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Kainer, K.U.; Ebel, T.; Ferri, O.M.; Limberg, W.; Pyczak, F.;
Schimansky, F.-P.; Wolff, M.:

**From Titanium to Magnesium - Processing by Advanced Metal
Injection Moulding**

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From Titanium to Magnesium – Processing by Advanced Metal Injection Moulding

Karl-Ulrich Kainer
Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht
Max-Planck-Straße 1
21502 Geesthacht, Germany
karl-ulrich.kainer@hzg.de
+49 4152 87 2542

Thomas Ebel
Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht
Max-Planck-Straße 1
21502 Geesthacht, Germany
thomas.ebel@hzg.de
+49 4152 87 1944

Orley Milagres Ferri
Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht
Max-Planck-Straße 1
21502 Geesthacht, Germany
orley_mf@yahoo.com.br
+49 4152 87 1917

Wolfgang Limberg
Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht
Max-Planck-Straße 1
21502 Geesthacht, Germany
wolfgang.limberg@hzg.de
+49 4152 87 1915

Florian Pyczak
Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht
Max-Planck-Straße 1
21502 Geesthacht, Germany
florian.pyczak@hzg.de
+49 4152 87 2545

Frank-Peter Schimansky
Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht
Max-Planck-Straße 1
21502 Geesthacht, Germany
frank-peter.schimansky@hzg.de
+49 4152 87 2513

Martin Wolff
Magnesium Innovation Centre, Helmholtz-Zentrum Geesthacht
Max-Planck-Straße 1
21502 Geesthacht, Germany
martin.wolff@hzg.de
+49 4152 87 1916

Abstract

Metal Injection Moulding (MIM) is a good candidate for economic manufacturing of complex shaped components in high quantities. This is especially true for materials which are rather expensive and hard to form like titanium alloys. However, the high affinity to interstitial elements as oxygen and carbon presents a specific challenge with regard to powder purity, handling and sintering as well as to binder system and its removal. In this paper three examples for manufacturing high quality samples of advanced materials are shown in detail. These comprise an optimisation of the well known Ti-6Al-4V alloy with regard to MIM processing and fatigue resistance by adding 0.5 wt% boron powder in order to effect a reduction in grain size. Secondly, MIM processing of intermetallic Ti-45Al-5Nb-0.2B-0.2C (at %) intended for application in turbine engines and turbochargers. And thirdly, the status of MIM of magnesium alloys is presented. In this case fabrication of biodegradable implants with adjustable porosity is the main motivation for the application of MIM.

Keywords

Metal Injection Moulding, titanium alloys, titanium-aluminides, fatigue, magnesium, tensile properties

1 Introduction

Metal injection moulding (MIM) is an established technology for the fabrication of components with complex geometry. The injection moulding process offers extraordinary possibilities to design fine structural details, thin walls and holes with nearly any desired shape. For larger quantities the economical advantage can be dramatic compared to casting or machining. To date, more than 20 years experience is available in terms of MIM processing of stainless steel or CoCr-alloy powders and manufacturers all over the world offer this technique commercially. By contrast, titanium and titanium alloy powders are still a speciality in the field of MIM processing.^{1,2} Though, especially in this case using MIM has several advantages. Machining and casting of titanium materials are difficult and time and cost consuming. In addition, the raw material is expensive, thus, resource efficient powder metallurgical methods can be beneficial. Furthermore, by MIM porous components can be produced, too, e.g. for medical applications to enhance bone fixation or enabling drug delivery.

Despite these advantages MIM of titanium and other oxygen-sensitive materials like magnesium or aluminium is definitively not standard yet. The sensitivity for oxygen and carbon requires adapted facilities and processes and the composition of the binder system is crucial. The availability of suitable powders and feedstock is not satisfactory to date. However, MIM of titanium and its alloys has been developed very successfully during the last decade³⁻⁸ and excellent properties can be achieved, if processing is performed adequately. In addition, since spring 2011 ASTM standard F2885-11 for MIM processed Ti-6Al-4V intended for surgical implants is established, revealing the industrial interest in this technique. In this paper two examples for successful MIM processing of titanium based materials are presented, applied to the well known Ti-6Al-4V alloy and the intermetallic titanium-aluminide Ti-45Al-5Nb-0.2B-0.2C (at%). Especially, the optimisation of the fatigue resistance of the Ti-6Al-4V alloy by adding boron powder is shown.

