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Final Draft
of the original manuscript:

Ferri, O.M.; Ebel, T.; Bormann, R.:

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In: Materials Science and Engineering A (2008) Elsevier

DOI: 10.1016/j.msea.2008.10.039

High cycle fatigue behaviour of Ti-6Al-4V fabricated by metal injection moulding technology

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Abstract

Titanium alloys exhibit an excellent combination of properties, including high specific strength and excellent bio-compatibility. Recently, parts made from titanium alloys such as Ti-6Al-4V have been manufactured by metal injection moulding (MIM) and exhibited excellent tensile properties (UTS > 800 MPa and ε > 15%). However to date, few information concerning the fatigue properties of such material has been presented in the literature. Thus, in the present study, the influence on the high cycle four point bending fatigue behaviour of Ti-6Al-4V alloy processed by standard and MIM techniques was examined. The first results indicated that the fatigue strength of components fabricated by MIM technique is significant lower than parts made by standard process. This behaviour is discussed in terms of mechanical properties, microstructure, composition, surface quality and crack initiation mechanisms.

Keywords: MIM, four point bending fatigue, Ti-6Al-4V alloy

1. Introduction

The Ti-6Al-4V alloy exhibits a high specific strength and stiffness, outstanding corrosion resistance and biocompatibility, which provide a great potential for biomedical applications. Consequently, titanium is a nearly ideal material for the development of medical bone reinforcement and replacement products [1]. However, the utilisation of titanium-based implants is limited by costly, multi-step process of fabrication and associated geometry design constraints. Metal injection moulding (MIM) is a technique that can provide minimization of such problems. MIM combines the material flexibility of powder metallurgy and the design flexibility of plastic moulding. Apart from the complex shape, many applications require good mechanical properties of the component, specially related to fatigue resistance. Favourably, the development of MIM using Ti-alloy powder has been very successful in recent years: alloys such as Ti-6Al-4V[2] and Ti-6Al-7Nb [3] can be manufactured by MIM with excellent strength levels (880 MPa, 815 MPa) and large plastic elongations (14.5 %, 8 %), respectively. Recently, Niinomi et al. [4] carried out an investigation concerning the fatigue properties of the Ti-6Al-4V components fabricated by MIM. However, little information on the crack initiation mechanisms and the influence of the porosity on high cycle fatigue behaviour of components fabricated by MIM are available on the literature. Therefore, in the present study, the influence of the fabrication process on the high cycle fatigue behaviour of Ti-6Al-4V alloy was investigated.

Usually, the high cycle fatigue tests are performed in a constant amplitude axial fatigue test machine following the ASTM standard (E466). According to this standard to ensure test section failure, the grip cross-sectional area should be at least four times the test section area. However, the manufacture of such component by MIM technique is complicated due to problems related to sample integrity after injection and sinter processes. Therefore, as a first step on the investigation of fatigue behaviour of Ti-6Al-4V components fabricated by MIM technique, the high cycle fatigue tests were performed in a four point bending configuration. Bending fatigue test requires a simple geometry, which can be easily processed by MIM technology.

2. Experimental

Two fabrication processes, standard and MIM technologies, were applied in order to obtain the Ti-6Al-4V specimens for the fatigue tests. A hot rolled and annealed commercial Ti-6Al-4V alloy cylinder with a diameter of 55 mm and a length of 250 mm supplied by Enpar, Germany was used to fabricate the first set of specimens. The samples were spark eroded to the final geometry shown in Fig. 1. In the following, these samples will be referred as reference material.

The second set of specimens was fabricated by MIM technology. The Ti-6Al-4V alloy powder used was supplied by TLS Technik GmbH, Germany, with a powder particle diameter below 45 μm as illustrated in Fig. 2. The powder has been produced by argon gas atomisation with respective impurities levels of: 880 μg/g in O, 165 μg/g in N and 20 μg/g in C.

The feedstock (mixture of metallic powder and binder) contained 32 vol. % of a binder system. The binder system is a mixture of polyethylene, paraffin and stearic acid. The metallic powder and the binder system were mixed in a Z-blade mixer at a temperature of 120°C during 2 hours under argon atmosphere. After granulation the feedstock was injected and moulded using an Arburg 320S machine. As moulded, the bending fatigue specimens measured approximately 10% bigger than the nominal values presented in Fig. 1. The specimens for tensile test were moulded in “dog-bone” shape with a nominal length of 89.8 mm and gauge diameter of 4.8 mm. The paraffin was removed by chemical debinding with heptane at 40°C during 20 hours. Final removal of the remaining binder and subsequent sintering were carried out in a single-step heat treatment run. Sintering was conducted in a cold wall furnace with tungsten heating elements and shield packs of molybdenum. Sintering was performed in vacuum of 10^{-5} mbar at 1250°C with a holding time of 2 hours, followed by controlled furnace cooling. A more detailed description of the MIM process performed can be found in [5].

Fatigue properties of titanium alloys are largely determined by surface defects [6], microstructures[7], and crystallographic textures [8]. In order to minimize the influence of surface difference, present in the specimens due to standard and MIM route fabrication, the samples were exposed to shot peening. The shot peening was conducted on an air-blast machine using fine zirconium oxide particles with a diameter of 500 μm. The air pressure was 4 bar with an exposure time of 10 seconds for each surface sample. The nozzle diameter was 6 mm and the work distance applied was 50 mm. No crystallographic texture effect was investigated in this study.

High cycle fatigue testing was done in four-point bending using a resonance machine fabricated by RUMUL. Tests were conducted under load control with a cyclic frequency of ~95 Hz (sine wave) at a load ratio $R=\sigma_{min}/\sigma_{max}$ of 0.2. All the experiments were carried out at room temperature in air. Three configurations were tested: reference material samples with shot peening, MIM samples with and without shot peening. The fatigue endurance limit was defined as 10^7 cycles.

The maximum initial tensile stress σ within the loaded bar was calculated using equation 1 where F is the applied force, L is the gauge length, W is the bar thickness and $(I_{xx})_r$ is the moment of inertia of rectangular cross section with corner radius r.

$$\sigma = \frac{FLW}{24(I_{xx})_r} \qquad \textbf{. 1}$$

The true moment of inertia $(I_{xx})_r$ about the neutral axis x-x is:

$$(I_{xx})_r = \frac{B(W-2r)^3}{12} + \frac{(B-2r)r^3}{6} + (1/2)(B-2r)(W-r)^2 r + 4r^4\left(\frac{\pi}{16} - \frac{4}{9\pi}\right) + \pi r^2\left[\frac{W}{2} - r\left(1 - \frac{4}{3\pi}\right)\right]^2 \qquad \textbf{. 2}$$

where B is the bar width and r is the corner radius.

