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Procedia Structural Integrity 28 (2020) 790–795

Structural Integrity Procedia

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1st Virtual European Conference on Fracture

Effect of artificial saliva on the fatigue and wear response of TiAl6V4 specimens produced by SLM

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Abstract

Additive manufactured (AM) parts made in TiAl6V4 alloy are increasingly used in medical prostheses and dental implants, because of its high strength, low weight and excellent biocompatibility. These components work under environmentally assisted cyclic loading, i.e. under corrosion-fatigue, and/or subject to wear conditions. Fatigue performance of additive manufactured alloys is significantly influenced by the porosities, residual stresses, which can reduce its strength when compared with traditional materials. This paper presents the results of a fatigue crack propagation study in titanium TiAl6V4 specimens produced by selective laser melting (SLM) under artificial saliva ambient. Tests were performed using standard 6 mm thick compact specimens (CT) tested at R=0.05 and with frequencies of 1 and 10 Hz. The main objective of the current research work was studying the corrosion effect on the fatigue crack propagation of Selective Laser Melting (SLM) specimens, and to compare the tribocorrosion behaviour of two different specimens: one produced by SLM and the other by the conventional/traditional method. The study concluded that: AM Titanium Ti6Al4V alloy exhibits a moderate effect of saliva ambient on fatigue crack nucleation and on fatigue crack propagation, the wear rate coefficients for SLM and conventional manufactured specimens is of the same order, and the mechanism of abrasive wear is mainly with grooves aligned with the direction of sliding.

Keywords: Additive manufacturing, Corrosion-fatigue, Abrasive wear, Titanium TiAl6V4 alloy.

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2452-3216
10.1016/j.prostr.2020.10.092

1. Introduction

The Ti6Al4V alloy with (α+β) structure was the first titanium alloy registered as an implant material in the ASTM standards (F-136-84), T. Akahori and M. Niinomi (1998). The titanium Ti6Al4V alloy is a light alloy commonly used in aerospace components, Guo and Leu (2013) and currently dominates the market of dental implant applications since it has a comparatively low Young's modulus (close to that of cortical bone), presents excellent biocompatibility and high cyclic fatigue resistance. Besides that, Ti6Al4V demonstrates a high resistance to corrosion and corrosion-fatigue, Zavanelli et al. (2000), since it develops a passivating oxide layer with a thickness of approximately 3 to 10 nm in the presence of oxygen, preventing a reaction between the bulk material and the environment (e.g. body fluids). However, under in vivo conditions in contact with body tissues and fluids for long periods of time and exposed to cyclic loads, the implants are subjected to the tribocorrosion phenomenon, which consists in the degradation of mechanisms due to the combined effect of wear and corrosion, Licausi (2013).

Unique and specific to the patient, it is possible to produce biomedical implants that comply with explicit surgical geometries by impersonating the mechanical properties of common bone, and assist in better cell and tissue integration. Selective Laser Melting (SLM) is an AM technology that uses a high-powered ytterbium fiber laser to fuse metallic powders together to form functional 3D parts, Razvan Udroiu (2012).

Conventional casting Ti-6Al-4V alloy typically exhibits good corrosion and corrosion-fatigue resistance in different environments when compared with steel and other metallic materials, Dimah et al. (2012). Dawson and Pelloux (1974) studied the crack propagation in the Ti-6Al-4V alloy for several environments, concluding on the frequency independent behaviour, which is typical of the alloy in vacuum, air and solutions with corrosion inhibitors. Baragetti and Arcieri (2018) obtained a reduction of about 20% in the fatigue life of Ti6Al-4V in notched specimens tested in a 3.5%wt NaCl solution, at frequency of 10Hz, in comparison with inert ambient tests. In spite of the good corrosion resistance of casting Ti-6Al-4V alloy, AM materials have some particularities, namely, anisotropy, the presence of crack defects, stress concentrations and residual stresses, which can change corrosion-fatigue behaviour. The aim of the present research work is studying the corrosion effect on the fatigue crack propagation of Selective Laser Melting (SLM) specimens and to compare the tribocorrosion behaviour of two different specimens, produced by SLM and by the conventional/traditional method.

