

Effects of Internal Defects and Residual Stress on Fatigue Properties of a Titanium Alloy (Ti-6Al-4V) Fabricated Via Electron Beam Melting (EBM)*

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ABSTRACT

A clear understanding of the fatigue properties of Ti-6Al-4V manufactured with electron beam melting (EBM) is needed to ensure performance in critical applications in the medical device and aerospace industries. In this work, the effects of residual stress and internal defects (pores and voids) on fatigue properties of EBM Ti-6Al-4V material in as-built, stress-relieved, and hot isostatic pressed (HIPed) conditions were evaluated. Conventional techniques were used to measure the chemical composition and quantify microstructures, and neutron diffraction was utilized to measure residual stresses arising from the EBM process. Post-processing did not alter chemical composition. Compared to the as-built condition, microstructure was unchanged for stress-relieved material and coarser for HIPed material. No significant residual stresses were measured for any of the three conditions. This indicates build platform and layer preheating lead to sufficient process temperatures to achieve full stress relief *in-situ*. The fatigue strengths at 10^7 cycles measured for the as-built and stress-relieved conditions were statistically similar and were measured to be 200 MPa to 250 MPa. A significantly higher fatigue strength at 10^7 cycles of 550 MPa to 600 MPa was measured for the HIPed condition. The increase in fatigue endurance limit was attributed to a reduction in internal porosity and void content.

INTRODUCTION

Electron beam melting (EBM), an additive manufacturing (AM) process, shows great promise for making medical devices and aerospace components through excellent shape control via computer aided design input. In these industries, it is of utmost importance to characterize and fully understand all influences on the fatigue properties as components are exposed to fatigue loading, which could lead to fatigue failures [1]. In previous EBM Ti-6Al-4V work, hot isostatic pressing (HIPing) has been shown to close internal porosity and improve fatigue endurance limit by approximately 20 % [2]. It was assumed that this improvement in fatigue properties was due solely to closure of stress-concentrating internal pores, but it is possible that other microstructural changes, such as residual stress relief, occur during HIPing and contribute to the improvement in fatigue properties. Residual stresses are known to affect Ti-6Al-4V fatigue properties both beneficially (compressive residual stresses) and deleteriously (tensile residual stresses) [3]. It is likely that the rapid heating and cooling of additive manufacturing processes [4] create residual stresses in as-built material. This has been predicted by theoretical modeling of additive manufacturing processes [5, 6], as well as experimentally verified in laser-based additively manufactured material [5]. Previous experimental and numerical modeling results showed that build platform and powder layer preheating reduce residual stresses and negative effects such as part warping [5]. In this work, the relative effects of internal pore closure and residual stress relief on fatigue properties were determined by analyzing and comparing three conditions: as-built (contains residual stresses and internal pores), stress-relieved (internal pores only), and HIPed (neither residual stresses nor internal pores).

MATERIALS AND METHODS

Sixty-four cylinders (24 mm diameter x 155 mm height, oriented vertically with long axis in the build-direction, a.k.a. z -direction) were built in a single build using Arcam S12 EBM equipment¹ (software version 3.2.114.13836, accelerating voltage 60 kV, layer thickness 70 μm , speed factor 35, standard Arcam build theme for Ti-6Al-4V) and gas-atomized Ti-6Al-4V powder (average particle size 70 μm). After additive manufacturing, one third of the parts were stress-relieved (5 h at 650 °C,

air, furnace cooling), one third were HIPed (2 h at 900 °C, argon, 12 °C/min heating and cooling rates, 100 MPa), and one third were left in the as-built condition. Chemistry, microstructure, and residual stress (neutron diffraction, gage volume 4 mm x 4 mm x 4 mm) were measured for each condition in addition to fatigue testing (tensile-tensile, load ratio R=0.1). The vertical orientation (tensile/fatigue stress axis in z-direction) was chosen as it represents the worst case for fatigue strength [2].

RESULTS

No significant differences in chemistry were observed between the three conditions (Table I). This eliminates the possibility that differences in chemistry between the conditions would be the cause of differences in fatigue results.