Furthermore, sintering of magnesium powder⁹ and the status of a MIM binder development suitable for magnesium is presented. Recently, magnesium alloys are also highlighted as appropriate biodegradable material for future orthopaedic applications,¹⁰⁻¹⁴ because Mg-alloys provide elastic moduli and strengths matching those of bone tissue.¹⁵ Corrosion products of magnesium, generated during biodegradation, support osteoconductivity.¹⁶ Porous structures of novel biodegradable materials would support the ingrowths of bone cells into the degrading implant (osseointegration).¹⁷ Via powder metallurgical processing route (PM-route) the generation of such parts with open porous, as well as nearly dense structures is possible.^{18,19} In this paper sintering of Mg-0.9Ca powder and the mechanical properties compared to cast material are presented. Furthermore, the status of binder development is introduced, aiming at MIM processing of Mg-based powders in order to manufacture small-sized and sophisticated biodegradable implants of specific design with high reproducibility

2 Experimental

2.1 Sample production

For all specimens produced in the framework of this paper the following MIM processing setup was employed.

Gas-atomized alloy powders with a diameter $< 45 \mu\text{m}$ were used in all cases, produced by the EIGA technique (**E**lectro **I**nduction **M**elting **G**as **A**tomization). The Ti-6Al-4V grade 23 powder was provided by TLS Technik GmbH, Bitterfeld, Germany. Pure Mg powder was delivered by SFM-SA Martigny, Switzerland and the Mg-master alloy powders (MAP) were produced by ZfW, Clausthal-Zellerfeld, Germany. Ti-45Al-5Nb-0.2B-0.2C (at%) was made inhouse from an ingot provided by GfE Gesellschaft für Elektrometallurgie mbH, Nürnberg, Germany. For producing Ti-6Al-4V-0.5B alloy boron powder (grade I, 95 % purity, $< 2 \mu\text{m}$) was supplied by H.C. Starck, Germany, and mixed with Ti-6Al-4V grade 23 powder before feedstock production.

As binder a mix of paraffin wax, stearic acid and polyethylene-vinylacetate copolymer (PEVA) was used. Additional polymer components like polybutadiene or PPco1PB were used for the Mg-feedstock production. Preparation took place inside of a glovebox system under controlled argon atmosphere by means of a z-blade kneader. The powder load amio65 vol% powder was mixed with binder.

Injection moulding was performed on an Arburg 320S injection moulding machine. The samples produced for tensile test measurements were shaped according to ISO 2740 as dog-bone specimens. As samples for 4-point-bending tests cuboids (44 mm x 5.5 mm x 3 mm) were produced. All fatigue samples were shot peened after sintering using zirconia particles with a diameter of 200 μm to ensure comparable surface quality.

Sintering of titanium materials was performed at temperatures between 1350 °C and 1500 °C for 2h under high vacuum in a cold-wall furnace with Mo-shielding and tungsten heater. In the following MIM processed Ti-6Al-4V will be referred to as Ti64 and Ti-6Al-4V-0.5B as Ti64-B, respectively. For investigation of the influence of porosity some samples were hot-isostatic-pressed with 100 MPa at 915 °C after MIM-processing. They are denoted by the suffix “+HIP”.

From the Mg powders, cylindrical compressive test specimens of 8 mm in diameter and 12 mm in length were produced by double sided, axial pressing at a surface pressure of 100 MPa in a manual mode press (Enerpac RC55, USA) Sintering of all Mg-parts took place in a hot wall furnace (XRetort, Xerion, Germany) at 630 °C for 16 to 64 h under argon gas. Part of the specimen was solution heat treated at 515 °C for 8 h, followed by fast cooling in water.

2.2 Characterisation

Tensile tests were performed on a servohydraulic structural test machine equipped with a 100 kN load cell at a strain rate of $1.2 \times 10^{-5} \text{ s}^{-1}$ at room temperature in air. In the case of Ti-45Al-5Nb-0.2B-0.2C [at%] additional tests were performed under air at 700 °C. The high-cycle 4-point-bending fatigue tests were performed on a resonance machine fabricated by RUMUL. The experiments were carried out in air at room temperature under load control with a cyclic frequency of $\sim 95 \text{ Hz}$ (sine wave) at a load ratio $R = \sigma_{\min} / \sigma_{\max}$ of 0.2. The fatigue endurance limit was defined as 10^7 cycles. Compression tests took place on a Schenck Trebel RM100.