2. Materials and testing

The material used in the current work was metal powder Titanium Ti6Al4V Grade 23 alloy, with a chemical composition indicated in Table 1, according with the manufacturer.

Table 1. Chemical composition of the Titanium Ti6Al4V alloy [wt.%].

Al	V	O	N	C
5.50 - 6.50	3.50 - 4.50	< 0.15	< 0.04	< 0.08
H	Fe	Y	Ti	
< 0.012	< 0.25	< 0.005	Bal.	

Experimental fatigue tests were performed using 6 mm thickness compact tension (CT) specimens, with the final geometry and dimensions shown in Fig. 1a), manufactured by Lasercusing®, with layers growing towards the direction of loading application. The samples were processed using a ProX DMP 320 high-performance metal additive manufacturing system, incorporating a 500w fibber laser.

After manufacturing by SLM, the specimens were subjected to a stress relieve heat treatment that consisted of a slow and controlled heating up to 670 °C, followed by maintenance at 670 °C±15 ºC for 5 hours in argon medium at atmosphere pressure and finally by cooling to room temperature in air.

Friction tests use one specimen produced by SLM process, as shown in Fig. 1b), which presents also the building direction and the surface submitted to tribocorrosion evaluation (external surface of XY-plane), Nianwei et al. (2016), and the other specimen produced by a traditional/conventional manufacturing process, both classified as Ti6Al4V (ASTM B348, Titanium alloy Grade V). Both specimens were polished using SiC abrasive grinding papers to an average surface roughness of 0.1 µm. The measurements of the roughness parameters were performed using a Mitutoyo Surfest – 500 profilometer in accordance with ISO standard 4287. A Rodenstock RM 600 laser profilometer was used in order to calculate the Ti6Al4V wear volume. Electrochemical experiments - potentiodynamic polarization curve was performed for both specimens. Further details are described in Vilhena et al. (2019).

Fatigue tests were carried out in agreement with the ASTM E647 standard, at room temperature using a 10 kN capacity Instron EletroPuls E10000 machine, under loading control and stress ratio of R = 0.05, with the frequencies of 1 and 10 Hz. For comparison purposes, a second batch of tests were conducted under corrosion-fatigue into artificial saliva solution, with the chemical composition indicated in Table 2. During these tests, the crack length was measured using a travelling microscope (45x) with an accuracy of 10 µm. Fig. 2a) shows the experimental apparatus, detailing the specimen, the box with corrosive fluid and the systems for the corrosion potential and crack length measurement.

Tribological test use reciprocating sliding with a Zirconia ball sliding against a Ti6Al4V specimen under different applied loads, lubricated with artificial saliva. A frequency of 3 Hz was used during 15 m sliding distance (stroke = 2 mm). Fig. 2b) presents the tribocorrosion cell showing the two electrodes assembly, Saturated Calomel Electrode (SCE) working as Reference Electrode (RE) and platinum wire working as a Counter Electrode (CE).

Table 2. Chemical composition of the corrosive solutions [g/l].

Corrosive solution	NaCl	KCl	$CaCl_2.2H_2O$	KH_2PO_4	$Na_2HPO_4.12H_2O$	KSCN	$NaHCO_3$	Citric Acid
Artificial saliva (pH=6.5)	0.6	0.72	0.22	0.68	0.856	0.06	1.50	0.03