Table I. Chemistry results (in wt%) shown for all three conditions compared to ASTM F2924. Uncertainties are given when available.

	N	C	H	Fe	O	Al	V
ASTM F2924	0.05 max	0.10 max	0.015 max	0.30 max	0.20 max	5.5-6.75	3.5-4.5
As-Built	0.054±0.008	0.015±0.0009	0.0003±0.00002	0.20	0.191±0.006	5.67	4.28
Stress Relieved	0.053±0.007	0.017±0.001	0.0005±0.0003	0.197	0.191±0.006	5.78	4.28
HIPed	0.055±0.008	0.015±0.0009	0.0008±0.00005	0.20	0.191±0.006	5.67	4.29

The microstructures of all three conditions (Figure 1) were as expected for EBM Ti-6Al-4V (equilibrium acicular or Widmanstätten). It was observed qualitatively that the stress relief heat treatment did not result in any apparent change in α lath thickness (i.e. grain size), but the HIPed microstructure did appear coarser compared to the as-built condition as expected from previous studies on EBM Ti-6Al-4V [2].

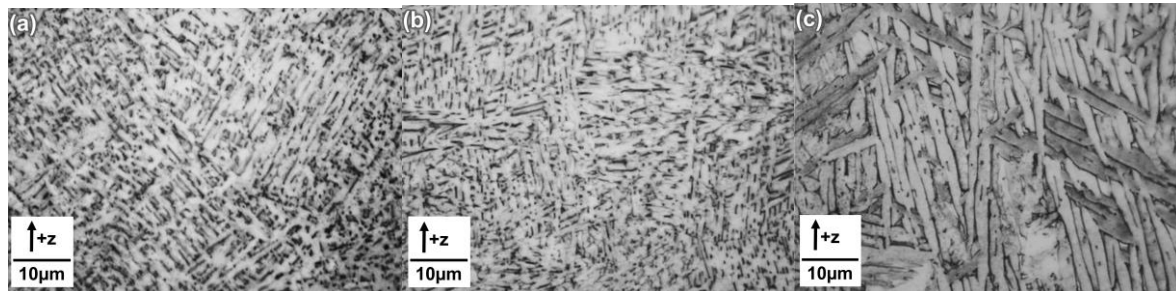


Figure 1. Optical microscope images for the (a) as-built (b) stress-relieved, and (c) HIPed conditions showing expected equilibrium acicular or Widmanstätten microstructure (α – lighter phase, β – darker phase).

We interpret the residual stress results (Table II) to indicate there are no significant residual stresses in any of the three conditions, as most stress values are within two standard errors (standard error = 30 MPa) of zero and are less than 5 % of the monotonic yield strength (approximately 870 MPa [2]). Also, there is no clear distinction in the stresses for different directions (x,y,z).

Table II. Residual stresses determined from the neutron measurements. The coordinates for each measurement are given with respect to the center of each sample. The standard error is 30 MPa.

Condition	x -coordinate (mm)	y -coordinate (mm)	Residual Stress (MPa)		
			X	Y	Z
As-Built	-2	0	17	24	17
	-2	4	-8	-17	-3
	0	4	53	78	49
	0	0	-38	-60	-55
	-2	4	-25	-45	-2
	-4	0	20	15	40
Stress Relieved	0	0	20	7	3
	3.5	0	-20	-30	-15
	0	3.5	12	2	21
HIPed	0	0	-29	-11	-16
	0	4	15	22	24

No significant differences were observed between the fatigue behavior of as-built and stress-relieved material (fatigue strength at 10^7 cycles between 200 MPa and 250 MPa), but HIPed material showed over 100 % improvement (fatigue strength at 10^7 cycles between 550 MPa and 600 MPa) compared to as-built and stress-relieved conditions (Figure 2). Voids were observed on all as-built and stress-relieved fracture surfaces, as well as river markings pointed to these voids as fatigue crack initiation sites (Figure 3). Neither voids nor internal pores were observed on the fatigue fracture surfaces of HIPed specimens (Figure 4).