Light microscopy and scanning electron microscopy (ZEISS – DSM962) were used to investigate the microstructure. The level of interstitial elements (O, N, C) were determined using a LECO melt extraction system (TC-436AR and CS-444). The residual porosity was calculated from the density of the sintered samples, measured using the Archimedes method. A density of 4.41 g/cm^3 for the dense Ti-6Al-4V material was determined by measuring a MIM+HIP sample. The porosity of the Ti-45Al-5Nb-0.2B-0.2C [at%] samples were

determined by optical pore analysis using Olympus Analysis Pro software. Grain size was determined according to ASTM E112-96 (linear intercept technique). Electron backscatter diffraction (EBSD) was performed on a ZEISS (ULTRATM 55) scanning electron microscope. Spatially resolved EBSD maps were acquired at 15 keV using a step size of 0.2 μm .

3. Results and discussion

3.1 Optimisation of fatigue behaviour of MIM Ti-6Al-4V

The most used and versatile titanium alloy today is Ti-6Al-4V even with regard to MIM processing. It is often used in highly loaded applications, thus, in addition to tensile properties fatigue behaviour usually is an important issue. Table 1 compares the microstructural and mechanical properties of the MIM samples processed in this study. Furthermore, in the table the minimum values specified by the ASTM B 348 standard for wrought material and by the new ASTM F 2885-11 standard for MIM processed Ti-6Al-4V are displayed.

The results reveal that in case of the Ti64 samples the sintering temperature influences the resulting colony size significantly, as expected. On the other hand no deteriorating effect on tensile properties can be observed. In all cases the ductility exceeds clearly the demands from the standards and the strength fulfils the requirements from the F 2885-11 standard and partly even the standards for wrought material. In addition, it is important to note that especially the yield strength depends on the strain rate applied during tensile testing. This has to be taken into account when comparing different studies. The pick-up of oxygen is varying within the typical range for laboratory tests and depends more on the number of sinter parts in the furnace than on temperature. In summary, the tensile properties achieved by MIM are excellent and more than sufficient for most applications.

In terms of fatigue behaviour the endurance limit of 450 MPa for as-sintered Ti64 appears to be high compared e.g. to typical values for stainless steel. On the other hand, for wrought Ti-6Al-4V a typical range between 500 and 800 MPa exists for the endurance limit. Closing the pores by HIP effects an increase of just 10% although the yield strength rises by 17%, indicating, that the porosity is not the main reason for the inferior fatigue resistance compared to wrought material. As well known fatigue properties of Ti64 are determined mainly by the grain size.²⁰ Unlike to the typical globular microstructure of wrought material, Ti64 reveals α -colonies with β -lamellae due to sintering above the β -transus. Table 1 and Fig. 1 show the much larger colony size compared to typical 10 μm of wrought material.

Refinement of the microstructure of Ti-6Al-4V without thermomechanical treatment is only possible by changing the alloy composition; therefore, Ti64-B samples were prepared. During the sinter process the elemental boron powder and titanium atoms react and form TiB particles. By pinning the grain boundaries during sintering these particles impede the growth of the β -grains. Furthermore, during cooling α -phase nucleates at the particles leading to more and finer α -grains. As shown in Table 1 the grain size is reduced to below 20 μm although the samples were sintered at 1400 °C. This strong difference is also visible in Fig. 1, showing EBSD images of the alpha phase for samples with and without boron addition, both sintered at 1400 °C. Interestingly, the residual porosity is reduced to 2.3% by the addition of boron, probably by the pinning of the grain boundaries. By this mechanism the pores remain in the region of the grain boundaries where diffusion is much faster than in the volume. The lower porosity causes an increase in yield strength of about 9% compared to Ti64 sintered at 1350 °C, while the ductility is reduced slightly, which is probably due to the titanium boride

particles. On the other hand, the effect on fatigue resistance is dramatic: the endurance limit is increased by 42% to 640 MPa. This value is well within the range of wrought material, although considerable residual porosity still exists.

The results show that Ti64-B could be a novel alloy specialised for fatigue load. First corrosion and biological tests proved the good compatibility even for medical applications.²¹ The biological tests show an accelerated settlement of cells and excellent proliferation. Furthermore, boron reveals no toxic effect within the low concentration used in this alloy.

