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Influence of oxygen on the fatigue behaviour of Ti-6Al-7Nb alloy

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Abstract. One of the challenges in PM Ti alloys is to control the impurities level. Oxygen affects the microstructure and the mechanical properties of titanium alloys. Ti-6Al-7Nb is a promising alloy to use in PM due to its outstanding biocompatibility and mechanical properties required for load bearing medical implants. In this work, the influence of the impurities content on the ductility, fatigue resistance and microstructure of Ti-6Al-7Nb alloy processed by metal injection moulding was examined. Tensile and fatigue specimens were manufactured using Ti-6Al-7Nb gas atomized powder. Depending on the thermal treatment time, various oxygen contents were introduced into the specimens. The resulting oxygen content was determined by melt extraction technique. Tensile tests and high cycle four-point bending fatigue tests at room temperature were performed. First studies about the effect of oxygen content on crack initiation and propagation were done by the observation of microstructures and fractured surfaces using light and electron microscopy (SEM).

Introduction

The control of the oxygen level plays a key role to successful powder metallurgy (PM) of titanium alloys [1,2]. The presence of oxygen causes high costs in the PM industry, affecting the mechanical behaviour of titanium alloys as it can significantly decrease its ductility [3,4]. In metal injection moulding (MIM), the impurities level comes from the powder production, handling and sintering of powders. It is necessary to understand how the oxygen affects titanium alloys to get an idea which levels of oxygen are tolerable in titanium alloys and which efforts to reduce and control oxygen levels are reasonable with respect to cost efficiency. Investigations [3,4] on MIM Ti-6Al-4V revealed the existence of a critical oxygen level at ~0.33wt.% as illustrated in Fig. 1 where the tensile elongation drops rapidly from 15% to 3%.

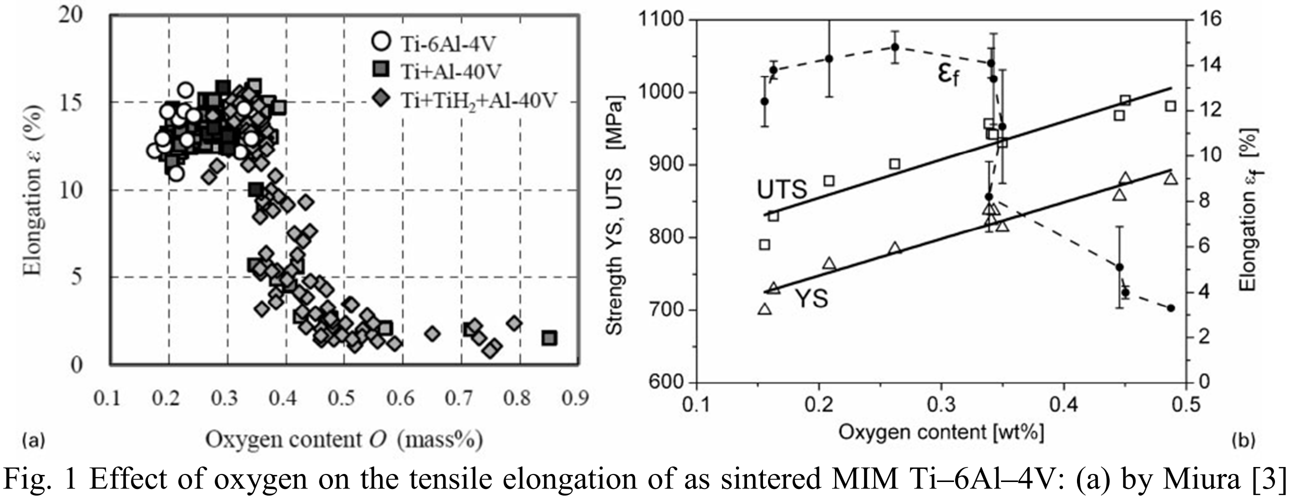


Fig. 1 Effect of oxygen on the tensile elongation of as sintered MIM Ti–6Al–4V: (a) by Miura [3] and (b) by Ebel et al. [4].