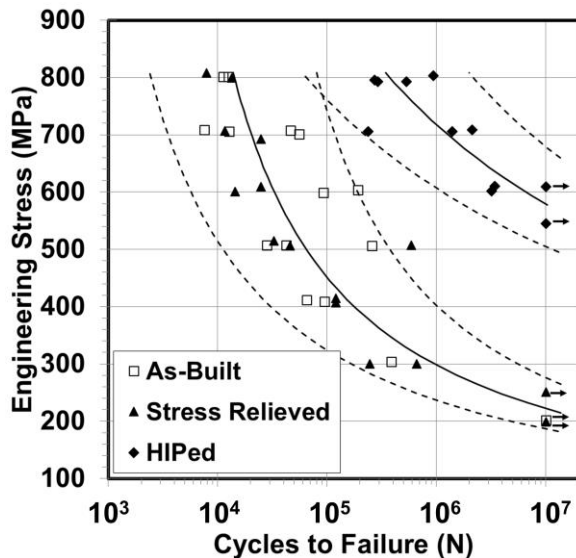


Figure 2. S-N curve fatigue results for all three conditions. Maximum tensile stress is plotted against cycles to failure. Arrows indicate sample did not fail (runout). No difference was observed between as-built and stress-relieved conditions, but HIPed material showed a significant improvement. Solid lines are from regression fit, and dotted lines are 95% confidence bounds from regression fit.

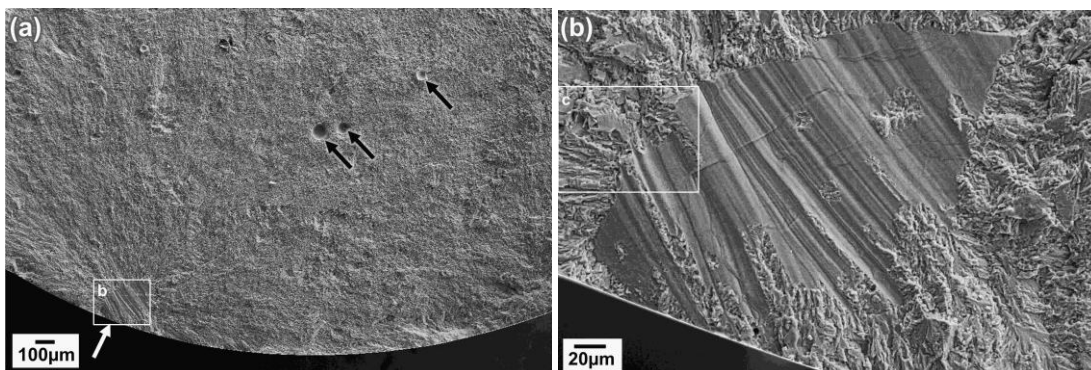


Figure 3. SEM images of representative as-built fatigue fracture surface: (a) internal pores (black arrows) and river marks point to void at fatigue crack initiation site (white arrow)

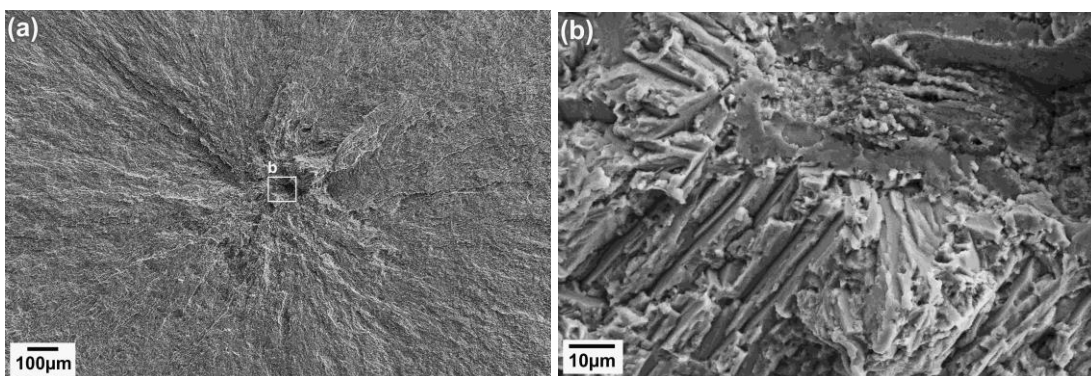


Figure 4. SEM images of representative HIPed fatigue fracture surface. (a) river marks point to initiation site at center of image, interior initiation, (b) no evidence of voids or internal pores at fatigue crack initiation.

