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Improving sinterability of Ti–6Al–4V from blended elemental powders through equal channel angular pressing

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ABSTRACT

Ti–6Al–4V billets were fabricated from a blend of commercially-pure titanium (CP Ti) and a master alloy (Al60V40) powders, using equal channel angular pressing (ECAP) as a tool for powder consolidation. The density of the compact in excess of 99% was achieved at a sintering temperature that was markedly lower than the temperatures reported for conventional blended elemental (BE) powder routes. A detailed electron microscopy investigation was employed to analyse the microstructures of the powder compact and account for its improved sinterability. The results indicated that the intense shear imposed by ECAP has generated localised strain heterogeneities in the CP Ti particles along with $\{10\bar{1}1\}_\alpha$-type mechanical twins. Shear-assisted elimination of Ti oxides on the powder particle surface gave rise to direct bonding between the Ti phases. While powder particles of CP Ti and master alloy were always separated by an interfacial oxide layer, interdiffusion of Al and Ti atoms was detected by STEM-EDX, which indicates the existence of diffusion bonding between unlike particles. The roles of severe plastic deformation in the evolution of interfacial and lattice structures of the powder particles are discussed with regard to the enhanced compaction and sintering performance.

1. Introduction

The high extraction and fabrication cost of titanium hinders its wider use for conventional engineering applications beyond the aerospace and medical implant ones. A recent focus of titanium technology has been put on the development of cost-effective approaches to manufacturing of titanium [1]. Low-cost titanium is anticipated to stimulate a multitude of civil applications, including those in the automotive industry [2–4], architecture, and cookware [5]. Among the different stages of titanium production, the most costly ones are associated with the fabrication of wrought products via traditional ingot routes [1]. A more economical alternative is the near-net shape manufacturing, such as powder metallurgy (PM). Numerous studies have been concerned with the production of titanium components by PM methods [6–14], mostly with the fabrication of alloy Ti–6Al–4V, through either blended elemental (BE) [7–9] or pre-alloying (PA) [12–17] techniques.

In general, PM approaches involve blending and compaction of powders to form "green compacts", followed by a vacuum heat treatment at elevated temperatures to facilitate densification of the component. The chemical homogeneity and the residual level of porosity have a critical influence on the mechanical properties of the components. For instance, the ultimate tensile strength of a sintered Ti–6Al–4V part having a 95% relative density attained only a value of 800 MPa, as compared to 930 MPa for a 99%-dense part [6]. Traditional "press-and-sinter" BE approach is characterised by a lower density of the product (typically 95% relative density) than the PA approach that employs hot-isostatic pressing (HIP), which can lead to fully-dense products. Nevertheless, the latter approach is more costly. Over the years, progress with BE technologies was made, owing to the use of hydrogenated Ti powder instead of sponge fines [18], secondary operations (e.g. hot forging) [19], and proprietary processes, such as the MR-9^{TM} process (Imperial Clevite) [8], all of which were reportedly able to raise the relative density of the products to a level in excess of 99%.

Compared with the PA approach, the sintering of green compacts comprising a mixture of different BE powders involves potentially more complicated mass transfer processes. The elimination of voids and the homogenisation of chemical composition hinge on the degree of metallurgical contact between particles and the efficacy of surface diffusion as well as volume (lattice) diffusion in individual particulate materials. Previous studies conducted by Ivasishin and co-workers [20] suggested that the sinterability of BE compacts was also subjected to influences of the chemistry of the master alloy (MA) powder and by the presence of impurity Fe, which tended to form a eutectic phase with Ti and produce a higher volume fraction of voids.

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We have recently investigated the fabrication of Ti–6Al–4V alloy via BE-PM approach, using equal channel angular pressing (ECAP) as a means of powder consolidation to explore the effects of severe plastic deformation on compaction and sintering kinetics of various powder blends [21,22]. The results revealed that the green compacts consolidated by ECAP readily attained over 99% of theoretical density. In particular, the blend comprising CP Ti powder and Al60V40 MA powder achieved chemical and structural homogenisation upon a vacuum treatment at 1100 °C for 4 h, a temperature markedly lower than the range of 1260–1350 °C reported for the sintering of cold-pressed compacts [19,23]. A further advantage of this fabrication route is that the mechanical properties of the resulting billets compare favourably to those produced through the more conventional PA+HIP routes.

The reasons for selecting Al60V40 MA powder are two-fold. First of all, in a previous work [15] we demonstrated that Ti–6Al–4V PA powder can be consolidated using ECAP. However, in contrast to expensive PA Ti–6Al–4V, further price reduction is expected from using CP Ti along with a MA. Secondly, Ivasishin et al. [4,23] reported that the choice of a MA as well as BE powder and their particle sizes had an effect on the density of compact before and after sintering. It was found that the use of elemental Al powder caused the occurrence of relatively large voids due to the formation of liquid Al accompanying the $3Al+Ti \rightarrow TiAl_3$ reaction during sintering. This effect could only be suppressed when very fine but expensive Al powder was used. Therefore, the powder blend incorporating CP Ti and Al60V40 MA has become the object of our research. The present paper addresses the microstructural factors that influence the sinterability of the ECAP-processed compact, including the evolution of deformation-induced defects inside the particles and the characteristics of the particle-particle interfaces that underwent the intense shear deformation under ECAP.

