

ON THE MICROSTRUCTURE AND PROPERTIES OF THE Ti-3Al-2.5V ALLOY OBTAINED BY POWDER METALLURGY

L. Bolzoni^{1,2,*}, E.M. Ruiz-Navas¹, E. Gordo¹

¹Department of Materials Science and Engineering, University Carlos III of Madrid,
Avda. de la Universidad, 30, 28911 Leganes, Madrid - Spain

²BCAST, Brunel University, Uxbridge, Middlesex, UB8 3PH, London - UK

*bolzoni.leandro@gmail.com

Keywords: Ti-3Al-2.5V, titanium powder metallurgy, blending elemental approach, homogeneous microstructure

Abstract

Ti-3Al-2.5V alloy was originally developed for the fabrication of hydraulic and fuel structures in conventional airplanes because it has intermediate performances (i.e. mechanical and corrosion resistance) compared to titanium and Ti-6Al-4V. The production of structural titanium components via powder metallurgy has been lately considered and a lot of interest has been shown from the aerospace industry due to the development of novel production methods which avoid the presence of chlorides. In this work the production and characterisation of the Ti-3Al-2.5V alloy produced considering the blending elemental approach is assessed. The alloy is consolidated by two conventional powder metallurgy routes (pressing and sintering and hot-pressing) in order to study the effect of the processing parameters. Complete diffusion of the alloying elements and homogeneous microstructures are generally achieved. The mechanical performances of the sintered products, which are comparable to those of the wrought alloy, are correlated to the relative density and microstructural features.

Introduction

Titanium is the 9th most abundant element on the earth or, equivalent, the 4th most abundant structural metal after iron, aluminium and magnesium, and it is a light metal normally found in different minerals such as the ilmenite (FeO·TiO₂) and rutile, which are the two most important from a commercial point of view [1]. Due to its position in the periodic table and, therefore, to its electronic structure, titanium is an electropositive and reactive metal characterised by a particular combination of properties like low density (i.e. 4.5 g/cm³, approximately 60% that of steel) and high strength (comparable to that of many types of steel) which confers it the highest specific strength among metals. Moreover, titanium is characterised by low elastic modulus (i.e. 110 MPa, about 50% that of steel) and, therefore, high modulus of resilience, outstanding corrosion resistance and biocompatible due to its passivation, as well as low thermal conductivity and low electrical conductivity [2].

Titanium is characterised by polymorphism, this is because the atoms pack themselves in a hexagonal structure (H.C.P.) at room temperature, known as α phase, and in a body centred cubic structure (B.C.C) starting from 882°C, labelled as β phase. This way, the alloying elements, which are normally used to improve the mechanical properties, can be subdivided as a function of the stabilizing effect [3].

On the one side, alpha-stabilising elements, such as aluminium, oxygen or nitrogen increase the beta transus temperature. On the other side, the beta stabilisers decrease the beta transus and are normally divided between isomorphous (such as V, Mo and Nb) and eutectoid, like Fe, Mn or Cr as a function of the phase transformation that they induce.

Titanium alloys are conventionally divided as a function of the phases that characterised the microstructure of the alloy at room temperature after an annealing treatment. Therefore, titanium alloys can be classified as follows: α -Ti alloys, which are single phase materials, $\alpha + \beta$ alloys (which have both α and β -stabilizers) and β -Ti alloys, which are commonly divided into stable and metastable β . Sometime, α -Ti alloys contain a small percentages of β stabilizers (1-2 wt.%) which permits the formation of small quantity of β phase, and are, thus, called *casi- α* or *super- α* alloys and an example is the Ti-3Al-2.5V alloy [4]. Precisely, the development of the Ti-3Al-2.5V alloy, which has intermediate performances (i.e. mechanical and corrosion resistance) compared to titanium and Ti-6Al-4V, is due to the aeronautical industry were it was originally use to produce hydraulic and fuel structures in conventional airplanes [5].

Titanium components are generally obtained by ingot metallurgy plus primary deformation processes and secondary operations (i.e. machining) or by casting methods. Nonetheless, due to the intrinsic nature of titanium, each fabrication route has its own inconveniences which make the production of titanium components more expensive in comparison to that of other metals. In particular, titanium reacts with the atmosphere and, therefore, has to be processed under vacuum, titanium has low thermal conductivity which makes it difficult to machine and molten titanium reacts with the cast house tools, like moulds and crucibles, forming a brittle superficial layer (i.e. α -case) which has to be removed.

