# Advances in the Metal Injection Moulding of titanium at Euro PM2014

The Euro PM2014 conference, which took place in Salzburg, Austria, September 21-24, presented an opportunity for the industry to review progress to-date on global developments relating to the Metal Injection Moulding of titanium. Dr Thomas Ebel, from Helmholtz-Zentrum Geesthacht (HZG), Germany, reports on a selection of key presentations from the event, including those with a focus on biomedical and dental applications, Metal Matrix Composites and complex 3D structures.

The Powder Metallurgy (PM) of titanium-based materials is of steadily increasing interest to both industry and the research community. The growing demand for titanium components in the aerospace and biomedical sectors has given rise to enhanced efforts not only to reduce the costs of raw materials and processing, but also to improve mechanical and structural properties and extend the technological capabilities for realising complex and fine structures. As a result, presentations on the PM of titanium are a common element of many materials conferences.

The Euro PM2014 conference in Salzburg was no different. However, unlike Euro PM2012 in Basel, Switzerland, no special interest seminar on MIM of titanium or a dedicated PM titanium session were arranged. A variety of titanium PM related presentations were spread across different sessions and the combination of PM and titanium was frequently encountered in the exhibition hall, where producers of powders and processing facilities offered their products.

#### **Biomedical applications**

Biomedical applications are of ongoing interest in the Metal Injection Moulding (MIM) sector. Two main titanium related topics were handled at the Euro PM2014 conference, namely porous materials and beta-titanium alloys based on the binary system Ti-Nb.

In the field of the Powder Metallurgy based manufacturing of porous materials there is always the challenge of retaining the geometrical shape of highly porous components during sintering. Daudt *et al* [1] presented an interesting work on the effect of plasma treatment before final sintering. The study was aiming at the production of stable titanium components by MIM using 70 vol% space holders, which commonly leads to deformed parts. In the first part of their work they determined the optimal ratio between binder, space holders and metal powder. They used gas atomised Ti powder with a diameter smaller than 45  $\mu$ m and rounded KCl space holders (355-500  $\mu$ m). As binder a combination of paraffin wax, polyethylene and stearic



Fig. 1 Porous Ti specimens with 72% solid load, without (left) and with (right) plasma treatment prior to sintering [1]



Fig. 2 Cross sections through pressed and sintered porous Ti material, using a steel punch (left) and an elastic cover of the punch (right) [2]

acid was applied. After optimisation of the homogenisation process for the feedstock a systematic study of different solid loads (72%, 75%, 80%) led to an optimal value of 80%.

The trend was that higher solid load led to increased stability. In the second part of the work the effect of plasma treatment was studied. Daudt et al compared the geometrical deformation of conventionally processed specimens with those exposed to a plasma treatment before sintering. Interestingly, they found a strong effect on the geometrical stability and also on open surface porosity. Two different parameter sets were applied for the plasma treatment and, with the parameters 75 Pa Argon, 15 min, 294 W, a significantly increased stability was observed.

Fig. 1 shows a comparison between specimens produced with

72% solid load with and without plasma treatment. The effect was analysed as being related to an increased crystallinity of the polyethylene and breakdown of polymeric chains due to the plasma treatment, but an induced initial sintering of the titanium particles at the surface is also assumed. Thus, a rather stable mantle during sintering seems to be provided by the plasma treatment, preventing the highly fragile material from collapsing.

Retaining the shape and open porosity at the surface of pressed and sintered porous titanium was also the aim of Savich *et al* [2]. The authors aimed at the production of porous cervical and thoracic vertebrae implants. However, they observed undesired partial closing of the surface pores during uniaxial pressing by the steel punch and tried to avoid this by using an elastic material as the interface between punch and powder. For powder they used spongy titanium and for the elastic interface a disc made from polyurethane. The effect on the surface can be seen in Fig. 2. Furthermore, the usage of the elastic material increased the overall porosity and the average pore size in a desired manner.