The alloy Ti-6Al-7Nb is an interesting material to be used in load bearing applications due to its favourable mechanical properties (UTS= 995-1010 MPa, elongation=10-15%, endurance limit at

10^7 cycles= 559-564 MPa) and its corrosion resistance [5]. Several Ti-6Al-7Nb components with properties similar to those of the wrought materials have been consolidated by PM [6]. However, fatigue properties of MIM components remain unclear. Ferri et al. [7] studied the fatigue behaviour of MIM Ti-6Al-4V alloy. It was found that working with oxygen levels below the tensile critical value, parameters such as surface quality and microstructure are more decisive than the interstitial content. Nevertheless, in a study of Lütjering [8] it was affirmed, that high cycle fatigue strength depends primarily on the resistance to dislocation motion, and therefore in most cases on the yield strength. It has been demonstrated [4] that the presence of oxygen increases the yield strength of MIM Ti-6Al-4V but no investigation about the dependence of the fatigue behaviour on oxygen has been done.

In this paper, the manufacture of Ti-6Al-7Nb components by metal injection moulding with different oxygen contents is performed. Finally, the influence of oxygen on the tensile properties and first conclusions about the effect on fatigue behaviour were drawn.

Experimental

Ti-6Al-7Nb powder. Gas-atomized powder from a commercial rolled rod material seen in Fig. 2 was produced by the EIGA technique (Electrode Induction Melting Gas Atomization) for these experiments. The used powder fraction had a particle size below 45 μm.

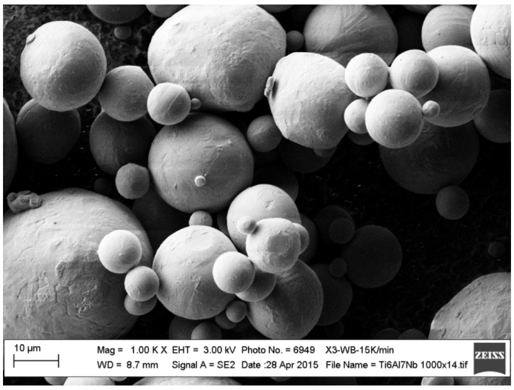


Fig. 2 Scanning electron micrograph of Ti-6Al-7Nb powder.

Metal injection moulding. Feedstock was prepared in a Z-blade mixer at 120°C during 4h. Ti-6Al-7Nb alloy powder was mixed with 35.5 vol% of a binder consisting of ethylene vinyl acetate (EVA), paraffin wax and stearic acid. Extrusion was used for feedstock homogenization.

Injection moulding was performed on an Arburg 320S injection moulding machine. Two different MIM samples were produced. Tensile test specimens with dimensions according to ISO 2740, shown in Fig. 3 a, were manufactured. For fatigue tests, four-point bending fatigue samples with dimensions shown in Fig. 3 b were produced.

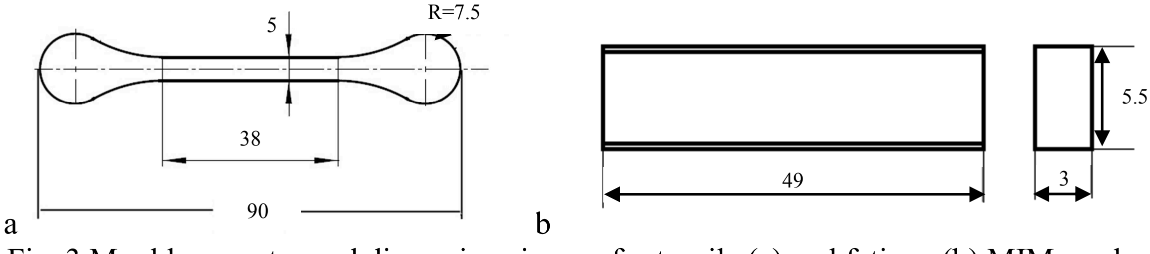


Fig. 3 Mould geometry and dimensions in mm for tensile (a) and fatigue (b) MIM production.

Debinding was performed in two steps. The first step was a chemical debinding of the green part in a hexane bath at 40°C during 15 hours. The second step a thermal debinding process at a temperature level between 450°C and 600°C under argon atmosphere.