The employment of powder metallurgy (PM) techniques permit to limit to some extent the problems related to the fabrication of titanium products because PM methods are near-net-shape processes where machining is avoided or very much limited [6] and sintering is, generally, carried out at temperatures below the melting point of titanium, which limits the interaction with the processing tools (i.e. sintering trays and containers). All of these aspects should reflect on a reduction of the final cost of titanium products. Moreover, in the last decade there have been new exciting development for the extraction of titanium by reduction processes, such as the ITP/Armstrong [7] and the Cambridge/FFC (Fray-Farthing-Chen) [8] processes and the PM industry could take advantage of that because the main product of these extraction processes is pure titanium in the form of powder.

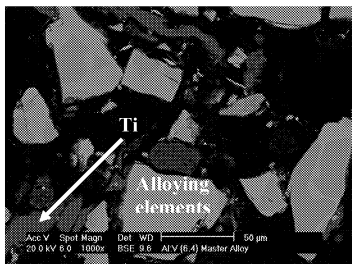
When it comes to the processing of titanium by PM techniques, which started in the 1970's [9], there are two main approaches depending on the nature of the powder, namely, prealloyed and blended elemental although in this last case there is also the possibility to use master alloys to add the alloying elements [10]. Originally, spherical prealloyed powders were shaped by advanced techniques such as hot isostatic pressing whilst blended elemental powder were processed by means of pressing and sintering [11]. Nonetheless, nowadays there are available prealloyed powders with irregular morphology because produced by comminution processes (i.e. hydride-dehydride - HDH) which can be employed for their shaping by the cheapest PM route of cold uniaxial pressing and sintering.

Most of the literature available about the PM of titanium focus on the Ti-6Al-4V alloy, because it still represents the titanium workhorse, whereas there are almost no study about the Ti-3Al-2.5V alloy. The aim of this work is to present, analyse and discuss the comparison of the microstructure and properties which can be obtained when processing the Ti-3Al-2.5V alloy by means of different powder metallurgy routes.

Experimental Procedure

The Ti-3Al-2.5V alloy powder was produced considering the blending elemental approach and the morphology of the starting powders is irregular which guarantee the shaping of the Ti-3Al-2.5V powder during its processing by cold uniaxial pressing and sintering. Some other details about the powder studied are shown in Table 1. Specifically the powders were mixed by means of a Turbula mixer during 30 min to guarantee homogenisation.

Table I. Details of the starting powders.

Property		Morphology
Density	4.48 [g/cm ³]	
Maximum particle size	< 90 [μm]	
Chemical composition	Ti (Balance), O (< 0.40 wt.%), N (~ 0.01 wt.%)	
β transus	935 [°C]	
Melting temperature	~ 1700 [°C]	

As it can be seen in Table 1, the produced Ti-3Al-2.5V powder has a density of 4.48 g/cm³, maximum particle size of 90 μm, oxygen content lower than 0.4 wt.%, nitrogen content of approximately 0.01 wt.% and its β transus is 935°C.

The produced Ti-3Al-2.5V powder was processed by two PM routes, namely, cold uniaxial pressing and sintering (P&S) and conventional uniaxial hot-pressing (HP), where the thermal energy is supplied by high-resistance heating elements. More in detail, for the P&S method the samples were pressed at 700 MPa using a uniaxial press and a floating die whose walls were lubricated with zinc stearate. On purpose no lubricant was added during the mixing of the powder to prevent or, at least, limit to the maximum extent the contamination of the powder. The green samples were sintered under high vacuum (10⁻⁵ mbar) during 2 hours while simultaneously ranging the sintering temperature in between 900°C and 1300°C. Heating and cooling rates were fixed at 5°C/s and the samples were laid into zirconia balls inside an alumina tray. In the case of the HP process, loose powder was poured into a graphite mould coated with low-reactivity BN spray and, initially, cold uniaxially pressed at 18 MPa. Sintering parameters were: sintering temperature range of 900-1300°C, applied uniaxial pressure 30 MPa, vacuum level of 10⁻¹ mbar, heating rate of 10°C/s and furnace cooling.

Concerning the characterisation of the sintered specimens, density values were attained by means of water displacement measurements based on Archimedes' principle. Micrographs of polished (silica gel) and etched (Kroll's reagent) samples were taken to study the microstructural features that characterise the materials. Finally a DIGI-TESTOR 930 hardness tester was used to perform Vickers hardness measurements.

Results and Discussion

The comparison of the variation of the relative density as a function of the processing temperature for P&S and HP samples is presented in Figure 1.