Savich et al also presented a study on the usage of a mixture of spherical and spongy powder for the production of permeable membrane holders, filters, aerators etc [3]. They observed that using only spongy powders leads to a high porosity due to the intrinsic porosity of the sponge but to a low permeability in terms of gas flow. However, the permeability significantly increases by adding spherical powder. The effect is based on the geometrical stability of the spherical powder particles during pressing, providing a fraction of larger pores. An optimum appears to be at 70 to 80% spherical powder.

There was also a poster shown with first results on the production of foams made from Ti-30Nb-13Ta-xMn (x= 2, 4, 6). Guerra *et al* [4]. The idea was to provide very low Young's modulus of the material similar to that of bone. In particular, in the study the effect of Mn on the mechanical properties was investigated, showing an increase of the elastic modulus with increasing Mn content. Suitable material was produced by mechanical alloying of elemental powders.



Fig. 3 SEM images of the Ti-17Nb microstructure without (left) and with (right) quenching. The quenched sample was etched after polishing in order to reveal the martensitic structure [5]

#### Beta-titanium alloys based on Ti-Nb

Adapting the elastic properties of an implant material to those of cortical bone is one of the motivations for research in the field of beta-titanium alloys based on Ti-Nb. Another is to avoid any potentially critical elements such as Al as alloying additions.

Bidaux et al [5] continued their previous work on Ti-17Nb processed by MIM and presented an interesting way to provide particularly low Young's modulus of the material. They tried to introduce the martensitic phase  $\alpha$ ", which shows a very low elastic modulus, by rapid quenching. The authors utilised gas atomised Ti powder  $(Dv_{50}= 14 \ \mu m)$  and angular Nb powder  $(Dv_{50}= 36 \ \mu m)$ . They mixed these powders with paraffin wax, low density polyethylene and stearic acid as binder and processed the feedstock by MIM. After sintering at 1400°C for 4h the porosity amounted to 4.5%. The guenching took place in water after an additional heat treatment at 900°C for 1 h. The microstructural analysis showed no effect on porosity and carbides are visible as observed in other earlier studies on MIM processing of Ti-Nb alloys. However, a clearly different picture can be observed in the SEM as shown in Fig. 3.

The finely distributed phase in the quenched samples was identified by XRD as the desired  $\alpha$ " phase. Fig. 4 shows the effect on tensile properties. While the ultimate tensile strength is not strongly affected, Young's modulus decreases from 76 GPa to 45 GPa after heat treatment. The decrease in the elongation may, on the one hand, be due to the properties of the  $\alpha$ " phase but also on some surface oxidation during quenching. Here, room for improvement appears to exist.

Amigó et al [6] also worked on Ti-Nb. They added Fe and Cr to the alloy in order to investigate their effect on beta-phase fraction and mechanical properties. Specifically, they tried to overcome the problem of inhomogeneous microstructures when blending and sintering elemental powder due to limited solid state diffusion. For this they compared material prepared by mechanical alloying with the blended elemental approach. They varied the addition of Fe and Cr in order to yield different Mo equivalents from 8 to 12%. However, they succeeded principally in fabricating the alloys by mechanical alloying, but porosity was higher and mechanical properties inferior compared to the samples prepared via blended elemental approach. This result is probably caused by larger particles after milling and increased hardness and less deformability. Further studies are planned.

#### Ti-6Al-4V and Ti/ZrO<sub>2</sub> composites

The powder metallurgical processing of titanium alloys is not only interesting for bone implants but also in the dental sector. A dental implant consists of two parts with rather different requirements in terms of mechanical, optical and biological properties. The part which is implanted into the bone should show excellent osseointegration and biocompatibility plus high mechanical toughness. Requirements for the abutment Adva Power Coa

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with bone for a dental implant for example or in contact with soft tissue such as gingiva. Fig. 5 shows the results for the cell migration for (a) the composite material compared to the reference Thermanox<sup>®</sup>, which is a tissue cell culture plastic being the control material used in biology, and (b) the PIM Ti-6Al-4V material compared to wrought Ti-6Al-4V and again Thermanox<sup>®</sup>.