To introduce different quantities of oxygen into the samples, the specimens were firstly pre-sintered, then thermally treated under oxygen-argon atmosphere and finally sintered. The pre-sintering step (Fig. 4 a) was performed in a cold-wall furnace with Mo-shielding and tungsten heater, at a temperature of 800°C under high vacuum for 1h using a molybdenum support with yttria coating. During the thermal treatment, specimens were exposed to oxygen (20%)-argon atmosphere of 1bar with a gas flow of 35 l/h for different times (see Table 1) at a temperature of 400°C (Fig. 4 b) inside a hot-wall furnace.

Table 1 Thermal treatment times at 400ºC.

Time [min]	3	10	30	60	900

Finally, the specimens were sintered (Fig. 4 c) in a cold-wall furnace at a temperature of 1350°C under high vacuum during 4h using again a molybdenum support with yttria coating. As a reference, some specimens were only sintered.

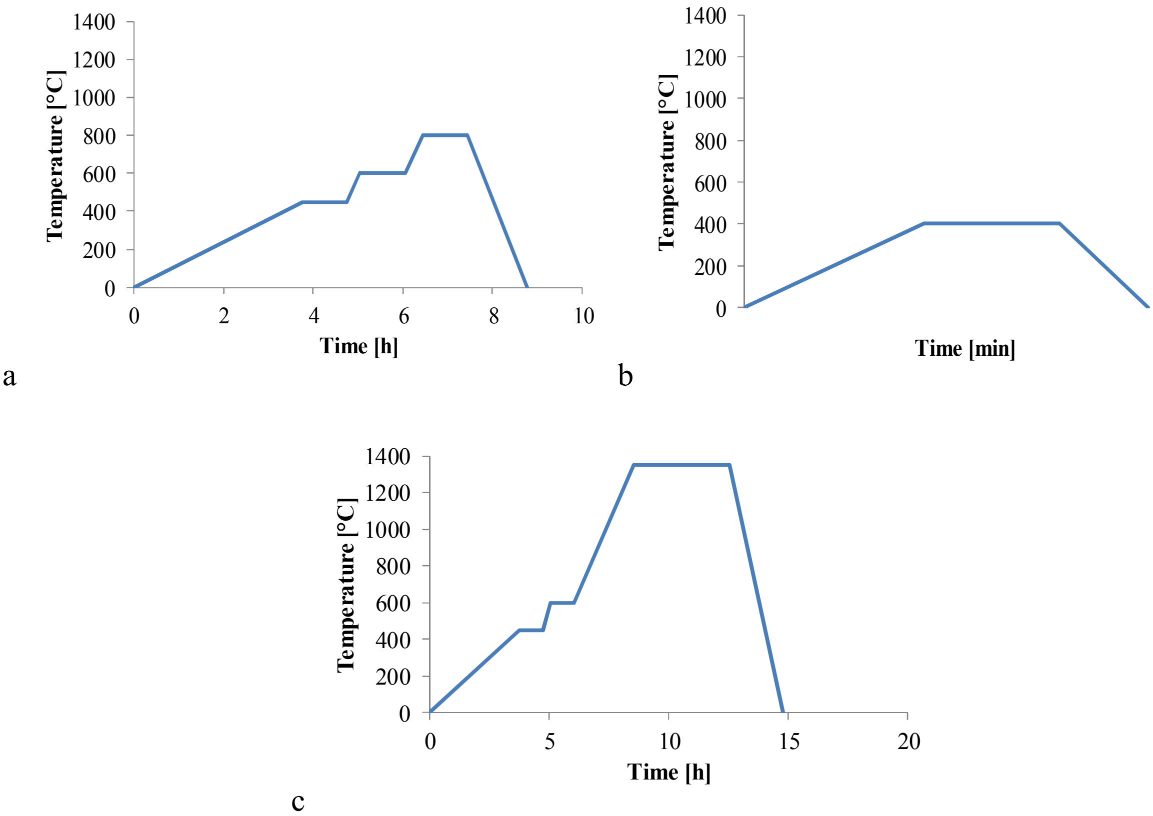


Fig. 4 Oxidation procedure: pre-sintering (a), thermal treatment (b) and sintering (c).