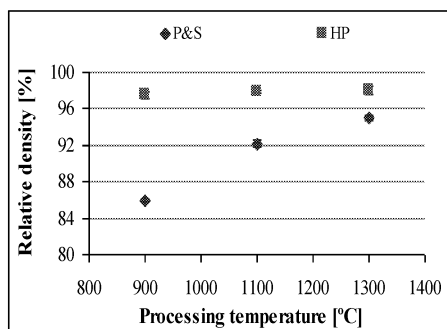


Figure 1. Relative density as a function of the processing temperature for P&S and HP samples.

By the analysis of the data of the relative density of the P&S samples (Figure 1), it can be seen that the relative density increases with the increment of the sintering temperature, which is the normal behaviour of cold uniaxial pressed and sintered components when the other processing parameters are kept constant. Nevertheless, it can also be noticed that the increment of the relative density is greater when raising the temperature from 900°C to 1100°C than from 1100°C to 1300°C. This behaviour seems to indicate that the densification of the samples takes place in the low part of the sintering temperature window studied whilst at 1300°C a greater part of the thermal energy supplied to the system is spent in grain growth, although this has to be confirmed by means of the microstructural analysis. The final relative density values of the Ti-3Al-2.5V alloy are directly comparable to those found by other authors when processing titanium and titanium alloys, mainly the Ti-6Al-4V alloy, by the P&S route [12, 13].

In the case of the Ti-3Al-2.5V specimens obtained by HP there is a very slight increment of the relative density, approximately 0.3%, with the increment of the processing temperature but the increasing trend is not as pronounced as for the P&S samples and it can be said that, in the sintering temperature window analysed, the sintering temperature has very little influence on the final relative density. No terms of comparison could be found in the literature for the processing of the Ti-3Al-2.5V alloy by HP. Nonetheless, the values shown in Figure 1 are comparable to those obtained for elemental titanium [14, 15].

By the comparison of the data plotted in Figure 1 for the P&S and HP specimens, it can be highlighted that the simultaneous application of a uniaxial pressure during the sintering of titanium alloys powders is very beneficial in terms of relative density especially at very low sintering temperature and this benefit fades with the increment of the processing temperature because the difference decreases from approximately 12% at 900°C down to 3% at 1300°C.

Representative optical micrographs of the microstructural analysis carried out to check the homogeneity of the distribution of the alloying elements and to characterise the microstructural features of the P&S and HP sintered Ti-3Al-2.5V samples are shown in Figure 2.

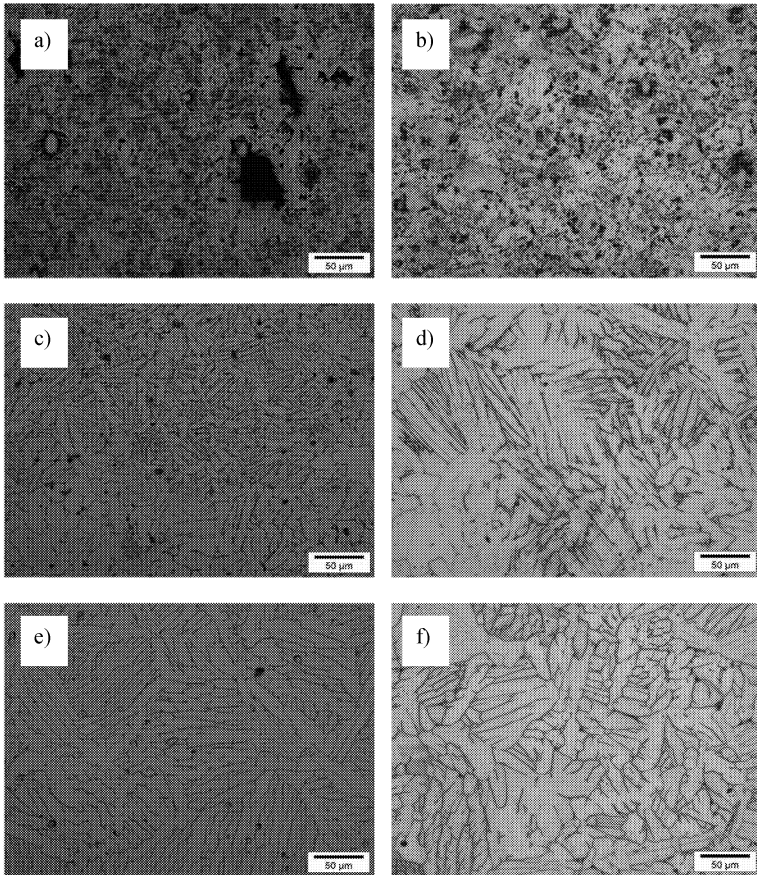


Figure 2. Optical micrographs of the samples sintered at 900°C: a) P&S and b) HP, sintered at 1100°C: c) P&S and d) HP and sintered at 1300°C: e) P&S and f) HP.