application, meaning either in contact

Fig. 4 Tensile tests of as-sintered and water-quenched Ti-17Nb specimens [5]



Fig. 5 Cell migration on  $Ti/ZrO_2$  (a) in contact with epithelial tissue and PIM Ti-6Al-4V (b) in contact with bone tissue in comparison with reference materials [7]

on the other hand are hardness and good aesthetics. Furthermore, the growth of bacteria should be suppressed, while good adhesion of epithelial tissue is desired. Thus, two different materials are required and former studies showed that Ti-6Al-4V is suitable for the implant section, while for the abutment a composite made from titanium and zirconia appears to be adequate. Because size and geometry of dental implants are generally standardised, Powder Injection Moulding (PIM) appears to be a promising technique for both parts. Additionally, two component (2C) Powder Injection Moulding could be applied, avoiding any additional joining process.

Auzene *et al* [7] reported on the biocompatibility of materials produced by PIM from Ti-6Al-4V and Ti/ZrO<sub>2</sub> composite, respectively. They used commercial feedstocks as raw material. For the composite they mixed 90 wt% titanium grade 4 feedstock with 10 wt% zirconia feedstock. Because of different binders the debinding processes for Ti-6Al-4V and Ti/ZrO<sub>2</sub> were different, but the sintering temperatures were quite similar. Ti-6Al-4V was sintered at 1350°C for 1 h, while the composite was sintered at 1300°C for 3 h. In view of 2C-PIM all these parameters and binders have to be adapted appropriately, but this should not in principle be a problem. Cytotoxicity experiments using organotypic cultures were conducted on the sintered materials and compared with other reference materials with wrought surgical Ti-6Al-4V (ASTM F 136 standard).

The trend for the PIM Ti-6Al-4V material was to show in contact with chick embryo bone increased cell migration and adhesion compared to the references. In the case of the composite in contact with chick embryo skin, interestingly, cell growth was significantly reduced, while a good adhesion of epithelial tissue was observed. Thus, both materials appear to be appropriate for their intended Metal Matrix Composites

Besides biomedical applications, there were numerous papers on the addition of particles to standard titanium materials, some of them aiming directly at Metal Matrix Composites (MMCs). One interesting way to form MMCs was presented by Biasetto et al [8]. In this study preceramic polymers were used for the in-situ forming of silicides and carbides in order to improve hardness and wear resistance of Ti-6Al-4V. For metal powders, equal portions of two size fractions of gas atomised Ti-6Al-4V powder were mixed (-250  $\mu$ m and -45  $\mu$ m). Then poly(methylsilsesquioxane) was added which decomposes at around 350°C. Under Ar or N atmosphere it transforms to  $SiO_{x}C_{y}$  ceramic phases. The polymer was crosslinked by a catalyst and added (9 vol%) to the metal powder together with 1 vol% PEG. Then the mixture was milled in a planetary ball-mill with the milling parameters as one subject of the investigation.

The milled material was shaped into discs by uniaxial pressing and sintered at 1400°C for 2 hours under argon. XRD-measurements confirmed the impression of SEM and EDS investigations that well distributed silicides and carbides were formed during the process. EBSD measurements identified the carbides as TiC and the silicides as  $Ti_5Si_3$ . Fig. 6 shows the effect of the particles on hardness and wear resistance. The hardness increased by about 12% while the wear resistance improved dramatically.