Light microscopy was used to investigate the influence of shot peening on the surface quality of MIM samples. The fracture surfaces of broken specimens were analyzed by a stereoscope (LEICA MZ95) in order to identify the crack initiation location. The microstructure and fracture surface of reference material and MIM samples were investigated using a scanning electron microscope (ZEISS – DSM962). The observations were focused upon the crack initiation location.

The impurity levels such as oxygen, nitrogen and carbon of reference material and MIM specimens were determined using a conventional LECO melt extraction system. The average equiaxed grain size, the alpha colony size and the percentage of porosity were measured using an image analysis system. The tensile tests were conducted for MIM samples at room temperature at a strain rate of $1.2 \times 10^{-5}\ s^{-1}$.

3. Results and discussion

3.1. Microstructure, tensile property and chemical composition

The reference material Ti-6Al-4V alloy used in this study exhibited a fully equiaxed microstructure, as shown in Fig. 3, with an alpha grain size of approximately 5 µm. The MIM samples presented a fully lamellar microstructure, as illustrated in Fig. 4. The alpha colony size, alternating alpha and beta plates with same orientation, was approximately 100 µm. The apparent porosity observed on MIM samples was 3.5 vol.% with near-circle shape and diameter size of approximately 10 µm. This significant difference on the microstructure is a consequence of fabrication process and it will influence the mechanical properties of the materials.

Table 1 compares the tensile properties of reference material and MIM samples. The values for the reference material were obtained from the supplier. The results of MIM samples were based on the tensile test of five so called "dog-bone" samples. Two main aspects contribute to lower strength of samples fabricated by MIM technique compared to reference material. Firstly, about 3.5 vol.% of porosity is present in the MIM sample. Secondly, the grain size of reference material samples is 20 times smaller than MIM samples. Thus, referring to the Hall-Petch effect, the material with finer microstructure is expected to exhibit higher yield strength. This is in agreement with the obtained results. Interestingly, despite of the porosity, the ductility of the MIM samples was not significantly affected.

Another parameter that could influence the mechanical properties of the material is the concentration of interstitial elements in both alpha and beta phases. The oxygen, carbon and nitrogen contents are given in Table 2. Theses elements increase strength and decrease ductility [9]. However, the higher concentration of interstitial elements in the MIM samples did not promote a significant decrease of ductility and an increase of strength compared to reference material samples. Therefore, predominant parameters that explain the tensile properties of the studied materials are most likely the porosity and grain size rather than of interstitial alloying elements effect.

3.2. Fatigue behaviour

The results of high cycle bending fatigue tests for reference samples with shot peening and MIM samples with and without shot peening are shown in Fig. 5. The MIM samples with shot peening showed a significant higher endurance limit (450 MPa) than those without shot peening (350 MPa). This behaviour can be explained by difference on surface quality and crack initiation mechanisms. It is well known that surface quality and residual stresses can affect the fatigue behaviour of engineering materials [10]. Figs. 6a and b illustrate the surface roughness of MIM samples without and with shot peening, respectively. These cavities observed on the surface of MIM samples without shot peening can be related to injection and debinding steps of MIM processing. During injection, the sample surface is exposed to higher shear stress compared to the interior regions of the sample due to turbulence effect promoted by fluid (hot feedstock) and mould wall interaction. Such shear stress could promote local separation of binder/powder mixture. This separation leads to regions where only binder is present with no powder particles. Consequently, the notches are formed during thermal debinding and sintering in these specific surface regions due to missing particle powder contact. Another possibility is the fact that the powder particles, on specific surface sample regions after injection, could be surrounded by only one binder component (e.g. paraffin) of the binder system. Therefore, during chemical debinding these particles will be removed together with the paraffin which leads to a local empty space.

These notches are, in the fracture mechanical point of view, cracks located directly on the samples surface. Consequently, the high cycle fatigue behaviour will be affected by the presence of such cracks. As described in the next section, fractographic analysis showed that the crack initiation location is related to the presence of these notches on the MIM samples surface. With application of shot peening, these notches were practically totally removed by local plastic deformation as can been seen in Fig. 6b. Additionally, shot peening induces compressive residual stress in the material surface, which leads to a increase of fatigue strength [11].

In order to evaluate the influence of the MIM process on the fatigue strength, four point bending fatigue tests were carried out on specimens fabricated by a standard process for comparison. The reference samples with shot peening demonstrated a fatigue endurance limit of approximately 890 MPa. Unfortunately, it is not possible to compare directly this result with literature values due to the lack of data related to high cycle four point bending fatigue test of Ti-6Al-4V alloy. Akahori et al. [12] carried out high cycle axial fatigue test with R=0.1 and found a value of 800 MPa for fatigue endurance limit of Ti-6Al-4V alloy with equiaxed microstructure. This lower literature value of fatigue endurance limit compared to our reference material is not unexpected. Morrisey et al. [13] described the influence of stress ratio R on mean and amplitude stresses for Ti-6Al-4V alloy at fatigue life of 10^7 cycles. Increasing of R leads to a decrease of stress amplitude and an increase of mean stress. Therefore, it is expected that tests with higher R values cause greater values for maximum stress. Furthermore, Niinomi et al.[4] investigated fatigue property of Ti-6Al-4V components fabricated by MIM in axial test configuration with R=0.1 and they found a value for endurance limit of approximately 380 MPa, which is close to the value obtained in this study (350 MPa) for MIM samples without shot peening. Additionally, it should be pointed out that the shot peening promoted an increase in the fatigue strength which could be an additional reason for the higher fatigue endurance limit observed for our reference material compared to literature values. Therefore, it is possible to assume that the results demonstrated in this work are in an acceptable range with literature data.

From the results obtained, it is obvious that the fatigue strength for MIM samples with shot peening is significant lower than the reference samples with shot peening. Possible explanation for such behaviour will be discussed in the next section.

3.3. Fatigue fractography

Fig. 7 shows a typical fracture surface from MIM samples without shot peening, indicating that the fatigue crack nucleation started at the surface. Based on the shape of the rough region, it is possible to suppose that the starting point was close to the centre of the sample surface. In Fig. 8a, the white arrow indicates the surface exposed to tensile stress during four point bending fatigue test. The white arrow in Fig. 8b appoints for the assumed crack initiation location. Two distinct regions are visible: one region shows a normal fracture surface, where it is possible to identify lamellae boundaries (area 1). The other region illustrated a powder surface (area 2), which demonstrate that in this region the particles were not connected. This disconnection of the particles resembles a notch located at surface sample, as described in the previous section and illustrated by Fig. 6a. Therefore, the fatigue strength directly depends on the size and location of such defects. The maximum observed size of the notches located at the samples surface was approximately 100 μm. Apparently, these defects are randomly distributed over the sample surface.

