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Research Paper

Mechanical behaviour of pressed and sintered titanium alloys obtained from master alloy addition powders

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ABSTRACT

The fabrication of the workhorse Ti–6Al–4V alloy and of the Ti–3Al–2.5V alloy was studied considering the master alloy addition variant of the blending elemental approach conventionally used for titanium powder metallurgy. The powders were characterised by means thermal analysis and X-ray diffraction and shaped by means of uniaxial pressing. The microstructural evolution with the sintering temperature (900–1400 °C) was evaluated by SEM and EDS was used to study the composition. XRD patterns as well as the density by Archimedes method were also obtained. The results indicate that master alloy addition is a suitable way to fabricate well developed titanium alloy but also to produce alloy with the desired composition, not available commercially. Density of 4.3 g/cm^3 can be obtained where a temperature higher than 1200 °C is needed for the complete diffusion of the alloying elements. Flexural properties comparable to those specified for wrought Ti–6Al–4V medical devices are, generally, obtained.

1. Introduction

Titanium is characterised by a combination of properties which differentiate it and that can be resumed in three main features. The first one is the relatively low density, being a 40% lighter than steel, making it a light metal (Leyens and Peters, 2003). Titanium presents strength comparable to that of steel; this aspect combine with the low density makes it the structural metal with the highest specific strength being more efficient in terms of weight/volume ratio and, therefore, resulting in a lighter structural component compared to steel or the other light alloy such as aluminum and magnesium with similar mechanical strength (Lütjering and Williams, 2003). Moreover, the high strength combined with the low elastic modulus, half of that of steel, results in a superior toughness or modulus of resilience (Russell and Lee, 2005). Third, titanium is highly reactive with oxygen forming a passivation layer which confers it an excellent corrosion resistance in many aggressive environments as well as an outstanding biocompatibility and, thus, suitable for the production of medical prosthesis. Even though of its mechanical properties and chemical stability, titanium is still mainly employed in high-demanding industries and not in high productive sector such as the automotive. The application of the powder metallurgy (P/M) route should lead to a decrement of the production cost of titanium which represents one of the mayor limiting factors for its spreading and avoid segregation of non toxic but heavy alloying elements such as Nb, Zr or Ta (Niinomi, 2002). Nowadays, the availability of cleaner starting powder compared to sponge fines,

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whose chlorides can cause problem during sintering hindering the closure of the residual porosity, should allow to obtain components with better performances. The two classical titanium P/M approaches are the prealloyed (PA) and the blending elemental (BE) route where this last one appears to be one of the most promising to produce low-cost titanium products (Moxson et al., 1998). Among titanium alloys, the Ti–6Al–4V is known as the workhorse of the titanium industry being used in many advanced applications, especially aerospace and military, while the Ti–3Al–2.5V is mainly used to attain tubular products for aircraft hydraulic and fuel systems although, lately, it has been considered for the fabrication of sports equipments as well as medical and dental products (Boyer et al., 1998). Until recently, the main approach taken for the introduction of orthopedic materials has involved adaptation of existing materials (Dewidar et al., 2006) and, therefore, there are not studied about the production of the Ti–3Al–2.5V alloy by conventional P/M. Lately, the Ti–3Al–2.5V has been processed by conventional (Bolzoni et al., 2012a) and inductive hot-pressing (Bolzoni et al., 2012b) to produced fully dense materials. The aim of this work is to study the fabrication of the previously mentioned titanium alloys by means of the conventional P/M route of pressing and sintering considering the master alloy addition approach, a variant of the BE route, and analyse the microstructural evolution of the alloys with the sintering temperature employed, focusing on the diffusion of the alloying elements to justify the properties obtained.

2. Experimental procedure

For the fabrication of the titanium alloys, a hydride-dehydride (HDH) elemental titanium powder and an Al:V master alloy were purchased from GfE Gesellschaft für Elektrometallurgie mbH. Some characteristics of the starting powders provided by the supplier are reported in Table 1.

The acquired Al:V (35:65) master alloy was milled in combination with the elemental titanium to reduce the particle size and adjust the composition with elemental aluminium purchased by Sulzer Metco Ltd. The optimised Al:V master alloy has the correct ratio of alloying elements (60:40) and a particle size lower than 63 μm (Bolzoni et al., 2011) and was, therefore, blended with elemental titanium in a "turbula" mixer during 30 min to obtained the Ti–6Al–4V and Ti–3Al–2.5V alloys. First, the characterisation of the titanium alloy powders was done in terms of differential thermal analysis (DTA), dilatometry and X-ray diffraction (XRD). DTA was carried out by means of a Netzsch STA 449C Júpiter DTA analyser, under inert atmosphere (Ar), where the loose powder was studied considering a thermal cycle composed of a heating (10 °C/min) up to 1400 °C, soak time of 15 min at temperature and cooling (10 °C/min). The same thermal cycle was employed for the dilatometric study of green samples which was done by means of a NETZSCH DIL 402 E dilatometer. A Bruker AXS D8-Advance analyser provided with a Cu $K_{\alpha 1}$ monochromator was used for the XRD analysis of the Ti–6Al–4V and Ti–3Al–2.5V powders and of the sintered specimens. The compaction of the powder was carried out with a uniaxial press applying 700 MPa and using zinc stearate as die wall lubricant for the floating die whereas sintering was done in a high vacuum tubular furnace with a vacuum level of approximately 10^{-5} mbar. Heating and cooling rate was fixed at 5 °C/min and the dwell time at 2 h whilst the sintering temperature was ranged between 900 °C and 1400 °C. The microstructure of the sintered samples was revealed by chemical etching (Kroll's reactant) and analysed by means of scanning electron microscopy in BSE mode while the distribution of the alloying elements was checked by EDS. The mechanical characterisation of the sintered samples was done by the three-point bending tests (ASTM B528) in order to measure the strength and the strain and hardness measurement.

3. Results and discussion

3.1. XRD analysis of the powders

The results of the XRD analysis carried out on the starting powder are shown in Fig. 1.

Regarding the XRD of the Ti–6Al–4V alloy (Fig. 1a), in the powder were identified all the elements used to prepare it. More in detail, they are titanium α phase, due to the HDH elemental titanium powder, elemental aluminium, and aluminium/vanadium phase labelled as Al_2V_3 due to the master alloy employed during the production of the powder. It is worth mentioning that the main peak of aluminium coincides with the second most intense peak of the titanium α phase (002 at 38.4°). This is the reason why the relative intensity of the main peak of the α phase (101 at 40.1°) is not the highest as expected and as it is for the Ti–3Al–2.5V powder (Fig. 1b). This and the lower relative intensity of the peak of the Al_2V_3 phase are the only differences between the XRD pattern of the two alloys and are mainly due to the amount of alloying elements added to the HDH elemental titanium powder.

Table 1 – Characteristics of the powders used for the production of the Ti–6Al–4V and Ti–3Al–2.5V titanium alloys (supplier specifications).

Characteristic	Elemental Ti	Al:V	Elemental Al
Composition [wt%]	>99.7	35:65	>99
Maximum particle size [μm]	<75	<6300	<150
Particle morphology	Angular	Granules	Spherical
Melting point [°C]	1667	1650–1725	660