Characterisation. The density of sintered parts was measured by the Archimedes method and reported as percentage of the theoretical density of titanium according to MPIF Standard 42, except that ethanol was used instead of water. The level of impurities (O, N, C) was determined by LECO melt extraction system using a TCH 436 apparatus for oxygen and nitrogen measurement and a CS 444 apparatus for carbon measurement.

Tensile tests were performed on a servohydraulic structural test machine equipped with a 100kN load cell at a strain rate of 1.2 x 10^{-5} s^{-1} at room temperature in air. Fatigue samples were subjected to shot peening using zirconia particles with a diameter of 200 µm to improve the surface quality of specimens providing appropriate conditions for fatigue tests [7]. High cycle fatigue tests were performed in a four-point bending configuration using a resonance machine manufactured by RUMUL. The experiments were carried out in air at room temperature under load control with a cyclic frequency of ~95 Hz (sine wave) at a load ratio $R=\sigma_{min}/\sigma_{max}$ of 0.2. The fatigue endurance limit was defined as 10^7 cycles.

The microstructure was examined by image analysis using a Zeiss DSM962 scanning electron microscope. Grain size was determined according to ASTM E112-96 (linear intercept technique). Fractured surfaces were observed with a LEICA stereoscope and analysed by SEM.

Results and discussion

MIM Ti-6Al-7Nb sintered specimens have acquired more than 97% of theoretical density. Tensile and fatigue sintered specimens in Fig. 5 show shrinkage of 12% compared to green specimens.

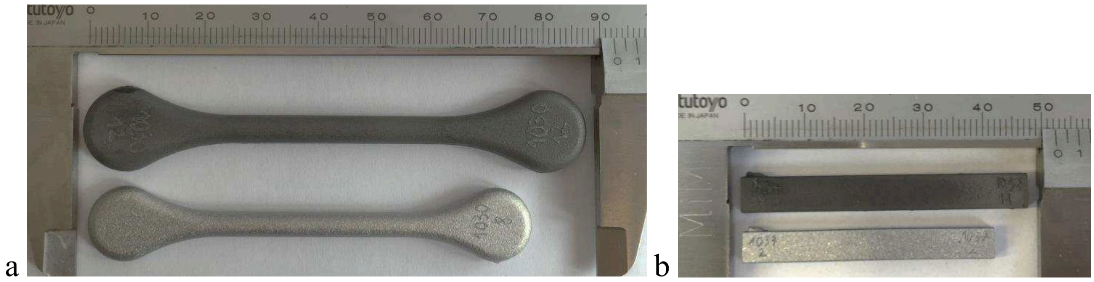


Fig. 5 Green and sintered tensile (a) and fatigue (b) specimens.

Sintered samples present a fully coarse lamellar microstructure, as illustrated in Fig. 6. The grain size was 66±11 µm in as-sintered samples and 85±6 µm when samples were thermally treated. The pre-sintering thermal cycle at 800°C causes the grain coarsening. The residual porosity has near-circle shape with maximum pore size of approximately 40 µm.