From the analysis of the micrographs of the samples processed at 900°C (Figure 2 a and b), it can be seen that regardless of the PM techniques employed to consolidate the Ti-3Al-2.5V powder the microstructure is composed of alpha grains and some alloying elements undissolved particles although some small $\alpha+\beta$ lamellae islands can be found most probably near the original master alloy particles. Moreover, it can be noticed that the sintering of the material has already started because many interparticle boundaries have disappear. Therefore, this processing temperature is to low to guarantee the complete diffusion of the alloying elements towards the titanium matrix also due to the fact that the sintering temperature is lower than the beta transus of the alloy.

Nevertheless, by the comparison of the processing route, it can be seen that P&S samples are characterised by the presence of relatively big irregular pores, some of which are a consequence of the Kirkendall effect [16] (i.e. different diffusion rate among the alloying elements and between them and titanium), which is not the case for the HP specimens. This difference clearly highlight the effect of the simultaneous application of a uniaxial pressure during the sintering. An increment of the processing temperature to 1100°C (Figure 2 c and d) leads to the formation of a more homogeneous microstructure composed by alpha grains and $\alpha+\beta$ lamellae even of their distribution seems to be still quite inhomogeneous. As it was checked by means of EDS analysis, this is because also the processing temperature of 1100°C does not assure the complete homogenisation of the alloying elements in the whole microstructure. Regarding the residual porosity, P&S samples consolidated at 1100°C are characterised by a homogeneous distribution of almost spherical pores mainly located at the grain boundaries whilst the HP samples have very tiny spherical pores indicating that the densification of the material is practically completed. When considering the micrographs of the components sintered at 1300°C (Figure 2 e and f), it can be seen that the microstructure of the Ti-3Al-2.5V samples is composed of by alpha grains and $\alpha+\beta$ lamellae typical of this alloy when slow cooled from above its beta transus and their distribution is homogeneous throughout the whole microstructure as consequence of the fact that the diffusion of the alloying elements towards the titanium matrix is completed. As for the samples processed at 1100°C, the residual porosity of the materials sintered at 1300°C is mainly spherical in shape and it is located at the grain boundaries but its volumetric percentage is significantly lower for both P&S and HP specimens in agreement with the relative density data shown in Figure 1.

The results of the Vickers hardness measurements performed on the cross-section of the P&S and HP specimens is displayed as a function of the processing temperature in Figure 3.

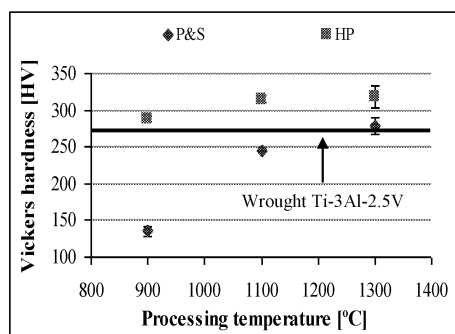


Figure 3. Vickers hardness as a function of the processing temperature for P&S and HP samples.

From the data of the Vickers hardness plotted in Figure 3, it can be seen that this property increases with the increment of the processing temperature for both P&S and HP samples. Nonetheless, as it was for the relative density, the increment experienced by the P&S specimens is much more pronounced with respect to that of the HP samples. Specifically, the hardness of P&S specimens increases from approximately 140HV at 900°C to 280HV at 1300°C whilst the hardness of HP samples increases from about 290HV at 900°C to 320HV at 1300°C.

Therefore, as in the case of the relative density, the simultaneous application of temperature and pressure for the consolidation of titanium alloys powders is much more beneficial at low sintering temperature and then the advantage fades with the increment of the processing temperature. As can be seen from the data plotted in Figure 3, hardness values comparable or higher than the value specified for the wrought Ti-3Al-2.5V alloys (i.e. 267 HV in the annealed state [4]) can be obtained with PM components. Specifically, for P&S samples a minimum sintering temperature of 1200°C seems that has to be used whilst for HP specimens higher values are always reached independently of the processing temperature. This behaviour is dictated by the level of relative density attained (Figure 1) which is always higher for HP samples more than from the homogeneity of the distribution of the alloying elements. Therefore, it seems that with a minimum relative density of 95%, PM components have higher hardness with respect to the wrought products, despite the fact of the presence of the residual porosity. This is due to the fact that the PM components analysed in this work have higher content of interstitial elements, especially oxygen, dissolved in comparison to the wrought alloy (see Table 1) which harden the material [17, 18].