3.2 MIM of titanium aluminides

Titanium aluminides consist of the two intermetallic phases α_2 and γ . The high oxidation resistance, high creep resistance and strength at temperatures up to 800 °C makes them suitable to replace heavy nickel superalloys in turbine engines or turbochargers. However, due to the hardness, brittleness and high temperature strength conventional forming is very difficult and expensive. In addition, the resulting microstructure depends strongly on details of temperatures, holding times as well as heating and cooling rates during sintering and cooling. In Fig. 2 the complicated binary phase diagram Ti-Al is shown,²² which reveals the necessity for keeping precise process control during all thermal processes. Powder metallurgy has to face this challenge, too, but on the other hand shaping is no problem and the alloy composition can be optimised without taken into account formability aspects. Thus, MIM is very interesting for processing this alloy class.

For MIM processing high temperatures close to the solidus are needed in order to gain sufficient sintering due to the low diffusivity of high temperature materials.²³ High sintering temperature implies increased risk for loss of light elements like Al and for oxygen uptake. In this paper the results for Ti-45Al-5Nb-0.2B-0.2C (at%) processed by MIM are presented. For sintering a temperature of 1500 °C close to the solidus proved to be adequate for achieving high density: the residual porosity amounts to values between 0.2 and 0.5% and the remaining pores are small and well rounded (Fig. 3). The reason for the low porosity is not clear yet. It cannot be totally excluded that a small amount of liquid phase is already created during sintering which improves the densification. On the other hand no distortion or melted surface is visible on the sintered samples. Fig. 3 reveals the typical microstructure after sintering for 2h and furnace cooling and displays a lamellar structure consisting of alternating layers of α_2 and γ phase. The average colony size is around 80 μm while the oxygen content was limited to 0.12%. Both values are well comparable to those of typical cast material. The white needle shaped features represent titanium borides.

In Fig. 4 the results of tensile tests on Ti-45Al-5Nb-0.2B-0.2C (at%) samples sintered for 2h at 1500 °C under vacuum are displayed. At 700 °C the strength is not reduced while plastic elongation increases from 0.2 to 1.0 %. This is commonly regarded as sufficient for application. Nevertheless, the yield point is significantly reduced at 700 °C. In Fig. 5 these results are compared with those of cast material. Although the microstructure and oxygen content of cast and MIM samples are almost identical, the cast material shows better strength. It is possible that the difference is caused by different specimen conditions. The cast samples were HIPped, machined to standard geometry and polished, while the MIM processed specimens were of dog bone geometry and just sintered and ground with fine abrasive paper. Further investigations have to be performed on this matter. However, these first results are very promising and further improvement is likely.

3.3 MIM of magnesium

3.3.1 Sintering

A SEM image of a sintered sample made from Mg-0.9Ca powder is shown on the left of Fig. 6. The residual porosity amounts to 3%. In the middle a magnification of the marked region is shown. The arrows point at areas of the brittle phase Mg_2Ca . After solution treatment, the porosity remains the same, but the amount of Mg_2Ca decreases significantly (right image). EDX analyses show higher oxygen content in the Mg_2Ca regions due to the fact that their location seems to be identical with the former oxidized surface of the original powder particles. Table 2 reveals the results of compression tests performed on the as-sintered, the sintered and heat treated (HT) and on casted and heat-treated samples.

The results show that sintering of Mg-alloys to components with properties superior to those of cast material is possible.

3.3.2 Binder development for MIM of magnesium

Because of the high vacuum pressure of Mg thermal debinding and sintering have to be performed under ambient pressure in argon. Therefore a binder component which evaporates completely under this condition has to be found. As shown in Fig. 7 PPco1BP appears to be a good candidate compared to PEVA used for titanium. The left side reveals the microstructure of samples containing PEVA and PPco1BP, respectively, after sintering while on the right reference samples without binder are shown, which were placed next to the specimen during the process. Without polymer containing neighbours these reference samples show a residual porosity of 15% after sintering at 630 °C for 8 h.