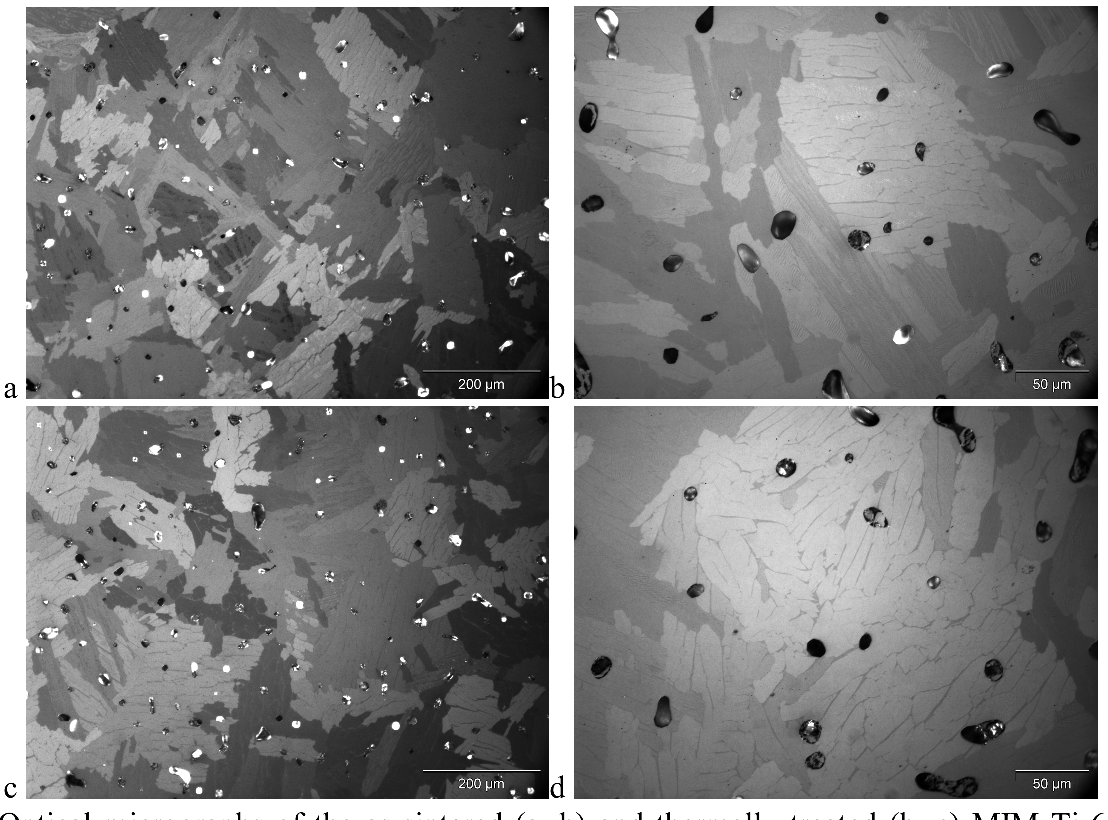


Fig. 6 Optical micrographs of the as-sintered (a, b) and thermally treated (b, c) MIM Ti-6Al7Nb tensile samples.

Different contents of impurities were introduced in samples depending on the exposition time during the thermal treatment step (see Table 2). Higher oxygen content is acquired when the specimens were exposed longer under the oxygen-argon atmosphere. The small variation of nitrogen or carbon content confirms the success in the exclusive introduction of oxygen impurities. Therefore, variations of carbon and nitrogen will not be considered in the investigation.

Table 2 Impurities content of samples subjected to different treatment times at 400ºC.

Treatment time [min]	O-content [wt.%]	N-content [wt.%]	C-content [wt.%]
As-sintered	0.22	0.01	0.03
3	0.38	0.03	0.04
10	0.41	0.02	0.04
30	0.43	0.02	0.04
60	0.44	0.02	0.04
900	0.60	0.03	0.04

Tensile test. The influence of the impurities content on the mechanical behaviour of MIM Ti-6Al-7Nb is proved by the tensile test results in Table 3 and Fig. 7. The increase of oxygen content leads to an increase of the tensile and yield strength. Both parameters have a linear dependence on the oxygen content with a high coefficient of determination (R^2).

Table 3 Average tensile parameters (n=5) of samples with different oxygen content.

O-content [wt.%]	Tensile strength [MPa]	Yield strength [MPa]	Plastic Elongation [%]	Young modulus [GPa]
0.22	795	692	16	100
0.38	876	759	16	92
0.41	886	775	12	95
0.43	893	783	11	101
0.44	898	788	10	99
0.60	925	823	3	100