Another method for providing a homogeneous distribution of ceramic

particles was presented by Neves et al [9]. The aim was to restrict grain growth during sintering by introduction of hard and small particles in the titanium matrix. Simply blending the powders commonly results in a poor distribution of the fine particles, resulting in an inhomogeneous microstructure. Instead, the authors used a colloidal technique to distribute the ceramic particles on the surface of the metal powder particles before pressing and sintering. For the study spherical titanium grade 1 with a mean diameter of 10 µm was used. As ceramic addition the authors chose fine alumina and zirconia powders. the latter one in two variations. namely monoclinic (m-ZrO<sub>2</sub>) and tetragonal yttria-stabilized (t-ZrO<sub>2</sub>). The Dv<sub>50</sub> value of the powders was between 300 and 400 nm. Suspensions were made with KNO<sub>3</sub> addition and pH-adjusted by using tetra methyl ammonium hydroxide (TMAH) and HNO<sub>3</sub> solutions. From each ceramic powder water based slurries were fabricated by mixing 1 wt% of the ceramics with 50 vol% Ti powder, where ammonium polyacrylate (PAA) was used as dispersant. High power ultrasound was applied to the slurries to avoid agglomerations. As binder, polyvinyl alcohol (PVA) was used. After spray-drying, cylinders were produced by uniaxial pressing and subsequently sintered at 1100°C for 30 min under vacuum. Fig. 7 shows the Ti-Al<sub>2</sub>O<sub>3</sub> granules after spray drying and the dispersion of the ceramic particles on the surface of the Ti spheres.



Fig. 6 Results for hardness and wear resistance for pure Ti-6Al-4V and particleadded material [8]

For all variants including pure Ti granules measurements on the dependence of the relative green density on the compacting pressure were performed and the results showed excellent compressibility even for the ceramics-including granules, especially the variant with  $t-ZrO_2$ . After sintering the total porosities were 8.5% (Ti-m-ZrO<sub>2</sub>), 7.3% (Ti-t-ZrO<sub>2</sub>), 3.7% (Ti-Al<sub>2</sub>O<sub>3</sub>) and 4.8% (pure Ti). The microstructures were very homogeneous. Interestingly, the investigation of existing particles in the Ti-matrix after sintering revealed significantly different behavior of the different composites. In the case of Ti-m-ZrO<sub>2</sub> corresponding ceramic particles were found mostly at the grain boundaries, while in the case of Ti-t-ZrO<sub>2</sub> Y<sub>2</sub>O<sub>3</sub> particles are formed and found at the grain boundaries, thus, Zr is dissolved in the matrix.

In contrast, the Al<sub>2</sub>O<sub>3</sub>-containing composite behaves differently; the ceramic particles appear to dissolve during sintering, so aluminium and oxygen atoms are distributed in the Ti-matrix and no particles are visible at the grain boundaries. In all cases Vickers hardness of the material increases significantly compared to the pure titanium material (Fig. 8). As reasons for this behavior the authors attribute the total oxygen content, smaller grain size and lower closed porosity.

#### Improving mechanical properties

Reinforcement of Ti-6Al-4V and increase of its Young's modulus in order to provide mechanical properties closer to those of high-strength steels or Ni-based alloys is the



Fig. 7 SEM images of titanium/Al<sub>2</sub>O<sub>3</sub> granules after spray drying [9]



Fig. 8 Vickers hardness as a function of added ceramics and oxygen content [9]



Fig. 9 Production scheme of reinforcement particles [10]

motivation of the work of Agote *et al* [10]. They added TiB and TiC, respectively, to Ti-6Al-4V powder and compacted the mixtures by Spark Plasma Sintering (SPS). The strengthening particles were produced by Self-propagating High-temperature Synthesis (SHS) using highly-loaded master alloys of around 80% of TiB and TiC respectively, mixed with Ti-6Al-4V. The raw materials were fine irregular boron (< 5 µm) and carbon (< 0.5 µm) powders and spherical Ti-6Al-4V powder (< 25 µm). As a result of the process sketched in Fig. 9 an improved interface between reinforcement particles and titanium matrix was gained.