As shown before, these notches were apparently not present on the surface of MIM samples after shot peening (Fig. 6b) due to local plastic deformation. A typical fracture surface of MIM samples with shot peening is illustrated in Fig. 9. The shape of rough region indicates that the crack initiation location was in some place near to the lower left corner. Scanning electron micrographs of fracture surface of the MIM sample with shot peening are shown in Figs. 10a and b. The white arrows in Figs. 10a and b indicate the surface under tensile stress and the crack initiation location, respectively. Note that for all samples the crack initiated at a location inside the samples instead of the surface, except for the samples marked with brackets in Fig. 5. In these samples, the local plastic deformation promoted by shot peening application was not sufficient to close the surface defect. Most probably, there is a critical shape and size of the surface defect that limits the shot peening benefit presented in the others MIM samples. As can been seen in Fig. 11 the crack initiated at the surface instead of the inner region. Therefore, the fatigue life of theses samples is closer to that of the MIM samples without shot peening.

Internal fatigue origins are not common, however after shot peening application, it is frequently encountered in titanium alloys [14-16]. Crack initiation in alpha/beta titanium alloys is invariably associated with the formation of quasi-cleavage facets [17]. The formation of such facets is related to the fact that the weak grain, favourably orientated for slip, generates a dislocation pile-up at the boundary with the neighbouring strong grain. The pile-up leads to the required combination of shear and tensile stresses on the unfavourably plane which, induces facet formation. Subsurface crack initiation sites in fully lamellar Ti-6Al-4V alloy were related to cross-colony slip-band fracture [18]. This fracture behaviour is also presented in the fracture surface of MIM sample with shot peening. The crack is supposed to nucleate at the intersection of two slip bands, as indicated by the white arrow in Fig. 10b. Apart from the cross-colony fracture facets, the presence of pores is another origin of the crack nucleation in MIM samples. In our investigations, the fracture surface of MIM samples with shot peening always had both features (pores and cross-colony fracture facets) present at its nucleation site.

The subsurface crack initiation site was also observed in all tested reference samples with shot peening and a typical fracture surface is illustrated in Fig. 12. As for the previous fracture surface pictures, the white arrows in Figs. 13a and b indicated the surface under tensile stress and the crack initiation location, respectively. The quasi-cleavage facets are also presented on the crack nucleation location of reference material samples (Fig. 13). However, the size of these facets for reference samples is smaller than the MIM sample, which is a consequence of the different microstructural scale. The size of theses facets are related to the slip-band length, which is directly related to the alpha grain size (~5 µm in the case of equiaxed microstructure) and alpha colony size (~100 µm for the fully lamellar microstructure). As described by Lütjering [19] the high cycle fatigue strength (resistance to crack nucleation) depends primarily on the resistance to dislocation motion, and therefore in most cases on the yield stress. Consequently, it is expected that a material with a fine equiaxed microstructure (reference samples) demonstrates higher fatigue resistance than a material with a relative coarse fully lamellar microstructure (MIM samples). Stubbington et al. [7] investigated the influence of microstructure size on the fatigue behaviour of Ti-6Al-4V alloy. They concluded that by refinement of the microstructure from fully lamellar to an equiaxed microstructure, the fatigue strength could be improved from 440 MPa to 670 MPa. However, the fatigue behaviour difference presented in this investigation cannot be explained only by microstructure size difference. In fact, the existence of pores in the MIM samples together with a larger microstructure are the mainly responsible factors for the significant decrease of the high cycle fatigue strength. Nevertheless, it is important to note that the fatigue strength of cast Ti-6Al-4V alloy (endurance limit usually in the range of 230 to 330 MPa) is much lower than the reference material (890 MPa) presented in this study. Therefore, it is possible to assume, based on our investigation, that Ti-6Al-4V alloy components fabricated by MIM present superior fatigue strength when compared to cast Ti-6Al-4V alloy.

4. Conclusions

This investigation has been the first step to determine the influence of the MIM process on the high cycle fatigue strength of Ti-6Al-4V alloy. It was shown that the fatigue properties of components fabricated by MIM technique are significantly lower than the components fabricated by a standard process. The main reasons for such behaviour are related to a coarser microstructure and the presence of pores in the MIM microstructure compared to the reference material. The application of shot peening on MIM specimens promoted an increase on the fatigue resistance, mainly due to change on surface roughness caused by local plastic deformation of the notches located at the surface of the sintered samples. Furthermore, the crack initiation location in the shot peening samples was observed to be at the subsurface level, which contributes to the observed improvement on the high cycle fatigue strength. Nevertheless, if the Ti-6Al-4V alloy components fabricated by MIM technique are to be used on applications, where a fatigue resistance in the range of wrought material is required, then an optimization of fabrication process is still necessary. Consequently, further experiments on the optimization of MIM process will be carried out in the near future.

Acknowledgments

The authors would like to thank those who assisted with this study: Dr. Mustafa Koçak and Dr. Jan Bober for the provision of fatigue test facilities and helpful discussions.

References

[1] M. M. E. Nyberg, K. Simmons, K. Scott Weil, Mat. Scie. and Eng. (2005) 336-342.

[2] Y. Itoh, T. Harikou, K. Sato, H. Miura, in: H. Danninger, R. Ratzi (Eds.), EURO PM 2004, vol 4, 2004.

[3] W. Limberg, E. Aust, T. Ebel, R. Gerling, B. Oger, in: H. Danninger, R. Ratzi (Eds.), EURO PM 2004, vol 4, 2004.

[4] M. Niinomi, T. Akahori, M. Nakai, K. Ohnaka, Innovations in Titanium Technology, in: M. N. Gungor, M. A. Imam, F. H. Froes (Eds.), TMS2007 136th Annual meeting & exhibition, 2007, pp. 209-217.

[5] B. Oger, T. Ebel, W. Limberg, The manufacture of highly-ductile and geometrically complex MIM-parts based on TiAl6V4, in: EURO PM 2006, vol 2, 2006, pp. 191-196.

[6] P. A. B. D. F. Neal, Acta Metallurgica 24 (1975) 59-63.

[7] C. A. Stubbington, A. W. Bowen, J. Mat. Sci. 9 (1974) 941-947.

[8] A. W. Bowen, Acta Metal. 23 (1975) 1401-1409.

[9] P. S. Prevey, Fatigue and fracture, in: Metal Hanbook, vol 19, Metal Park, 1986, pp. 829-853.

[10] G. E. Dieter, Mechanical Metallurgy, MCGraw-Hill, New Yourk, 1986.

[11] D. T. Stephen, W. S. David, D. L. Chris, G. S. Paul, The Minerals, Metals & Materials Society, 1997, p. 239-243.