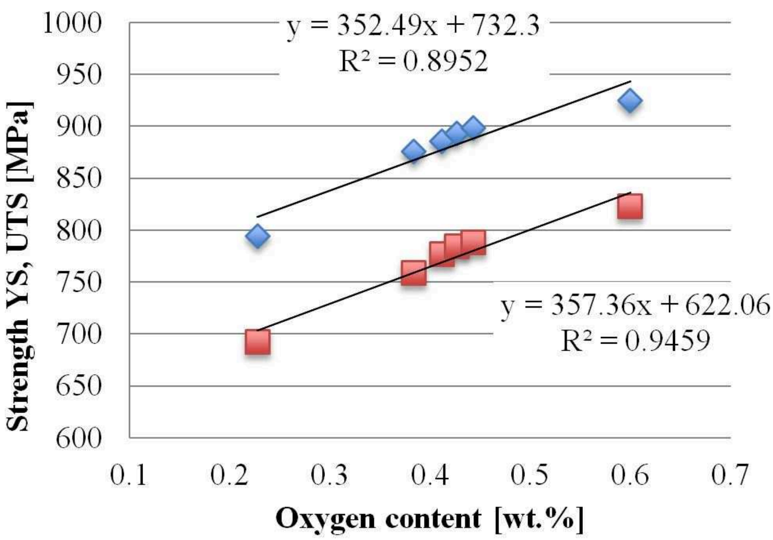


Fig. 7 Influence of oxygen on the tensile strength and yield strength of MIM Ti-6Al-7Nb.

The strengthening behaviour in MIM Ti-6Al-7Nb by oxygen presence is mainly due to the oxygen solid solution into α phase. No strengthening mechanism by grain size reduction is detected during microstructure evaluation.

Good ductility is shown by the as-sintered samples as a result of the lowest impurities content. Its fracture surface shows mostly the dimple rupture characteristic of MIM ductile materials (see Fig. 8).

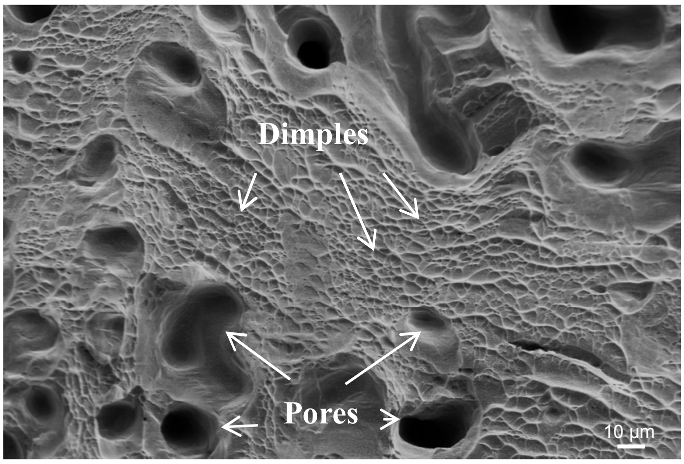


Fig. 8 Typical ductile fracture with equiaxed dimples in case of an as-sintered sample.

The ductility drops significantly when the content of oxygen reaches 0.38 wt.% (see Fig. 9). Above this value, areas with transgranular brittle fracture characterized by facets on the fracture surface are found as it is illustrated in Fig. 10.

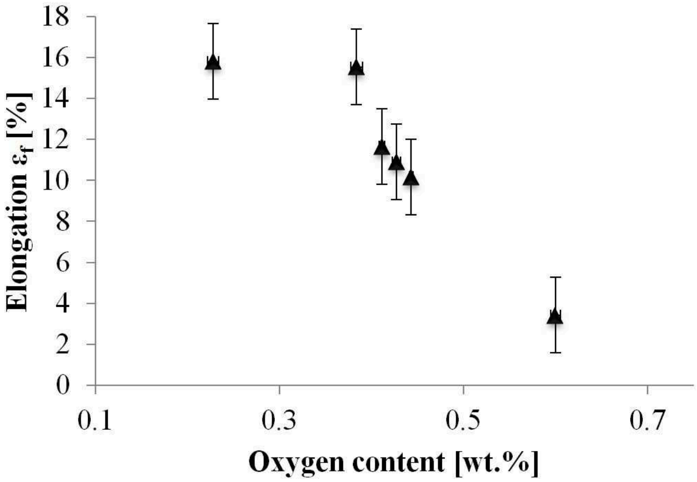


Fig. 9 Dependence of elongation on the oxygen content of MIM Ti-6Al-7Nb samples.

