti-15Mo represent, to the best of the authors' knowledge, one of the first datasets describing fatigue behavior of this alloy in the additively manufactured state. Although the achieved fatigue performance did not meet expectations based on its high

ultimate tensile strength (1211 ± 8 MPa [8]), a pronounced influence of process-induced defects was clearly identified. It can be assumed that further optimization of processing parameters and reduction of defect population could lead to a substantial improvement in fatigue performance. In combination with the use of recycled materials, Ti-15Mo may therefore represent a promising candidate for future engineering applications.

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