[12] T. Akahori, M. Niinomi, Mater. Sci. Eng. A243 (1998) 237-243.

[13] D. L. M. R. J. Morrissey, T. Nicholas International Journal of Fatigue 21 (1999) 679-685.

[14] M. Y. P. Costa, H. J. C. Voorwald, W. L. Pigatin, V. A. Guimarães, M. O. H. Cioffi, Mater. Research 9 (2006) 107-109.

[15] S. A. Namjoshi, V. K. Jain, S. Mall, Eng. Mater. and Tech. 124 (2002) 222-228.

[16] X. P. Jiang, C. S. Man, M. J. Shepard, T. Zhai, Mater. Sci. Eng. A 468-470 (2007) 137-143.

[17] M. R. Bache, Inter. J. Fatigue 21 (1999) 105-111.

[18] B. L. B. R. K. Nalla, J. P. campbell, J. O. Peters, R. O. Ritchie, Met. and Mater. Transac. 33A (2002) 899-918.

[19] G. Lütjering, Mat. Scie. and Eng. A 243 (1998) 32-45.

Figure(s)

Figures

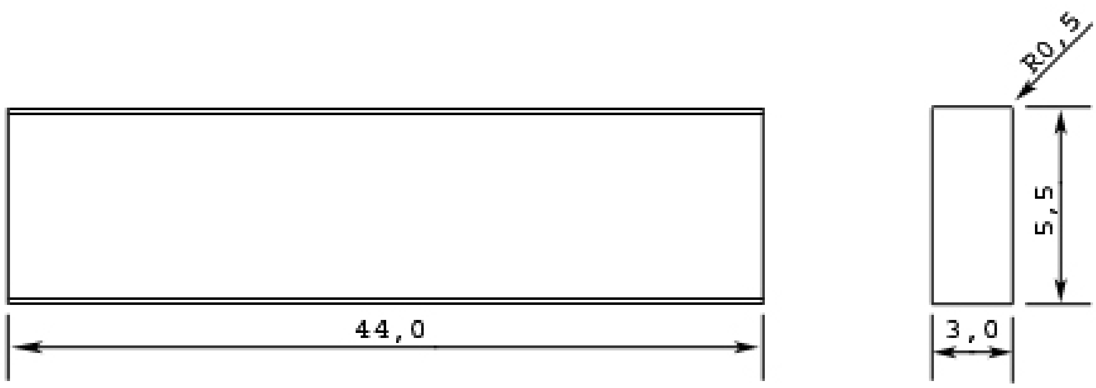

Fig. 1. Geometry and dimensions of bending fatigue specimens. Units are in millimetres.

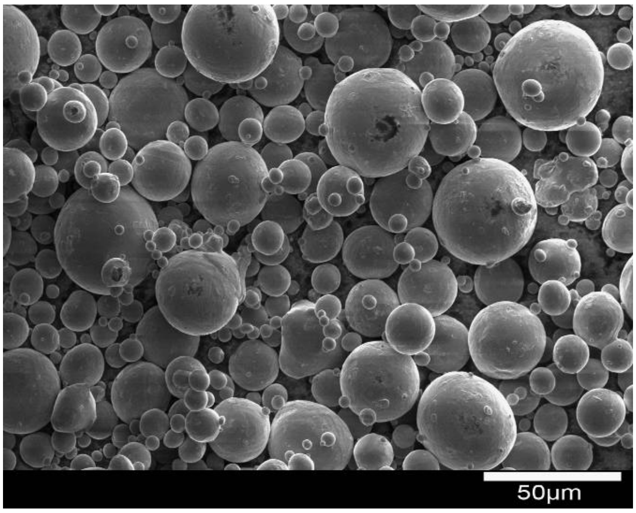

Fig. 2. Scanning electron microscope image (SE-mode) of the powder fraction size used for the present metal injection moulding experiments.