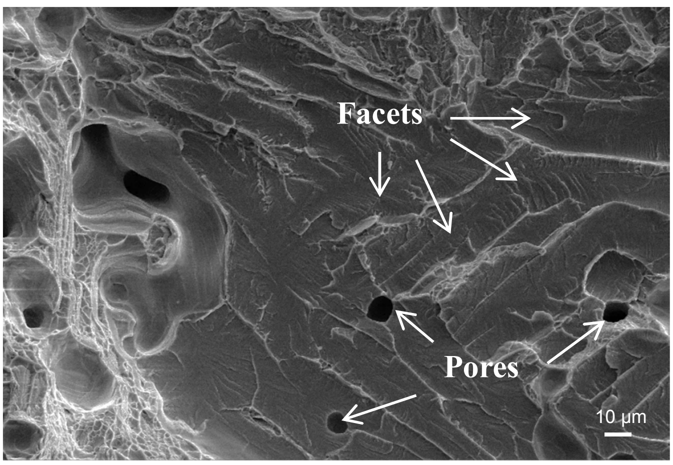


Fig. 10 Transgranular brittle fracture of thermally treated samples.

Ebel et al. [4] investigated in MIM Ti-6Al-4V the dependence of the yield strength on the oxygen content (Fig. 1). For the same increase in oxygen, the growth of tensile and yield strength in Ti-6Al-4V is bigger than in Ti-6Al-7Nb. The dependence of the mechanical behaviour on oxygen content of both materials is qualitatively similar but, in case of Ti-6Al-7Nb, the reduction of the ductile elongation is not as abrupt. Moreover, a higher critical oxygen content is found (0.38 wt.%).

Fatigue test. Four-point bending fatigue test were performed to find the endurance limit of MIM Ti-6Al-7Nb with different oxygen contents. The results represented in Fig.11 show that the oxygen increase causes a reduction of the fatigue resistance.

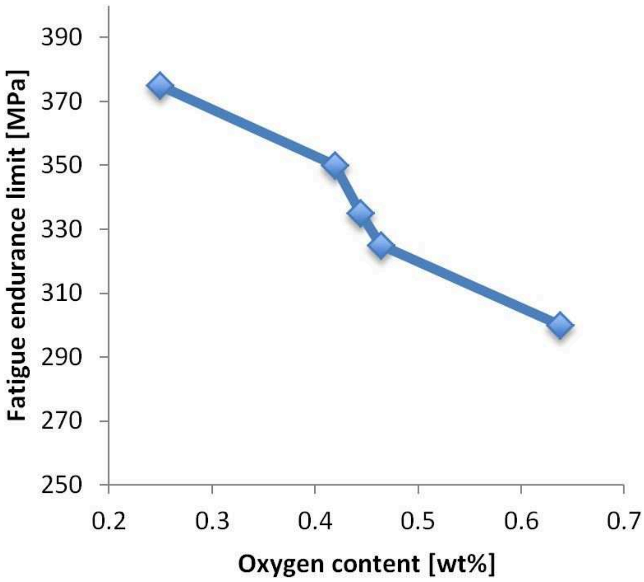
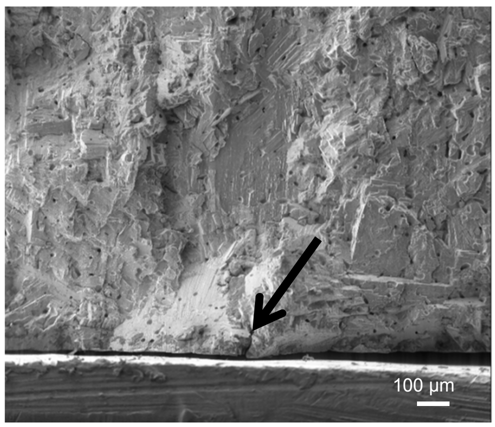


Fig. 11 Influence of oxygen content on the fatigue endurance limit (10^7 cycles) of MIM Ti-6Al-7Nb.