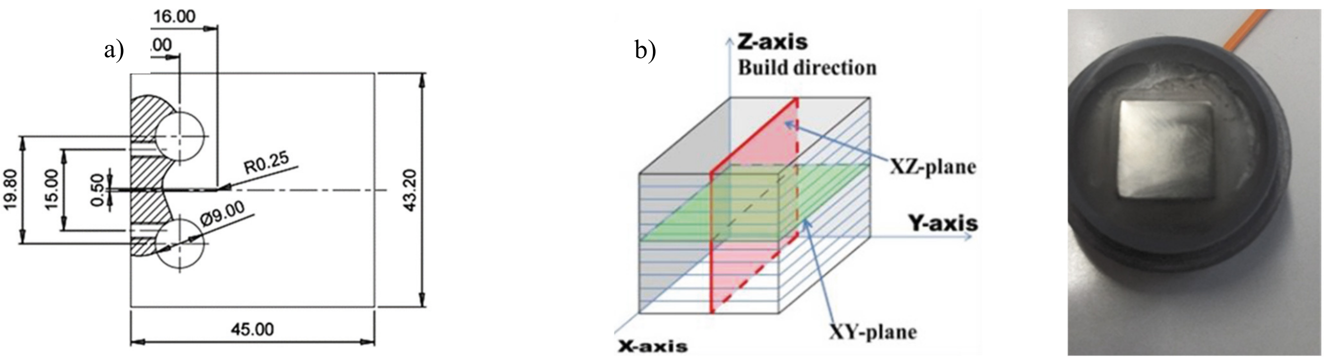

Fig. 1. (a) Specimen geometry (dimensions in mm) for fatigue tests; (b) Tribocorrosion specimen and growth direction.

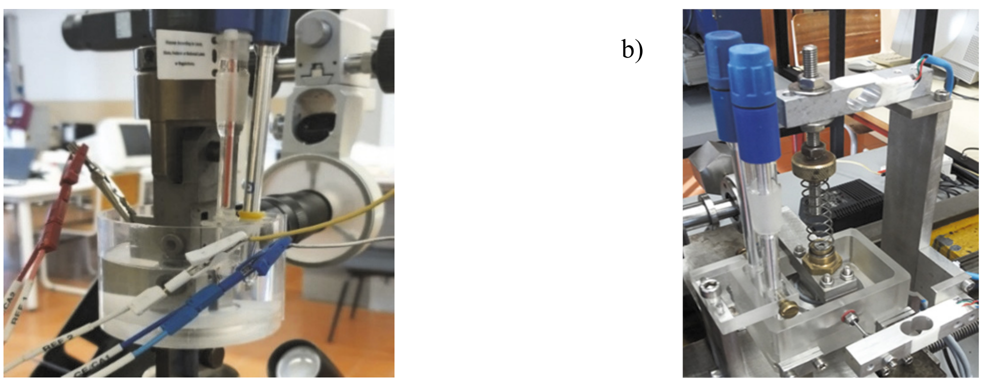

Fig. 2. Testing apparatus (a) Fatigue test and box with corrosive solution and system for corrosion potential measurement; (b) Tribocorrosion cell.

3. Results and discussion

Figs. 3 summarizes the results obtained for the fatigue crack growth in terms of the da/dN-ΔK curves, for tests carried out in air at the frequency of 10 Hz and the environmental tests in artificial saliva, at 1 and 10 Hz. The results obtained do not confirm previous studies by Dawson and Pelloux (1974) and Baragetti and Arcieri (2018) on traditional manufactured TiAl6V4 alloys; the current study shows a significant corrosive damage expressed by an acceleration effect on the fatigue crack propagation for the two frequencies. It was observed an important effect of the corrosion ambient on the fatigue crack growth rate, which increases with decreasing ΔK. The increasing effect is of more than 20% in crack propagation rate under artificial saliva. The effect of the frequency is quite reduced in regime II of Paris' law, having only a moderate accelerating damage for low ΔK values.

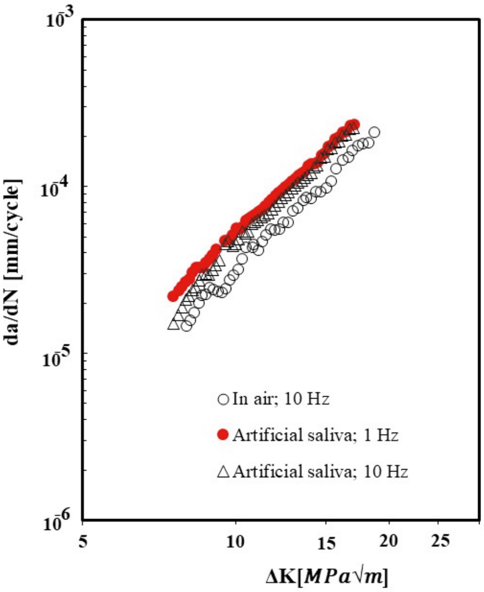

Fig. 3. Effect of corrosive solution on the da/dN-ΔK curves for 1 and 10 Hz tests.