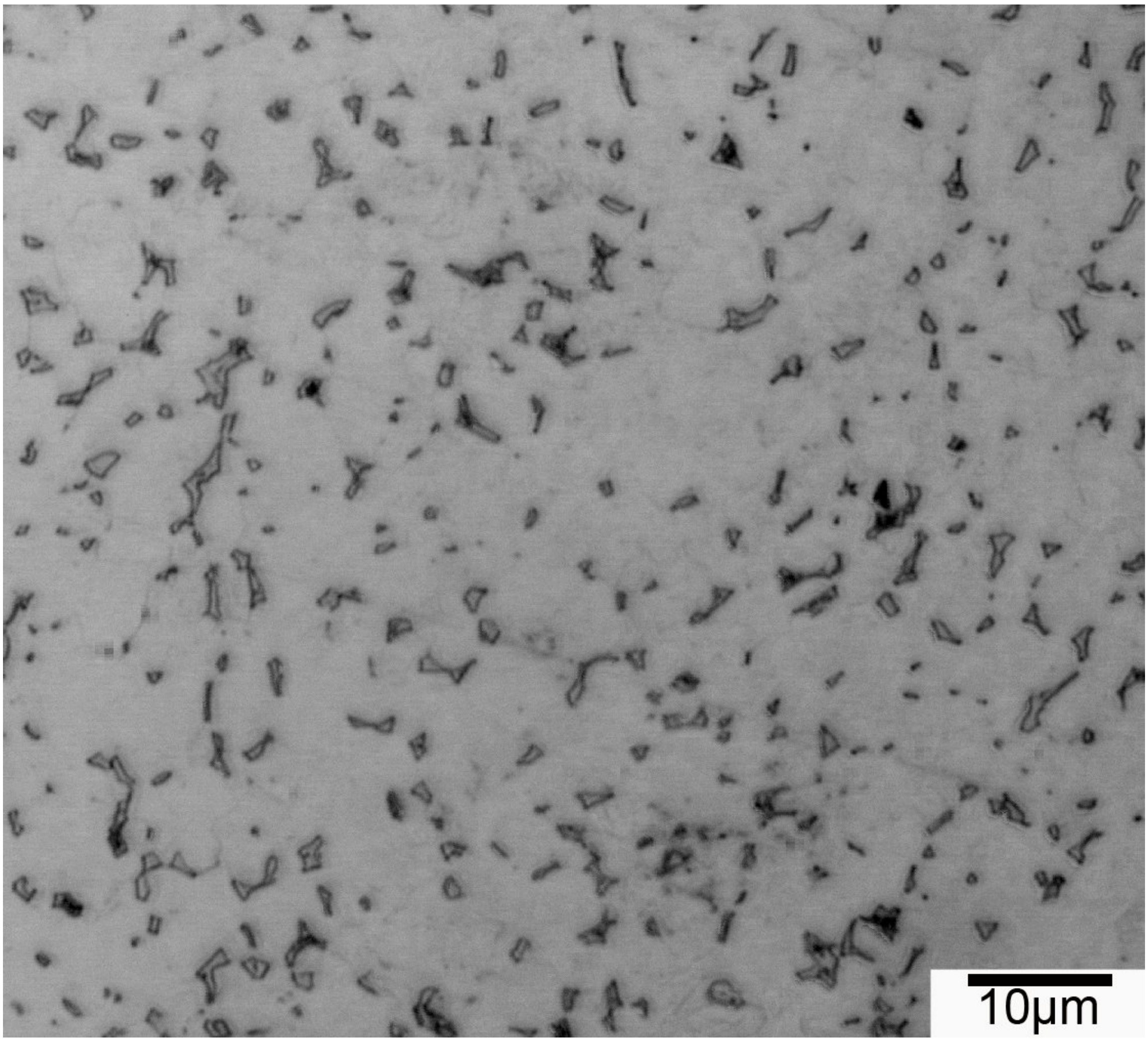

Fig. 3. Light microscopy of reference material (equiaxed microstructure)

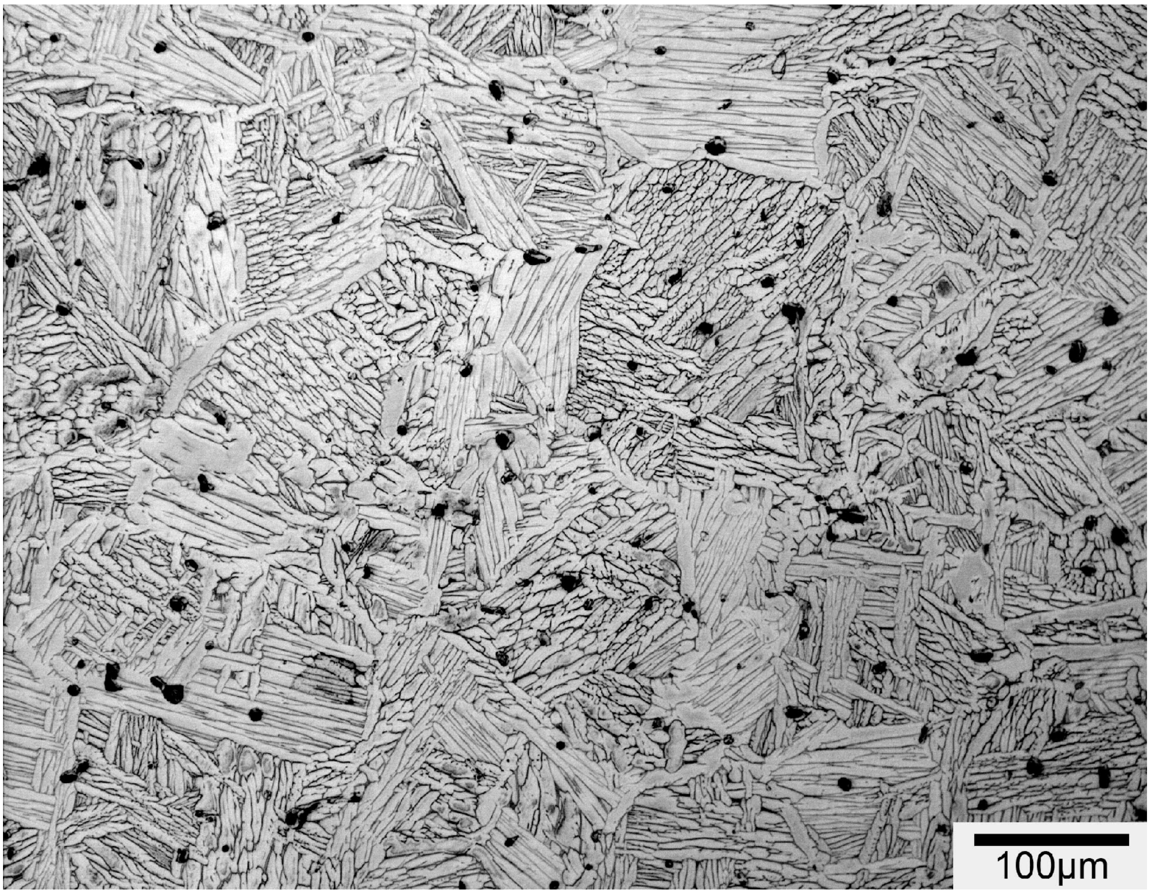

Fig. 4. Light microscopy of MIM sample (fully lamellar microstructure)

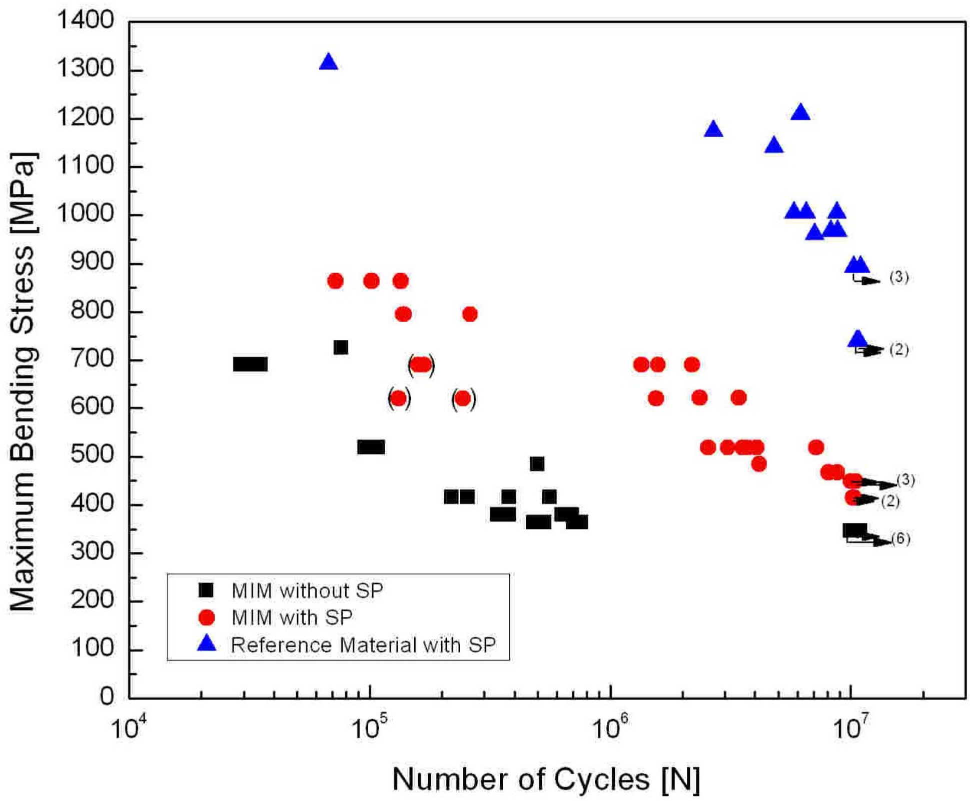

Fig. 5. S-N curves for reference material samples with shot peening (SP) and MIM samples with and without shot peening (SP).