Fractography of fatigued specimens revealed the possible region for the crack initiation in low (Fig 12. a, b) and high oxygen content (Fig 12. c, d) specimens. The shot peening treatment creates residual compressive stresses in the surface of the samples preventing premature cracks in the rough MIM specimen surface. The crack nucleation seems to take place below the surface of the samples due to this.

a 

b 

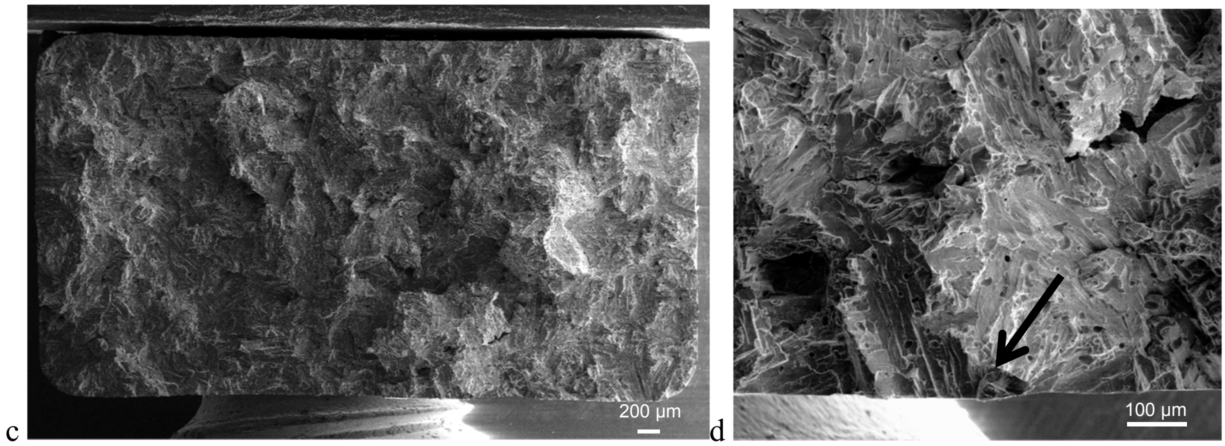


Fig. 12 Typical fatigue fracture surface of low oxygen (a, b) and high oxygen (c, d) content. Possible crack nucleation area.

Comparing the results with previous investigations [7], the fatigue life of shot peened MIM Ti-6Al-4V specimens can be up to 10^7 cycles under a stress amplitude of 450MPa when the oxygen content is 0.19 wt.%. In case of MIM Ti-6Al-7Nb samples, fatigue resistance is lower (375MPa) having an oxygen content of 0.25wt.%.

It is known [9] that an increase of oxygen content improves the fatigue resistance of unalloyed titanium. However, in MIM Ti-6Al-7Nb, the fatigue endurance limit is reduced. One cause could be the existence of locally brittle areas that cannot tolerate big strengths. Probably, the higher strength Ti6Al7Nb cannot negotiate local stress concentrations in such areas by plastic deformation as well as softer pure titanium. There are some investigations about the microstructural factors influencing the fatigue properties. Donachie [9] stated that the most important parameters affecting the fatigue behaviour of titanium alloys are the colony size of α and β lamellae and the width of the alpha lamellae in fully lamellar structures. At present, no investigations in the lamellar structure of the material were done.

Summary

In the present work, the influence of oxygen on the mechanical properties of Ti-6Al-7Nb processed by MIM was investigated and evaluated.

- Tensile and yield strength show a positive linear dependence on oxygen content. Both strength parameters increased in 10% when the oxygen content varies from 0.22 to 0.38 wt.%.
- The ductile behavior of the alloy is drastically affected if certain oxygen content is exceeded. The elongation drops by 13% when the oxygen content is above 0.38 wt.%.

Based on the results obtained, a comparison with the previous investigation of Ti-6Al-4V [4] can be made:

- The dependence of tensile properties on the oxygen content of Ti-6Al-7Nb is similar to Ti-6Al-4V.
- Ti-6Al-7Nb appears to be more tolerant to oxygen than Ti-6Al-4V in tensile behaviour.

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