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Materials Science Forum, Light Metals Technology V (2011)
Trans Tech Publications

DOI: 10.4028/www.scientific.net/MSF.690.181

Materials Science Forum *Online: 2011-06-14*
ISSN: 1662-9752, Vol. 690, pp 181-184
doi:10.4028/www.scientific.net/MSF.690.181

Metal Injection Moulding of Titanium

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Keywords: Metal Injection Moulding, Titanium alloy, Fatigue, Grain refinement, Titaniumborides

Abstract. A route to handle titanium specific challenges as oxygen and carbon sensitivity when applying Metal Injection Moulding is presented. It is shown that by appropriate processing components with excellent mechanical properties can be manufactured from Ti-6Al-4V alloy powder. In addition, alloy modification by simple powder metallurgical methods is performed in order to optimise the fatigue properties by changing the microstructure of the sintered part.

Introduction

Metal Injection Moulding MIM could be an effective alternative for the processing of titanium based alloys: titanium is rather expensive and difficult to be machined, casted or forged. Thus, at least two of the benefits of MIM as being a very resource efficient technique and facilitating fragile and complex geometries would come into application. However, even if MIM of titanium shows a steadily growing interest, its commercial availability is still strongly limited [1-7]. The main reason is the strong affinity of titanium to interstitials, namely O, N and C. They influence the mechanical properties strongly by increasing the strength, but deteriorating the ductility at even small amounts. Their effect can be summed up to an oxygen equivalent calculated by $O_{eq} = c_O + 2\ c_N + 0.5\ c_C$, c_O, c_N and c_C being the concentrations of O, N and C, respectively. In addition, Fig 1 shows the principal difference of MIM and wrought microstructures. The MIM microstructure has a different morphology, is coarser and possesses residual porosity. Thus, basically most of the mechanical properties will be inferior compared to those of wrought material. In this paper, it is shown, that these obstacle can be overcome by appropriate processing and choice of raw material and even slight alloy modifications, so excellent mechanical properties can be achieved.

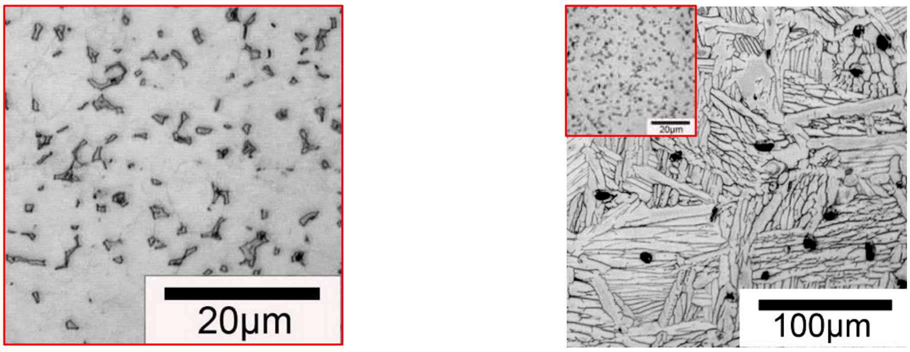

Fig. 1 a) Typical equiaxed microstructure of wrought Ti-6Al-4V

b) Typical microstructure of MIM processed Ti-6Al-4V (inset shows Fig. 1a in same scale).

Requirements for Metal Injection Moulding of Ti-6Al-4V

Guideline for successful application of MIM is to minimize any interstitials pick-up within the entire production chain from powder production to sintering. At HZG a MIM route specifically designed for reaching this goal is successfully applied:

- As powders only gas-atomized products are used, usually the ELI (extra low interstitials) variant of Ti-6Al-4V, also named as grade 23 in the ASTM B348 standard or ASTM F 136 for medical applications. This takes into account the fact that during processing an additional oxygen pickup cannot be completely avoided.

- As binder a mix of paraffin wax and a polyethylene derivate is used. The materials and their percentage are chosen such that at sintering time a minimum of carbon and oxygen is contributed to the furnace atmosphere. Paraffin wax is extracted by solution in heptane or hexane and the residual binder decomposes by thermal debinding prior to sintering.
- Sintering is done under high vacuum atmosphere in a cold wall furnace with molybdenum shielding and tungsten heater. During sintering a vacuum of 10^{-5} mbar to 10^{-4} mbar is typically provided.