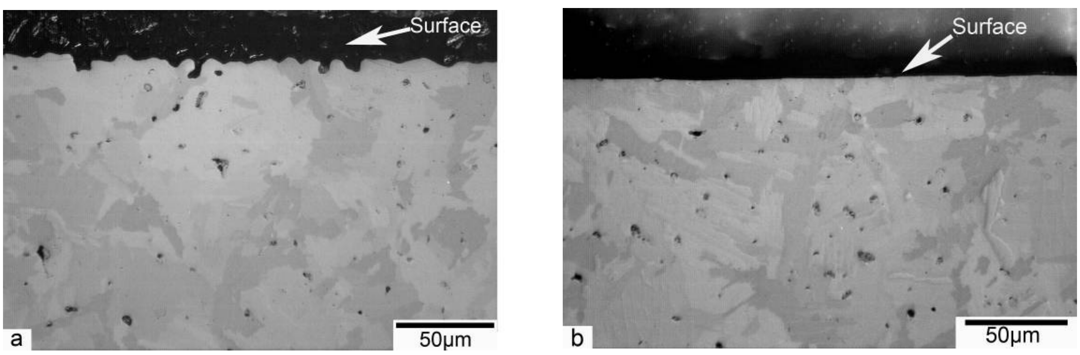

Fig. 6. Surface quality of MIM samples without application of shot peening (a) and with application of shot peening (b).

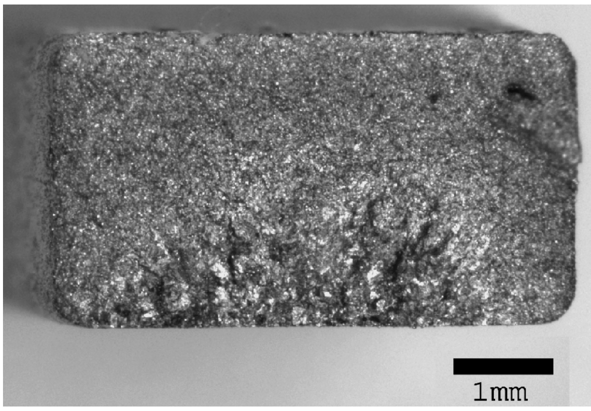

Fig. 7. Typical fracture surface of MIM sample without shot peening

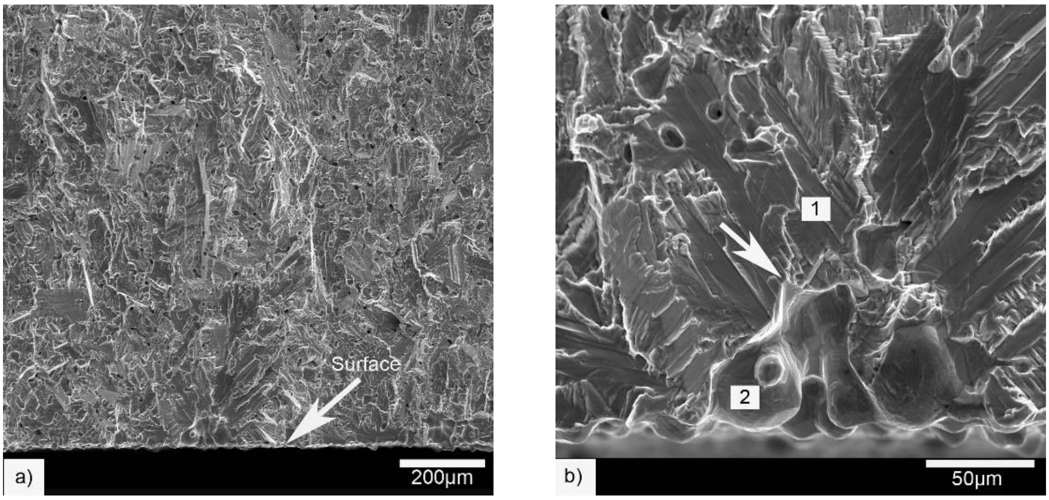

Fig. 8. Fracture surface of MIM sample without shot peening, a) lower magnification b) higher magnification of crack initiation location.

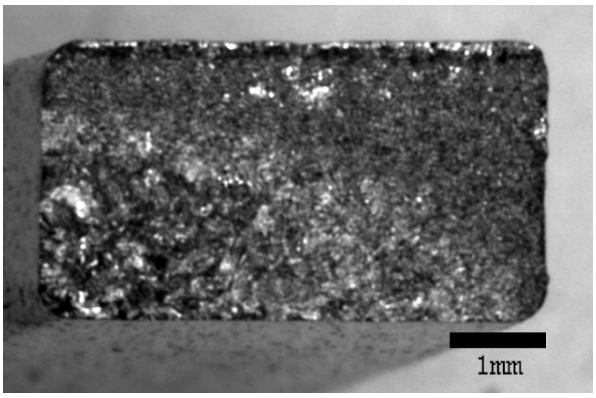

Fig. 9. Typical fracture surface of MIM sample with shot peening

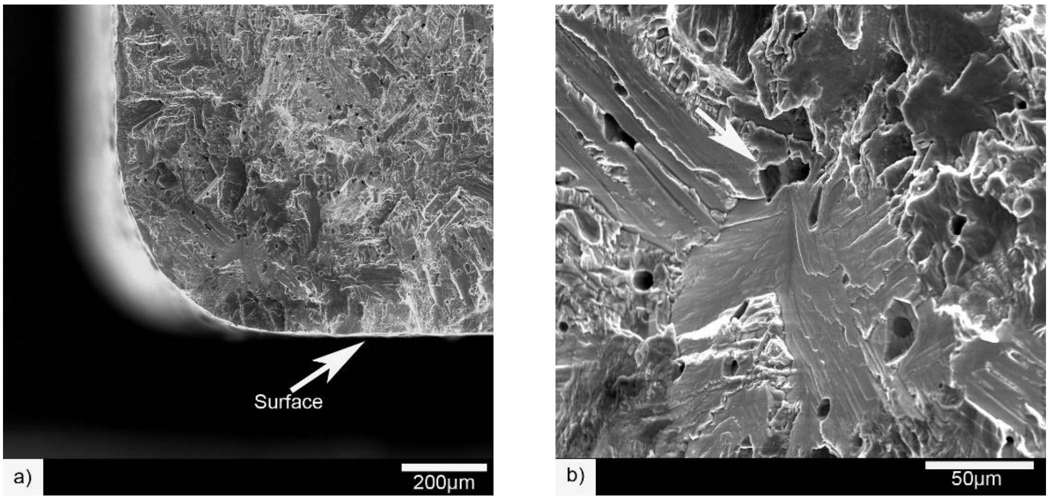

Fig. 10. Fracture surface of MIM sample with shot peening, a) lower magnification b) higher magnification of crack initiation location.