Densification was performed by SPS after mixing the reinforcement with more Ti-6Al-4V powder. Two variants of the final composition were fabricated, namely 10 vol% and 20 vol% TiB and TiC, respectively. The basic processing parameters for SPS were a maximum temperature of 1100°C for 2 min under vacuum of 10<sup>-2</sup> Pa. Typical microstructures of the sintered material are visible in Fig. 10, where a significant grain refinement can be observed (around 20-30 µm) compared to pure Ti-6Al-4V. While the TiB particles are distributed quite uniformly over the matrix, the carbides appear to be mostly at the grain boundaries. For all composites the density was higher than 99% of the theoretical value. The Vickers hardness increased from 324 (pure Ti-6Al-4V) to a range from 433 (10 vol% TiB) to 541 (20 vol% TiC). First mechanical tests reveal a significant increase of the Young's modulus to about 170 GPa for the composites with 20 vol% reinforcement

Fatigue resistance is an essential property for application under dynamic load. PM processed material tends to show inferior behaviour compared to wrought material due to residual porosity and different microstructure, especially with larger grain size. Limberg *et al* [11] investigated the possibility



Fig. 10 Microstructures of the sintered material, left: TiB added, right: TiC added Ti-6Al-4V [10]



Fig. 11 Microstructures of MIM processed Ti-6Al-4V and Ti-6Al-4V + 0.2  $Y_2O_3$  [11]

to restrict grain growth in Ti-6Al-4V during sintering by adding small ceramic particles. They applied uniaxial pressing and MIM for the processing of spherical Ti-6Al-4V powders (< 45 µm) and mixed these powders with 1 wt% of different oxide particles (MgO,  $CeO_2$ ,  $ZrO_2$ ,  $Y_2O_3$  and CaO, sizes between 200 and 600 nm) during feedstock production. As binder a mixture of paraffin wax, polyethylene-vinylacetate and stearic acid was used. The MIM feedstock was also used for the production of uniaxial pressed cylinders. Sintering was performed under vacuum for 2 h at 1350°C. In all cases, other than the variant with yttria addition, the porosity after sintering was around 3 to 3.5%. Adding yttria led to a high porosity of 12.3%. However, a dramatic decrease of the colony size to 25 µm compared with 130 µm of pure Ti-6Al-4V was observed. By a reduction of the yttria content to 0.2% and 0.1%, the porosity was reduced to 6.1% and 3.4% and the colony size amounted to  $30 \ \mu m$  and  $50 \ \mu m$ , respectively. All additions of ceramic particles resulted in a reduction of the colony size, but the strongest effects were observed in the case of yttria and CaO (40 µm). For these compositions tensile test specimens were made by MIM and tested. Fig. 11 shows the microstructures of pure Ti-6Al-4V and Ti-6Al-4V + 0.2  $Y_2O_3$ .

The specimens containing 0.1% and 0.2% yttria showed high plastic elongation of around 15% and strength comparable to pure Ti-6Al-4V, while the CaO containing specimens showed high strength, but brittle behaviour (Fig. 12). The microstructural investigation revealed that the CaO particles decompose during sintering and the atoms are dissolved in the matrix. The oxygen atoms in particular harden, but embrittle, the material as seen in the tensile test. Thus, the best candidate for grain refinement appears to be  $Y_2O_3$ , according to this study.

Piemme *et al* [12] showed that Ti-6Al-4V processed by MIM can show excellent mechanical properties, including fatigue resistance, if all individual processes are carefully and consistently mastered and the fabrication is limited to the processing of titanium alloys. They presented mechanical data and their corresponding



Fig. 12 Tensile tests of MIM processed Ti-6Al-4V plus different oxide particles [11]

reproducibility of commercially fabricated components. Because of confidentiality, no details of feedstock and process were given, but the mechanical data exceeded the ASTM F2885 for MIM fabricated medical components from Ti-6Al-4V by a considerable margin. After MIM processing and subsequent HIP treatment average values for yield strength, ultimate tensile strength and plastic elongation were 860 MPa, 964 MPa and 19.8% respectively. Oxygen content was about 1700 µg/g. The mechanical properties equal practically those commonly known from wrought Ti-6Al-4V. This is even true for the endurance limit of around 620 MPa at 10 million cycles. According to the authors the good fatigue properties are the results of an optimised microstructure.