Figs. 4a) and b) present SEM observations of the failure surfaces of specimens tested in air and in artificial saliva, respectively. Both cases reveal irregular surfaces with small plastic deformation. However, specimens tested in saliva show a higher tendency to failure inter deposed layers and forming steps between the layers.

Fig. 5 shows the steady state coefficient of friction for Ti6Al4V produced by SLM and by a conventional method, sliding against a ZrO2 ball and lubricated with artificial saliva under different applied loads (3, 5 and 7 N). Regarding the friction behaviour, the Ti6Al4V obtained by SLM and by the conventional method exhibited similar COF in the order of 0.41- 0.51. The wear rate coefficients (k) for Ti6Al4V (SLM vs. conventional) is in the same order with k (Conventional) slightly larger than K (SLM), as follows: k (SLM) = 6.2 x 10-4 mm3/N.m and k (Conventional) = 6.7 x 10-4 mm3/N.m. The corrosion resistance of Ti6Al4V obtained by SLM is higher than the Ti6Al4V produced by a conventional method, as shown in Table 3: higher potential range of passive film, ΔE, lower passive current density, ipass, and higher corrosion potential, Ecorr.

Table 3. Electrochemical properties.

Specimen	Ecorr (V vs SCE)	ipass (μA/cm2)	ΔE (V vs SCE)	Corrosion rate (mm/year)
SLM	-0.37	0.46	-0.25 to +0.72	1.86×10^{-03}
Conventional	-0.40	0.74	-0.29 to + 0.38	3.37×10^{-03}

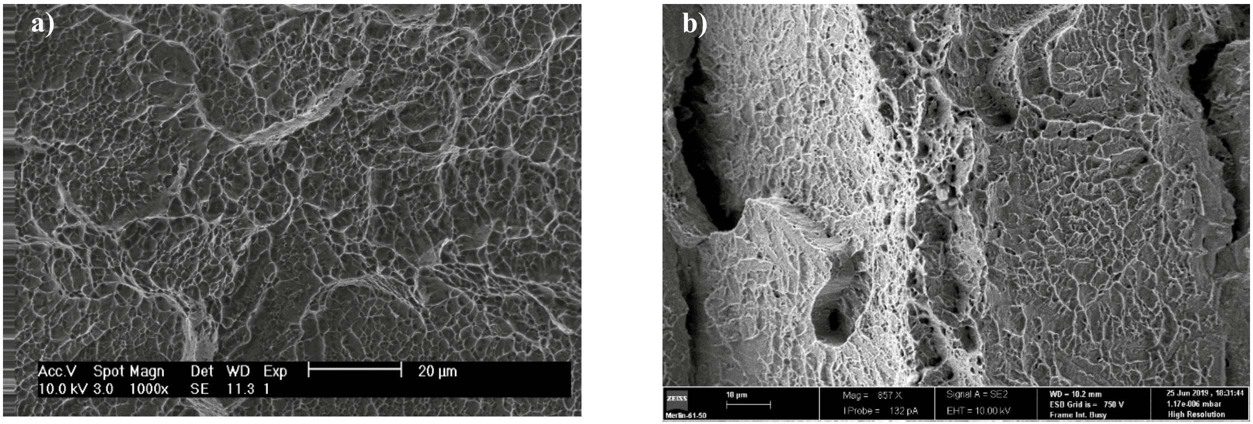

Fig. 4. Effect of corrosive solution on the fatigue failure surfaces (a) In air; (b) In artificial saliva.