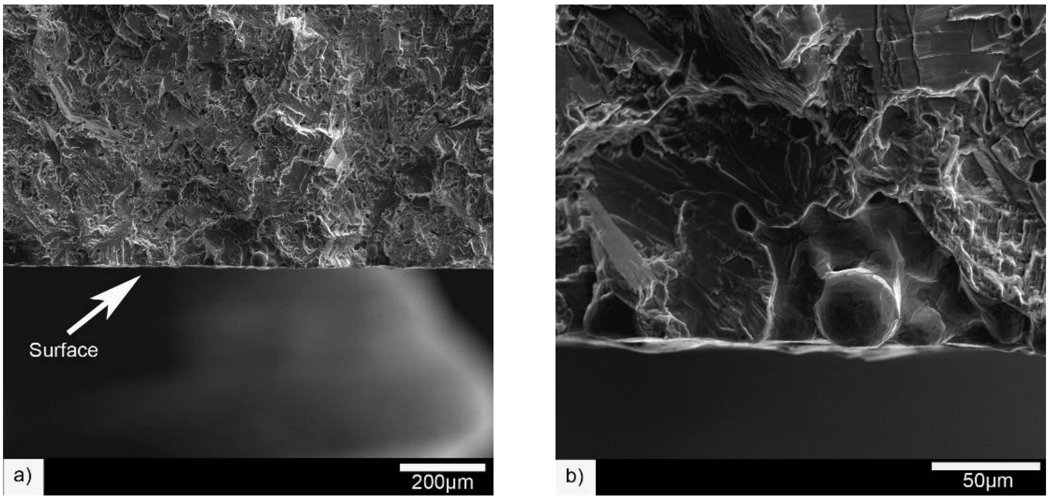

Fig. 11. Typical fracture surface of MIM samples (Fig. 5 with brackets) with shot peening, a) lower magnification b) higher magnification of crack initiation location.

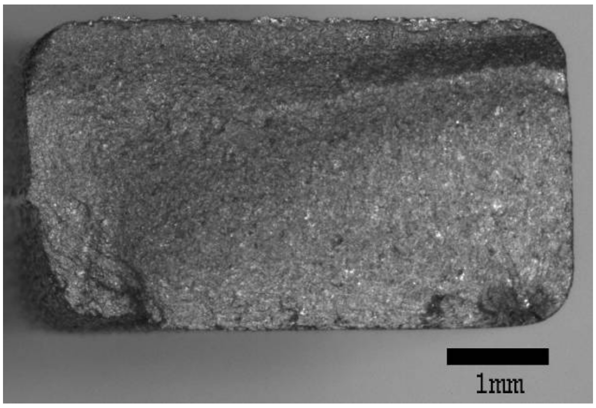

Fig. 12. Typical fracture surface of reference material.

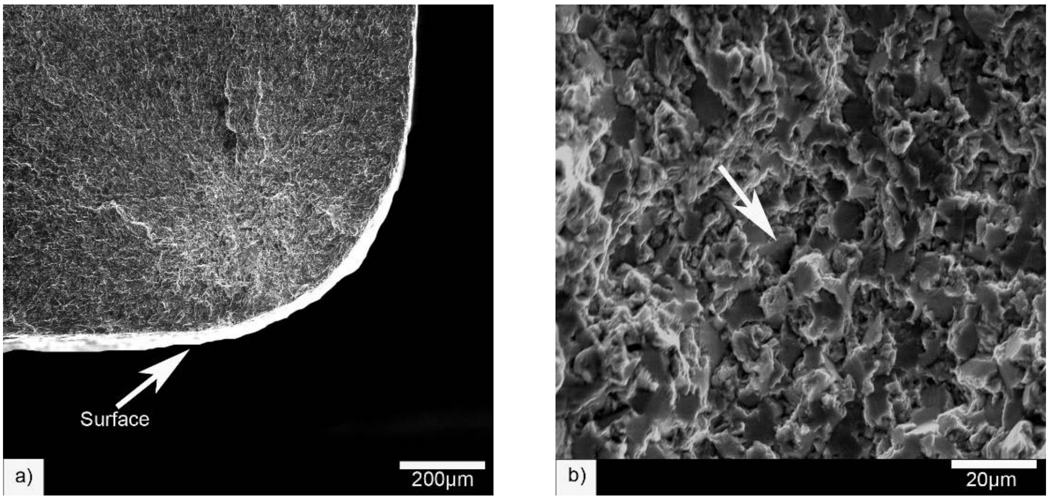

Fig. 13. Fracture surface of reference sample with shot peening, a) lower magnification b) higher magnification of crack initiation location.

Tables

Table 1. Tensile properties of melted and MIM configurations.

Material	Yield strength, [MPa]	Tensile strength, [MPa]	Percentage elongation, [%]
Reference Material	Min. 826	Min. 895	Min. 10
MIM	700	800	15

Table 2. Chemical concentration of interstitial alloying element.

Material	O [μg/g]	C [μg/g]	N [μg/g]
Reference Material	1260	150	164
MIM	1900	450	180

*** Detailed Response to Reviewers**

Ms. Ref. No.: MSEA-D-08-01973
Materials Science & Engineering A

Reviewer #1: Comment #1: In the introduction, first paragraph and last sentence, the authors mentioned that they did not find any publication about fatigue properties of titanium fabricated with MIM. They should have a look to the paper of Niinomi, Mitsuo and other from the Department of Biomaterials Science, Institute for Materials Research, Tohoku University published at the Conference Innovations in Titanium Technology as held at the 2007 TMS Annual Meeting; Orlando, FL; USA; 25 Feb.-1 Mar. 2007. The paper presents the mechanical properties of Ti-6Al-4V, particularly its tensile and fatigue properties, fabricated by MIM (Ti64). They found that the fatigue strengths of Ti64 subjected to HIP is the highest in the low- and high-cycle fatigue-life regions. Its fatigue strength is comparable to that of wrought Ti-6Al-4V. The authors should at least add a reference to this paper.