#### 3D titanium structures

Piemme *et al* also introduced an interesting technique for the production of 3-dimensional complex structures, for example as a tissue integration layer for bone implants. They utilised sacrificial inserts to form structures during the injection moulding process. The inserts can be made by Additive Manufacturing techniques, so the freedom of geometry is very high. After production, they are inserted in the mould and the feedstock is injected around and into

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Fig. 13 3D structures fabricated by MIM and use of a sacrificial insert [12]

the part. After moulding the insert is removed in a similar way to spaceholders in porous materials and the part is sintered. The technique is also suitable for providing special surfaces, for example in terms of roughness. Fig. 13 shows an example of an ingrowth surface (a) along with a close-up (b). A layer with finer porosity and organic appearance is shown in (c).

The realisation of 3D structures by means of Powder Metallurgy techniques was the subject of two more studies presented at Euro PM2014. Jurisch *et al* [13] worked on a special variant of screen printing which is commonly utilised for the production of solar cells or piezoelectric devices. It combines MIM and Additive Manufacturing for the production of miniaturised intricate parts in high numbers. Even inner structures such as closed channels are possible. It is a layer by layer technique and its steps are outlined in Fig. 14.

As with MIM, a powder-binder mixture is used. The authors applied gas atomised Ti-6Al-4V powder with a particle size smaller than 25  $\mu$ m and an organic and water soluble binder. For the production of the part the structured screen is positioned on a substrate and the printing paste is applied, so one layer of the part is produced. After drying the next layer is fabricated by repeating the process with the same screen or by changing the screen a change in the geometry can be performed. After forming, the parts are debound and sintered in the usual manner known from MIM.

The rheology of the paste is extraordinarily important for the success of the process, so the formulation of the printing paste was one of the subjects for investigation in the study presented. Different components such as surfactants and defoaming agents have to guarantee a homogeneous paste. The authors determined a paste with a powder loading of 43 vol% being most suitable. After the sintering of test patterns such as those shown in Fig. 15 at 1250°C for 1 h under vacuum the typical basketweave structure of alpha and beta phases were observed as the microstructure. However, oxygen and carbon contents were rather high, so some studies for improvement will be performed in the future.

The Additive Manufacturing of components from Ti-6Al-4V was the third technique presented for the production of 3D structures. Kirchner et al [14] performed a study on the process window for Electron Beam Melting (EBM). The aim was to optimise process parameters such as scan speed and beam current in relation to build rate, geometrical stability and mechanical properties. For the investigation they used gas atomised Ti-6Al-4V powder in the range of 45 to 105 µm. During the work, the authors recycled the powder up to ten times and observed just a small increase in oxygen. In each process ten parts of 50 mm x 10 mm x 14 mm were fabricated and investigated. In terms of density a decrease with increasing scan speed was determined including a sharp drop at a certain limit. When scan speed was too low in connection with a high beam current swelling of the top surface and enhanced aluminium loss was observed. In contrast, the porosity of 0.25% was nearly independent of the process parameters and appeared to be caused by gas inclusions in the powder particles. The microstructure showed isotropic characteristics with a dependence of the size of alpha plates on the scan speed. Nearly 100% martensitic alpha' phase was produced if the scan speed was high enough. For reasonable parameter sets the ultimate tensile strength was between 970 and 1030 MPa combined with more than 10% elongation. A certain limit was also detected for the beam current; if the value was too small a sudden drop in density occurred. Fig. 16 represents the process window for scan speed and beam current as the result of the study. A good quality can be produced in the parameter field between 100 and 200 J/m.

lain Todd, from The University of Sheffield, UK, gave

a comprehensive presentation on EBM in the programme of the Special Interest Seminar on Progress in Additive Manufacturing. He pointed out that although the mechanical properties of Ti-6Al-4V processed by EBM yield excellent values, there is still no breakthrough in terms of commercial application. He showed that tensile (Fig. 17) and fatigue (Fig. 18) properties are in the range of wrought material if the process is properly performed. For example, the typical forming of microstructural columns during EBM processing can be reversed by subsequent heat treatment such as HIP, with the additional benefit that any pores are closed.