As shown in Fig. 7 the use of PEVA as component for the MIM of Mg-0.9Ca results in a residual porosity of 27.5 % exceeding the green porosity. Also the reference specimen shows an increased residual porosity of 18.7 %. Carbon residuals in the pores of the PEVA containing sample observed by line profile analysis (LPA) (see yellow square), may be a major obstacle for the sintering process.²⁴ Increased carbon content could not be observed on the other specimens. Furthermore, Mg may reduce the molecular chain of the acetate group in the PEVA molecular chain and react with the containing oxygen before the thermal decomposition of the molecular chain is done. In contrast, the PPco1BP containing samples and their references sinter successfully to 13.6 % porosity and 12.6 %, respectively. A recent study shows that the additional use of paraffin wax and stearic acid does not influence the sintering result.

4. Summary

The results show that Metal Injection Moulding of oxygen sensitive materials is possible, if their specific properties and demands are adequately taken into account. Especially binder composition and sintering equipment and processing are crucial. MIM of commonly used titanium alloys like Ti-6Al-4V as well as of titanium aluminides leads to sound properties, which let MIM appear to be an alternative manufacturing method for small and complex shaped parts made from these materials.

Tensile properties of MIM processed Ti-6Al-4V alloy are comparable to standard values for wrought material and especially high ductility can be achieved if interstitial content is limited. Furthermore, applying an additional hot isostatic pressing process which closes the residual

porosity leads to excellent properties exceeding all standard limits by far. In the case of titanium aluminides nearly dense material can be achieved just by sintering and the tensile properties are close to those of cast material.

For fatigue loaded applications a fine microstructure is decisive and an example of grain refinement by adding boron powder was given. The modified alloy Ti-6Al-4V-0.5B shows a fatigue limit of 640 MPa, well within the range of wrought material although 2.3% porosity still exists. This shows the potential of powder metallurgy for optimising mechanical properties to the needs of the application with low effort by alloy composition tailoring.

The sintering results of Mg-feedstock point out the general suitability of Mg-alloys for MIM. Using of polypropylene-co-1-butene (PPco1PB) as binder component results in successful sintering of the Mg-0.9Ca powder material.

5. References

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a) MIM Ti64, sintered at 1400 °C / 2h

b) MIM Ti64-B, sintered at 1400 °C / 2h

Fig. 1. EBSD images of α -phase visualising the effect of boron addition on grain size

Fig. 2. Binary phase diagram of Ti-Al.²² The circle marks the region where sintering takes place.

Fig. 3. Typical SEM micrograph of Ti-45Al-5Nb-0.2B-0.2C (at%) processed by MIM.

Fig. 4. Tensile test results performed at room temperature (RT) and at 700 °C: Ti-45Al-5Nb-0.2B-0.2C (at%) alloy processed by MIM, sintered under vacuum.

Fig. 5. Comparison of tensile test results between MIM processed and cast Ti-45Al-5Nb-0.2B-0.2C (at%) alloy, performed at room temperature (RT) and at 700 °C.

Fig. 6. SEM-micrographs of sintered Mg-0.9Ca powder: as-sintered (left and middle) and after solution heat-treatment (right). White arrows pointing at Mg₂Ca precipitates.

Fig. 7. Microstructure of PEVA and PPco1BP containing Mg-specimen and there binder free reference specimen after sintering (630 °C / 8 h)

Table 1. Microstructural and mechanical properties of the MIM processed samples based on Ti-6Al-4V in comparison to ASTM standards.

	Ti64		Ti64+HIP	Ti64-B	ASTM B 348		ASTM F 2885-11	
	1350 °C	1400 °C	1350 °C	1400 °C	Grade 23	Grade 5	as sintered	+ HIP
Residual porosity	3.6%	3.3%	0.0%	2.3%	-	-	-	-
Colony size [µm]	148	247	174	18	-	-	-	-
Oxygen content [wt%]	0.23	0.19	0.23	0.20	< 0.13	< 0.20	< 0.20	< 0.20
Yield strength [MPa]	720	744	841	787	> 760	> 828	> 680	> 830
Ultimate tensile strength [MPa]	824	852	937	902	> 825	> 895	> 780	> 900
Elongation to fracture [%]	14	15	17	12	> 10	> 10	> 10	> 10
Endurance limit [MPa]	450	-	500	640	-	-	-	-

Table 2: Results of compression tests on differently processed Mg-0.9Ca samples

	Compressive yield strength [MPa]	Ultimate compressive strength [MPa]	Compressibility [%]
As sintered	81	247	18
Sintered + HT	90	328	22
Cast + HT	45	290	25