2. Experimental

The elemental powder blend used in this study comprised a CP Ti (ASTM Grade 2) powder supplied by Sumitomo, Japan, and a binary MA powder having a chemical composition of 60 wt% of Al and 40 wt% of V (Al60V40, supplied by CMC, USA), hereafter designated as MA. The CP Ti powder contained an oxygen level of 0.217 wt% with trace impurities (H, N, C, Fe), each below 0.02 wt%. The particle sizes of the CP Ti and Al60V40 powder used were less than 150 μm and 160 μm, respectively.

The CP Ti powder was mixed with the Al60V40 MA powder on air using a SPEX SamplePrep 8000 Mixer/Mill^{TM} in the absence of any grinding medium for 1 h at a clamp speed of 875 cycles/min. Consolidation of the mixed powder blend was performed using a custom-designed ECAP apparatus with a die channel of 10 mm in diameter and 90° angle, the detailed operation of which was described elsewhere [21]. The powder materials were heated up inside the ECAP die to a temperature of 400 °C and extruded with a vertical punch moving at a speed of 1 mm/s. The extrusion of the compact was opposed by a back-pressure of 210 MPa applied through a second punch at the exit channel.

Sintering treatment of the compact was performed in an alumina tube furnace with a continuous flow of argon gas for 4 h at 1100 °C, followed by furnace cooling. The rates for heating and cooling were both controlled to be 2 °C/min. The bulk chemistry of the sintered billet was analysed using the inductively coupled plasma atomic emission spectroscopy (ICP-AES) method, while the nitrogen and oxygen levels were specifically measured with a LECO TCH600 (H) apparatus using the Ti LECO analysis method. The composition of the billet, cf. Table 1, meets the ASTM standard (F-1472) for wrought Ti–6Al–4V alloys.

Table 1
Chemical analysis of a billet sintered 4 h at 1100 °C.

Element	Ti	Al	V	O	N	C	Fe
wt%	Bal.	5.69	3.91	0.14	< 0.01	0.01	0.03

Microstructural examinations were performed in a JEOL 7001F FEG scanning electron microscope (SEM). As-received CP Ti and MA particles were mounted in resin and ground with SiC papers (up to 2400-grit), followed by mirror-polishing with a colloidal silica suspension (OP-S, Struers). Backscattered electron (BSE) imaging mode was adopted to distinguish between different phases in the powder materials based on the atomic number (Z) contrast. The imaging mode also provided crystallographic orientation contrast in single-phase regions via electron channelling. Internal structures of the billets consolidated by ECAP were investigated in detail with a Philips CM20 transmission electron microscope (TEM) operating at 200 kV. Energy dispersive X-ray spectroscopy (EDX) was employed to analyse the chemistry at particle interfaces using a JEOL 2100F FEG TEM operating at 200 kV under a scanning transmission electron microscope (STEM) mode. Thin-foil specimens for the TEM investigations were prepared by slicing the billets in a direction perpendicular to the pressing axis with a low-speed saw. The slices of a thickness of ~200 μm were subsequently dimpled to around 50 μm thickness, followed by ion-beam milling using a Gatan PIPS^{TM} system at an anode voltage of 5 kV and a milling angle of 4°.

3. Results and discussion

3.1. Initial powder materials

SEM images in Fig. 1 illustrate the morphologies and microstructures of the initial powder materials. The CP Ti and the MA particles both appear to be highly facetted and irregular in shape. Cracks were commonly observed on the particles, suggesting that these particles were produced by crushing in their brittle states (e.g. the hydrided state of Ti). Fig. 1(b,c) reveals the cross-sections of the CP Ti particles. The particles are seen to contain equiaxed grains with an estimated grain size of the order of 10 μm, with pronounced mechanical twins (up to 1 μm width) inside the individual grains. These twins are believed to have been introduced by crushing, although the possibility that the twins may stem from the grinding/polishing process could not be entirely ruled out. Also seen is the dark spotty contrast uniformly distributed in the Ti matrix, cf. Fig. 1(c), the origin of which may be associated with local aggregates of dislocations, or less likely, light-element inclusions that gave rise to a darker atomic number (Z) contrast. On the other hand, the cross-sections of the MA particles clearly exhibited a two-phase microstructure, cf. Fig. 1(d,e). The occurrence of two Al–V phases was also confirmed by X-ray diffraction data [22]. This is in agreement with the binary Al–V phase diagram, where the alloy composition coincides with a two-phase field containing Al_3V and Al_8V_5 intermetallic compounds [24]. In contrast to the CP Ti, mechanical twinning was not readily observed in the MA powder under similar imaging conditions, see Fig. 1(f). Instead, the intragranular structure of Al_3V phase (i.e., the darker phase in the figure) was found to be decorated by a dense distribution of precipitates (brighter phase), which had a tendency to develop heterogeneously along grain boundaries and dislocations in the bulk.

3.2. Deformation structures after compaction by ECAP

The green compact of the CP Ti and MA powder consolidated by ECAP is illustrated by an optical micrograph in Fig. 2. The CP