However, concerns exist on the one hand because of defects inherent in the usage of powders and in the process. Examples of defects are porosity or inclusions in the powder, lack of fusion or delamination during deposition, different microstructural orientations in the build, for example connected with turning points or heat sinks and even errors in the STL files. Most of the defects can be controlled to a greater or lesser extent, but not avoided completely. So, on the other hand, there is a lack of understanding related to processing conditions and their effect on materials properties which has to be overcome by more basic scientific studies. As one example Todd showed detailed investigations on fatigue behavior based on x-ray computer tomography.

#### Other Ti-based materials

Besides Ti-6Al-4V there were a few presentations on other titanium materials. The use of titanium hydride was the subject of the work presented by Petroni et al [15]. The motivation for the use of hydride powders is the lower cost and the good compacting properties. The authors performed a hydriding treatment of Ti sponge fines and sieved two fractions, < 355 µm and < 150 µm respectively. The powders were uniaxially cold pressed and the compaction behaviour compared with a theroretical model developed by Gerdemann and Jablonski in order



Fig. 14 Scheme of the 3D screen printing technique [13] ©Fraunhofer IFAM Dresden



Fig. 15 Test structures fabricated by 3D screen printing (green, brown and sinter state) [13] ©Fraunhofer IFAM Dresden



Fig. 16 Process window for EBM processing of Ti-6Al-4V powders [14]

to separate the influences of initial density, sliding and fracture, and elastic and plastic deformation on the final green density. Furthermore, they compared the results with the usage of commercial HDH powder. After determining the right fitting parameters the authors observed a good agreement between model and experimental data. They concluded that the initial density is the most important factor in determining the final density. However, in the case of HDH powder also sliding and fracture

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Fig. 17 Typical tensile properties (UTS) of Ti-6Al-4V processed by different techniques (Data sources: Titanium, A Technical Guide, M. Donachie, Arcam AB and University of Sheffield)





and deformation play a role, rather different to the just hydrided powder. In all cases deformation is more important than sliding and fracture.

The creep properties of MIM processed titanium aluminides were the topic of the last study presented in this report. These variants of titanium alloys intended for high-temperature applications suffer from brittleness and hardness of the intermetallic phases they consist of. Conventional shaping is very difficult, thus PM methods are interesting - including MIM for smaller components. Ebel *et al* [16] assessed the creep properties of MIM processed Ti-45Al-5Nb-0.2B-0-2C (all contents in at%), also known as TNB-V5 alloy. They focussed on the primary creep because this is often the limiting factor for technical applications of this material class. For the feedstock pre-alloyed TNB-V5 powder with a particle size smaller 45 µm was mixed with a binder consisting of paraffin wax, polyethylene-vinylacetate and stearic acid. From this, specimens for compression and tensile creep test as well as for tensile tests were produced. Sintering took place at 1500°C for 2 h and a very low porosity of around 0.5% was yielded. The authors varied the cooling rate from sintering temperature and by this changed mainly the width of the lamellae and the grain size of the microstructure. The effect on creep resistance is quite strong, as shown in Fig. 19. While for tensile strength the fast cooling (100K/min) showed the highest value (560 MPa), for creep resistance slow cooling is beneficial, probably because of the larger grains. Compression creep tests were also performed with arc-melted material representing an as-cast condition. As Fig. 19b reveals, the slow cooled MIM specimens showed even better creep resistance. Thus, MIM of titanium aluminides appears to be competitive with casting, particularly in terms of creep properties.

#### Outlook

As shown in this report, Euro PM2014 featured a wide variety of contributions related to titanium. However PM titanium suffers from a rather small community compared to steel processing. Thus, at PM conferences titanium is usually a relatively small subject whilst at titanium conferences Powder Metallurgy is featured in a rather minor way. Specialised meetings are therefore important as the Ti Powder Metallurgy conference first held in Brisbane, Australia, in December 2011, first demonstrated. Here, PM titanium specialists from the whole world met and the conference was continued successfully in December 2013 in Hamilton, New Zealand. The event will be held again this year, from August 31 to September 3 2015. in Lüneburg, Germany.