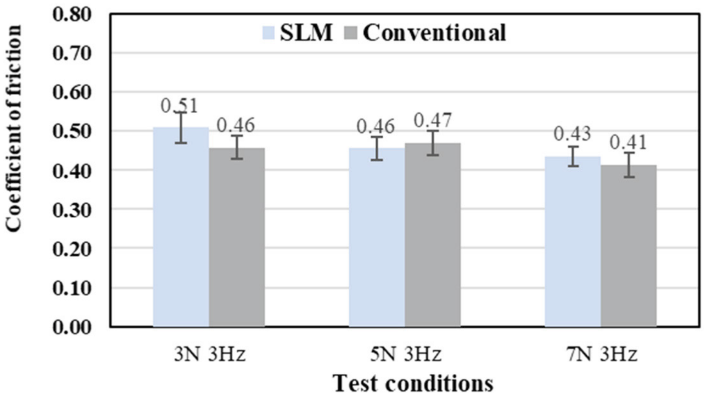

Fig. 5. Coefficient of friction for Ti6Al4V sliding against a ZrO2 ball and lubricated with artificial saliva.

Fig. 6 shows the wear track of the Ti6Al4V specimen produced by SLM. Fig. 6a) presents a typical 3D profile obtained by laser stylus profilometer, while Fig 6b) shows a SEM micrograph. It was observed that the wear mechanism for both specimens is mainly abrasive wear with grooves aligned with the direction of sliding.

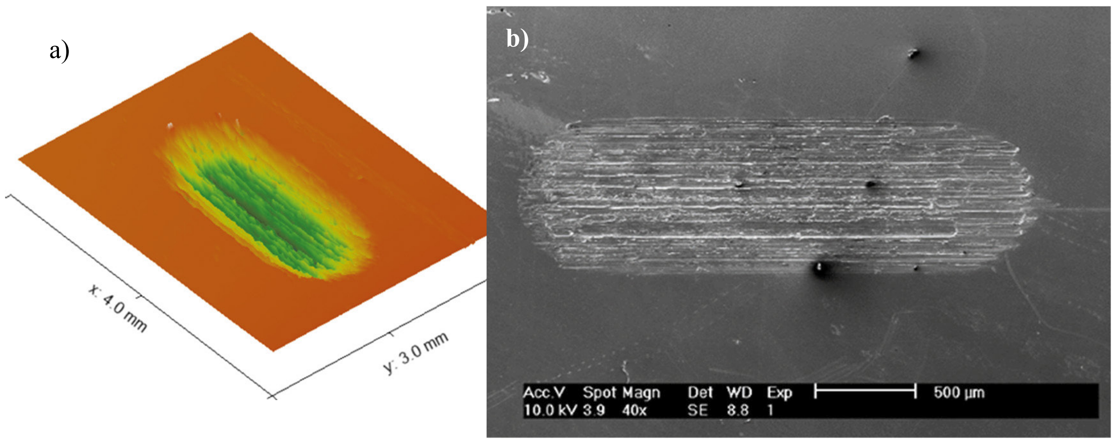

Fig. 6. Wear track of Ti6Al4V specimen produced by SLM (a) 3D profile obtained by laser stylus profilometer; (b) SEM micrograph.

4. Conclusions

This study analyses the effect of artificial saliva on the corrosion-fatigue and wear behaviour of TiAl6V4 specimens produced by SLM. The following conclusions can be drawn:

- AM Titanium Ti6Al4V alloy exhibits a moderate effect of saliva ambient on fatigue crack nucleation and on fatigue crack propagation, which increases with decreasing ΔK;
- The frequency effect on da/dN curves is negligible in the Paris' law regime and only moderate for lower ΔK;
- The wear rate coefficients (k) for SLM and conventional manufactured specimens is of the same order, in spite of the higher corrosion resistance of Ti6Al4V obtained by SLM;
- SLM specimens mainly exhibit an abrasive wear with grooves aligned with the direction of sliding.

Acknowledgements

The authors would like to acknowledge the sponsoring under the project no. 028789, financed by the European Regional Development Fund (FEDER), through the Portugal-2020 program (PT2020), under the Regional Operational Program of the Center (CENTRO-01-0145-FEDER-028789), and the project POCI-01-0247-FEDER-042536, financed by European Funds, through program COMPETE2020, under the Eureka smart label S0129-AddDies. Finally, this research is also sponsored by FEDER funds through the program COMPETE – Programa Operacional Factores de Competitividade – and by national funds through FCT – Fundação para a Ciência e a Tecnologia –, under the project UIDB/00285/2020.

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