Experimental

Results presented in this paper were gained on samples made from gas atomised spherical Ti-6Al-4V ELI (ASTM grade 23) alloy powder supplied by TLS Technik GmbH, Germany. The alloy powder had a maximum particle size of 45 μm and showed impurity levels of 1000 μg/g in O, 100 μg/g in N and 200 μg/g in C. For the blending with boron amorphous powder (grade I, 95% purity, particle size < 2 μm) supplied by H.C. Starck was used. The binder system used in this study consisted of paraffin wax, polyethylene vinyl acetate (EVA) and stearic acid. The feedstock contained 35 vol% of binder. Samples with and without 0.5 wt% of boron addition were prepared. In the latter case simple powder blending prior to mixing with the binder was performed. The feedstock was prepared using a Z-blade mixer at a temperature of 120 °C under argon atmosphere for two hours. After granulation of the feedstock, cuboids (44 mm x 5.5 mm x 3.0 mm (sintered)) for 4-point bending fatigue tests and tensile specimens (standard “dog bone”, length 80 mm and diameter 4.5 mm (sintered)) were injection moulded on an Arburg 320S machine.

The debinding process was carried out in two steps: chemical and thermal debinding. During the chemical debinding, the samples were immersed in heptane at 40 °C for 20 hours in order to remove one component of the binder system, in this case the wax content. Thermal debinding of the remaining binders and subsequent sintering were performed in a single-step heat treatment. Maximum sintering temperatures from 1250 °C to 1400 °C were applied. The sintering was carried out in vacuum of 10^{-5} mbar with a holding time of two hours at maximum sinter temperature, followed by controlled furnace cooling (10 K/min). Some samples were exposed to a further HIP process carried out at Bodycote HIP N.V. with a maximum temperature of 915 °C for two hours at 1000 bar.

Fatigue samples were shot peened using fine zirconium oxide particles with a diameter of 500 μm. Four-point bending high cycle fatigue 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. The assumed fatigue endurance limit was 10^7 cycles.

Results

Table 1 summarises the tensile test results in dependence on the parameters sintering temperature, HIP and boron addition. Bold numbers reveal the exceeding of the minimum requirement from the standard ASTM B348-02 grade 23 for this property. The corresponding standard values are shown in the lowest row. It is obvious that none of the samples fulfil the demands with regard to interstitial content. On the other hand the oxygen equivalent of all samples keeps below the limit required for grade 5 of 3000 μg/g. This shows that the applied MIM route ensures a sufficient low interstitial level with regard to application. In addition, this is revealed by the fact that all samples show a high ductility above 10 %. All mechanical requirements for standard grade 23 are fulfilled by the HIPped samples and the alloy with boron addition. It is obvious, too, that the grain size depends strongly on the sintering temperature. Thus, care has to be taken concerning this parameter. Boron effects a drastic reduction in grain size as visible in table 1. It is caused by the forming of TiB particles during heating to sintering temperature. These particles act as obstacles for grain growth by pinning of the grain boundaries [8-9]. In addition, they work as additional nucleation sites for α-phase during cooling when the β-transus is crossed.

Table 1: Tensile properties determined on MIM processed Ti-6Al-4V samples.

Parameters	Porosity [%]	$\sigma_{0.2}$ [MPa]	UTS [MPa]	ε_f [%]	Grain size [µm]	O_{eq} [µg/g]
1250 °C	5.7	680	784	**10.8**	93	2657
1350 °C	3.6	720	824	**13.4**	148	2867
1400 °C	3.3	744	**852**	**14.7**	247	2500
1350 °C + HIP	0.0	**841**	**937**	**17.1**	174	2902
1400 °C, 0.5B	2.3	**787**	**902**	**11.8**	18	2483
ASTM B348-02, grade 23	*n/a*	*> 760*	*> 825*	*> 10*	*n/a*	*< 2300*
ASTM B348-02, grade 5	*n/a*	*> 828*	*> 895*	*> 10*	*n/a*	*< 3400*