DISCUSSION AND CONCLUSIONS

Neutron diffraction results (Table II) suggest negligible residual stresses in all of the EBM Ti-6Al-4V conditions tested (as-built, stress-relieved, and HIPed). It should be noted that the stress state is averaged over the gage volume. Such convolution prevents sharp strain features from being detected but was necessary due to incoherent scattering and strong absorption. However, fatigue results and knowledge of EBM Ti-6Al-4V thermal history do support this conclusion. Fatigue results (Figure 2) showed no difference between as-built and stress-relieved conditions, which suggests residual stresses are at least small enough to have negligible effect on fatigue properties. Also, at the high background temperature from layer preheating in EBM (above approximately 600 °C [7]), it is likely full stress relief occurs during building, considering typical EBM build times are well in excess of the literature time to full stress relief at the same temperature (5 h, 600 °C [3]). Compared to as-built and stress-relieved material, HIPed material showed a drastic improvement in fatigue strength at 10^7 cycles over as-built material (more than 100 %). Because of the residual stress and fractography results of this study, we can now more conclusively attribute this improvement to closure of internal pores and voids. Chemistry results showed no significant differences between conditions (Table I), eliminating it as a variable in this work. Coarsening during HIPing (Figure 1) most likely affected the fatigue results, but it was a deleterious effect compared to the beneficial effect of internal pore/void closure, suggesting the fatigue improvement due to internal pore/void closure is even greater than measured. The deleterious effect from coarsening is known from the results of previous work on Ti-6Al-4V, of the same microstructure as the present work, which reported an increase in fatigue endurance limit from 500 MPa to 600 MPa for a decrease in α lath thickness from 10 μm to 1 μm [8]. It was difficult to quantify the effect of coarsening in the present work, as the specific relationship between α lath thickness and fatigue properties (e.g. Hall-Petch) is unknown. However, the comparatively small coarsening observed in the present work (approximately 1.6 μm HIPed to 0.8 μm as-built) gives some indication of the expected magnitude of effect on fatigue properties.

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FOOTNOTES

¹Commercial names are identified in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by NIST, nor does it imply that they are necessarily the best available for the purpose.

REFERENCES

- [1] J.R. Davis, Handbook of Materials for Medical Devices, in, ASM International, Metals Park, OH, 2003.
- [2] U. Ackelid, M. Svensson, Additive Manufacturing of Dense Metal Parts by Electron Beam Melting, in: Materials Science and Technology Conference, MS&T Partner Societies, Pittsburgh, PA, 2009, pp. 2711-2719.
- [3] G. Welsch, R. Boyer, E.W. Collings, Materials Properties Handbook: Titanium Alloys, ASM International, Metals Park, OH, 1994.
- [4] L. Qian, J. Mei, J. Liang, X. Wu, Influence of Position and Laser Power on Thermal History and Microstructure of Direct Laser Fabricated Ti-6Al-4V, Materials Science and Technology, 21 (2005) 597-605.
- [5] N.W. Klingbeil, J.L. Beuth, R.K. Chin, C.H. Amon, Residual stress-induced warping in direct metal solid freeform fabrication, Int. J. Mech. Sci., 44 (2002) 57-77.
- [6] A. Vasinonta, J.L. Beuth, M. Griffith, Process maps for predicting residual stress and melt pool size in the laser-based fabrication of thin-walled structures, J. Manuf. Sci. Eng.-Trans. ASME, 129 (2007) 101-109.
- [7] Arcam S12 User Guide, in, Arcam AB, Molndal, Sweden.
- [8] G. Lutjering, A. Gysler, Fatigue: A Critical Review, in: G. Lutjering, U. Zwicker, W. Bunk (Eds.) Titanium '84 - Science and Technology, Deutsche Gesellschaft fur Metallkunde EV, Munich, 1985, pp. 2065-2083.

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