Conclusions

The main conclusion that can be drawn from the analysis of the processing of the Ti-3Al-2.5V alloy by means of different powder metallurgy route is the fact that the complete diffusion of the alloying elements and, thus, homogeneous microstructure are obtained (at high processing temperatures) as well as almost fully dense materials with mechanical properties comparable to those of the wrought alloys. Powder metallurgy could, therefore, be employed to obtain components made out of titanium alloys in a cheaper way with respect to the conventional metallurgy route.

Acknowledgements

The authors want to acknowledge the financial support from the Spanish Ministry of Science through the R&D Projects MAT2009-14448-C02-02 and MAT2009-14547-C02-02, and from Regional Government of Madrid through the ESTRUMAT (S2009/MAT-1585) project.

References

- [1] I.J. Polmear, *Light Alloys. Metallurgy of the Light Metals*, 2nd ed., Edward Arnold, 1989.
- [2] A.M. Russell, K.L. Lee, *Structure-property Relations in Nonferrous Metals*, Wiley-Interscience, 2005.
- [3] R.I. Jaffee, H.M. Burte, *Metallurgical Synthesis*, in: R.I. Jaffee (Ed.) *Proceedings of the Titanium Science and Technology Conference*, Plenum Press, N.Y., 1973, pp. 1665-1693.
- [4] R. Boyer, G. Welsch, E.W. Collings, *Materials Properties Handbook: Titanium Alloys*, in: A. International (Ed.), Ohio, USA, 1998.
- [5] P.A. Russo, R.S. Seagle, *Properties of Titanium for Industrial Applications with Emphasis on Ti-3Al-2.5V*, in: Webster/Young (Ed.) *Industrial Applications of titanium and Zirconium: 3rd Conference*, ASTM International, 1984, pp. 99-112.
- [6] R.M. German, *Powder Metallurgy Science*, 2nd Edition ed., MPIF - Metal Powder Industries Federation, Princeton, USA, 1994.

- [7] S.J. Gerdemann, Titanium Process Technologies, *Advanced Materials & Processes*, 159 (2001) 41-43.
- [8] G.Z. Chen, D.J. Fray, T.W. Farthing, Direct Electrochemical Reduction of Titanium Dioxide to Titanium in Molten Calcium Chloride, *Nature*, 407 (2000) 361-364.
- [9] W. Schatt, K.-P. Wieters, Powder Metallurgy. Processing and Materials, EPMA - European Powder Metallurgy Association, Shrewsbury, UK, 1997.
- [10] F.H. Froes, The Manufacturing of Titanium P/M, The Symposium on High Performance P/M Components, Coimbra, Portugal, 2002, pp. 1-16.
- [11] F.H. Froes, S.J. Mashl, V.S. Moxson, J.C. Hebeisen, V.A. Duz, The Technologies of Titanium Powder Metallurgy, *JOM*, 56 (2004) 46-48.
- [12] O.M. Ivasishin, Cost-effective Manufacturing of Titanium Parts with Powder Metallurgy Approach, *Materials Forum* 29 (2005) 1-8.
- [13] F.H. Froes, O.M. Ivasishin, V.S. Moxson, D.G. Savvakina, K.A. Bondareva, A.M. Demidik, Cost-effective Synthesis of Ti-6Al-4V Alloy Components via the Blended Elemental P/M Approach, in: W. TMS, PA (Ed.) Symposium on TMS Symposium on High Performance Metallic Materials for Cost Sensitive Applications, Seattle, WA, 2002.
- [14] C.G. Goetzl, V.S. de Marchi, Electrically Activated Pressure Sintering (Spark Sintering) of Titanium Powders, *Powder Metallurgy International*, 3 (1971) 80-87.
- [15] C.G. Goetzl, V.S. de Marchi, Electrically Activated Pressure Sintering (Spark Sintering) of Titanium Powders, *Powder Metallurgy International*, 3 (1971) 134-136.
- [16] F. Seitz, On the Porosity Observed in the Kirkendall Effect, *Acta Metallurgica*, 3 (1953) 355-369.
- [17] R.I. Jaffee, I.E. Campbell, The Effect of Oxygen, Nitrogen and Hydrogen on Iodide Refined Titanium, *Transactions of the American Institute of Mining and Metallurgical Engineers*, 185 (1949) 646-654.
- [18] R.I. Jaffee, H.R. Ogden, D.J. Maykuth, Alloys of Titanium with Carbon, Oxygen and Nitrogen, *Transactions of the American Institute of Mining and Metallurgical Engineers*, 188 (1950) 1261-1266.