Change:

In the introduction, first paragraph:

"However, no data of titanium components fabricated by MIM technology related to fatigue properties are available on the literature yet." Removed

"Recently, Niinomi et al. [4] carried out an investigation concerning the fatigue properties of the Ti-6Al-4V components fabricated by MIM. However, little information on the crack initiation mechanisms and the influence of the porosity on high cycle fatigue behaviour of components fabricated by MIM are available on the literature." *Added*

Comment #2: In the Experimental, first paragraph second sentence, the authors mentioned that the reference material is "Melted and annealed commercial Ti-6Al-4V" and was supplied by Enpar in Germany. It is not clear what it is meant by "Melted". Is it cast material? Based on the microstructure shown in figure 3, I would say that it is not a cast material but bar stock obtained by forging, hot-rolling and cold forming and it is in a Mill Annealed condition. The equiaxe structure is obtained by a globularization heat treatment done after a thermomechanical treatment. To my knowledge, Enpar is supplying Ti64 bar in forged, hot rolled, hot or cold formed and cold drawn quality. This may explain the elevated mechanical properties obtained with the reference material. Such mechanical properties (especially fatigue) are difficult to obtain with cast material. In addition, cast material shows, normally, a well defined Widmanstatten structure. The authors should verify and report the exact state of the material since it is determining the mechanical properties.

Change:

In the Experimental, first paragraph second sentence:

"Melted and annealed commercial Ti-6Al-4V" Removed

"A hot rolled and annealed commercial Ti-6Al-4V alloy" *added*

Obs.: all melted terms in the following text was replaced by standard or reference material

Comment #3: In the Experimental, page 5, last paragraph starting with "Fatigue properties of …", in the last sentence, the authors said that no crystallographic texture was observed for the studied microstructures, it is not clear if the authors determined experimentally this claim. Did they measure the texture to arrive to this finding and how they looked at the texture? The authors should clarify this point.

Change:

In the Experimental, page 5, last paragraph:

"No crystallographic texture was observed for the studied microstructures."Removed

"No crystallographic texture effect was investigated in this study."added

Comment #4: In the Experimental, page 6, paragraph starting with "The maximum tensile…", I think the authors should refer to the "The maximum initial tensile stress…" since the maximum tensile stress may change during the fatigue test.

Change:

In the Experimental, page 6, paragraph:

"The maximum tensile stress"Removed

"The maximum initial tensile stress"Added

Comment #5: In Figure 5, the triangles represent the "Base Metal with SP". I think the authors meant the "Reference Metal with SP". The legend should be modified accordingly.

Change:

The legend was modified

Comment #6: In section 3.3 Fatigue Fractography, page 13, the authors mentioned that "it is expected that the reference material with a fine equiaxed microstructure demonstrates higher fatigue resistance than the MIM samples. It is expected that the fatigue strength is proportional to the Y.S. But, it would be interesting to know what are the contribution of the microstructure and the contribution of the porosity. To start this discussion, the authors should refer to papers such as the one published by Stubbington and Bowen in the Journal of Materials Science 9 (1974) 941-947. They would be able to discuss further the impact of microstructure and annealing of properties. This paper shows the impact of the beta-anneal (similar to a sintering cycle) on the fatigue strength when compared with other controlled microstructures via thermomechanical treatment. One interesting addition to this work would be the fatigue behaviour of the reference material subjected to a sintering cycle which, most likely, will give a Widmanstatten structure and a significant grain growth. This experiment would give good indication about the effect of internal porosity and microstructure on the fatigue strength. It is unfortunate that the authors did not have this experiment in this paper. But, I don't think it is required for this paper. But the authors should try to compare their results to investment cast material which is the usual benchmark to MIM material.

Change:

To start this discussion, the authors should refer to papers such as the one published by Stubbington and Bowen…

"Stubbington et al. [7] investigated the influence of microstructure size on the fatigue behaviour of Ti-6Al-4V alloy. They concluded that by refinement of the microstructure from fully lamellar to an equiaxed microstructure, the fatigue

strength could be improved from 440 MPa to 670 MPa." Added in In section 3.3 Fatigue Fractography last paragraph after the sentence: "Consequently, it is expected that a material with a fine equiaxed microstructure (reference samples) demonstrates higher fatigue resistance than a material with a relative coarse fully lamellar microstructure (MIM samples)."

But the authors should try to compare their results to investment cast material which is the usual benchmark to MIM material.

Added in section 3.3 Fatigue Fractography last paragraph, last sentence

"Nevertheless, it is important to note that the fatigue strength of cast Ti-6Al-4V alloy (endurance limit usually in the range of 230 to 330 MPa) is much lower than the reference material (890 MPa) presented in this study. Therefore, it is possible to assume, based on our investigation, that Ti-6Al-4V alloy components fabricated by MIM present superior fatigue strength when compared to cast Ti-6Al-4V alloy." Added

Comment #7: In the Conclusion, the authors refer to a "standard melting process". As mentioned before, this is not clear. There is not such standard. The authors must refer to the precise metallurgical state of the reference material.

Change:

"standard melting process" removed

"a standard process"added

Comment #8: In the Conclusion, last sentence, the author suggest that MIM could be only used if the fatigue behaviour is improved via optimisation of fabrication process. I think that the actual value of the fatigue strength is fairly good when compared to cast material (not to forged material as in this paper). In addition, the design of the component can be "adjusted" to operate properly, despite the lower fatigue strength, which can lead to application of MIM even where "good" fatigue resistance is required. Don't be to strong with the MIM material!

Change:

"Nevertheless, if the Ti-6Al-4V alloy components fabricated by MIM technique are to be used on applications, where a good fatigue resistance is required, then an optimization of fabrication process is still necessary" removed

"Nevertheless, if the Ti-6Al-4V alloy components fabricated by MIM technique are to be used on applications, where a fatigue resistance in the range of wrought material is required, then an optimization of fabrication process is still necessary" added

Reviewer #2: Correct some minor mistakes in writing. Is "cavity" the correct word? Maybe "notch"?
The reference material is specified as melted material. Please explain the production route and how it achieves the fine microstructure. Fig. 3 does not reveal the grain boundaries.

Change:

All cavity terms during the following text was changed by nocth
The Fig. 3 was changed by a figure with higher magnification.

Further addition to the discussion:

Change:

In the section 3.2. Fatigue behaviour, third paragraph after. “Therefore, it is expected that tests with higher R values cause greater values for maximum stress.” Addition of: “Furthermore, Niinomi et al.[4] investigated fatigue property of Ti-6Al-4V components fabricated by MIM in axial test configuration with R=0.1 and they found a value for endurance limit of approximately 380 MPa, which is close to the value obtained in this study (350 MPa) for MIM samples without shot peening.”