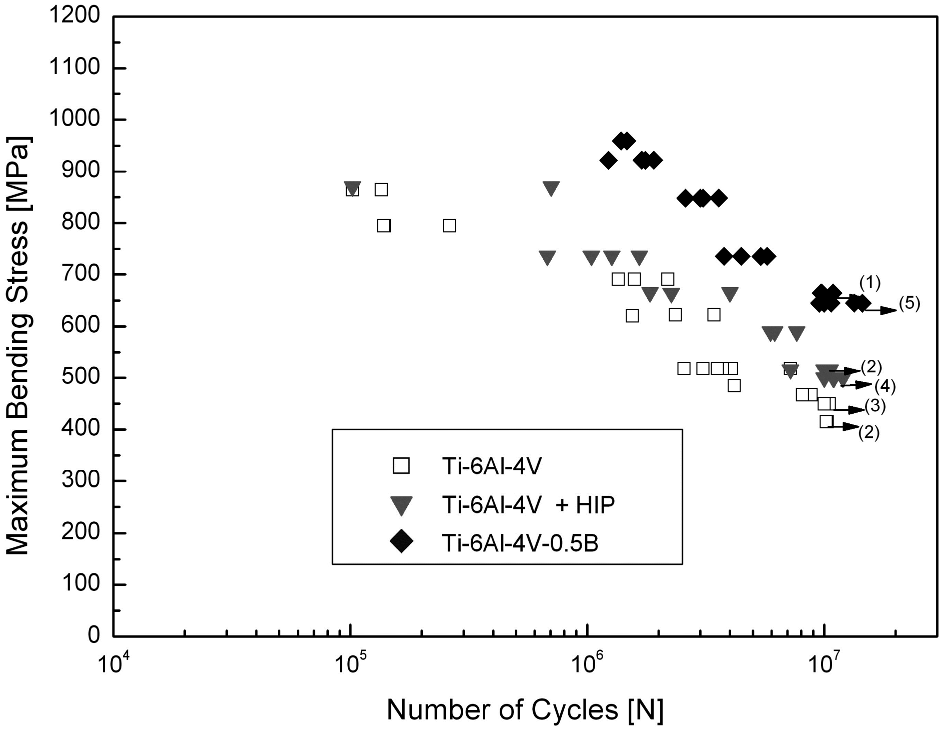

Fig 2: Results of 4-point bending test fatigue experiments.

The fatigue experiments reveal the effects of porosity and grain size. In Fig 2 the Wöhler diagram of three samples is shown [10-11]. The numbers in parentheses denote the amount of samples that survived at a specific stress level (run out). The white squares refer to samples sintered at 1350 °C, the gray triangles to samples sintered at the same temperature, but exposed to a subsequent HIP treatment. According to table 1 the gain with regard to YS by closing the porosity amounts to 121 MPa, while the endurance limit increases from 450 to 500 MPa just by 50 MPa. On the other hand, the finer grain size of the boron added samples effect an increase by 190 MPa. An endurance limit of 640 MPa is equivalent to that of good wrought material.

Summary

By application of a MIM route adapted to the requirements of titanium excellent tensile properties of the sintered components made from Ti-6Al-4V alloy powder can be achieved, even fulfilling the demands from standards for wrought material. The processing parameters, especially the maximum sintering temperature, influence strongly microstructural parameters as grain size and residual porosity and have to be considered carefully. However, if the parts are exposed to fatigue load a small change in composition should be taken into consideration. Grain refinement is more important than porosity for the value of the endurance limit. MIM processed Ti-6Al-4V-0.5B would be a good candidate for a material with high fatigue resistance because the TiB particles effect a drastic reduction in grain size.

References

[1] R. German, Powder Injection Moulding International, Vol.3 (2009) 4, 21-37

[2] B. Oger, T. Ebel, W. Limberg, Proceedings EUROPM 2006, Vol 2., 191-196, EPMA, Shrewsbury (2006)

[3] Y. Itoh, T. Harikou, K. Sato, H. Miura, Proceedings PM World Conf. 2004, Vol. 4, EPMA, Shrewsbury (2004)

[4] R. Zhang, J. Kruszewski, J. Lo, Powder Injection Moulding International, Vol.2 (2008) 2, 74-78

[5] E. Nyberg, M. Miller, K. Simmons, K. S. Weil, Mat. Sc. Eng. C 25 (2005) 336– 342

[6] S. Guo, X. U, J. Xiang, R. Ang, X. He, M. Li, S. Duo, W. Li, Trans. Nonferrous Met. SOC. China 16 (2006) 701-704

[7] D. Hill et al., *Scripta Mater.,* 52, (2005), 387-392.

[8] S. Tamirisakandala et al., Scripta *Mater.,* 53, (2005), 1421-1426.

[9] T. Ebel, Powder Injection Moulding International, Vol.2 (2008) 2, 21-30

[10] O.M. Ferri, T. Ebel, R. Bormann, Mat. Sc. Eng. A 504 (2009) 107–113

[11] O.M. Ferri, T. Ebel, R. Bormann, Mat. Sc. Eng. A 527 (2010) 1800–1805