Fig. 19 Creep curves of MIM processed Ti-45Al-5Nb-0.2B-0.2C (TNB-V5) with different cooling rates applied. Left: tensile creep, right: compression creep [16]

#### References

The papers reviewed in this report are published in the proceedings of the Euro PM2014 conference, Salzburg, Austria, 21-24 September 2014, and organised by the European Powder Metallurgy Assiciation (EPMA), Shrewsbury, UK.

 N F Daudt, L. Classen, M Bram, H P Buchkremer, Manufacturing of Highly Porous Titanium Samples by MIM with Temporary Space Holders.

[2] V V Savich, A I Shelukhina, G
A Sheko, A M Taraykovich, S A
Bedenko, Formation of Anisotropic
Structure of Porous Vertebral
Implants by Compating Spongy
Titanium Powder through the Elastic
Punch Facing.

[3] V V Savich, A M Taraykovich, S

A Bedenko, Porous Permeable Materials from Titanium Powders with Various Particle Shapes.

[4] C Guerra, C Aguilar, D Guzmán, P A Rojas, S Lascano, L Perez, Production and Characterization of Mechanical Properties of Ti-Nb-Ta-Mn Alloy Foams for Biomedical Applications.

[5] J.-E. Bidaux, R. Pasquier, M. Rodriguez-Arbaizar, H. Girard, E. Carreño-Morelli, Low Elastic Modulus Ti-17Nb Processed by Powder injection Moulding and Post-Sintering Heat Treatment.

[6] A. Amigó, J. Zambrano, S.

Martinez, V. Amigó, Microstructural Characterization of Ti-Nb-(Fe-Cr) Alloys Obtained by Powder Metallurgy.

[7] D. Auzene, T. Jacquemin, J.L. Duval, C. Egles, J.M. Popot, Biological Characterization of Materials Produced by Powder Injection Moulding (PIM) for Dental Applications.

[8] L. Biasetto, F. Bonollo, A. Fabrizi,
 P. Colombo, Development and
 Characterization of Ti6Al4V Matrix
 Composites, Formed In Situ via the
 Preceramic Polymer Route.

[9] R. G. Neves, B. Ferrari, A.J. Sanchez-Herencia, and E. Gordo, Design of Ti Microstructure by Addition of Ceramic Particles by Colloidal Techniques.

[10] I. Agote, M.A. Lagos, Study on the Effect of Processing Method and Type of Reinforcements on Titanium Matrix Composite Properties.

[11] W. Limberg, T. Ebel, K.U. Kainer, Addition of Rare Earth Elements to MIM-Processed TiAl6V4.

[12] J.A. Grohowski and J.C. Piemme, Titanium MIM: Technology for Medical Device Manufacturing.

[13] M. Jurisch, T. Studnitzky, O.
 Andersen, B. Kieback, 3D Screen
 Printing for the Fabrication of Small
 Intricate Ti-6Al-4V Parts.

[14] A. Kirchner, B. Klöden, J. Luft, T. Weißgärber, B. Kieback, Process Window for Electron Beam Melting of Ti-6Al-4V. [15] S.L.G. Petroni, E.T. Galvani, C.A.A. Cairo, C.C. Girotto, V.A.R. Henriques, Compaction and Densification Behaviour of Titanium Hydride Powder.

[16] T. Ebel, J. Soyama, M. Oehring, F. Pyczak, K.U. Kainer, Creep resistance of intermetallic TNB-V5 alloy processed by Metal Injection Moulding

#### Author

Dr. Thomas Ebel Helmholtz-Zentrum Geesthacht Centre for Materials and Coastal Research Max-Planck-Straße 1 D-21502 Geesthacht Germany Tel: +49 (0) 41 52 / 87 - 1944 Email: thomas.ebel@hzg.de www.hzq.de