



Very strong pure titanium by field assisted hot pressing of dual phase powders



B. Srinivasarao^a, J.M. Torralba^{a,b}, M.A. Jabbari Taleghani^a, M.T. Pérez-Prado^{a,*}

^a IMDEA Materials Institute, C/ Eric Kandel, 2, 28906 Getafe, Madrid, Spain

^b Department of Materials Science and Engineering, Universidad Carlos III Madrid, Leganés, Madrid, Spain

ARTICLE INFO

Article history:

Received 3 January 2014

Accepted 25 February 2014

Available online 5 March 2014

Keywords:

Titanium

Composites

Powder metallurgy

Phase transformations

Field assisted hot pressing

ABSTRACT

Very strong pure Ti, with room temperature compressive yield stresses up to 1.80 GPa, was developed by field assisted hot pressing of a mixture of hcp (α) and fcc powders produced by mechanical milling. During consolidation the fcc phase decomposes into α -Ti and a dispersion of TiC particles. Tuning the pressing temperature and time allows controlling the grain and particle size and, thus, the yield strength and plasticity.

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1. Introduction

High strength pure titanium is an excellent candidate for several structural and biomedical applications as it has good specific properties, high corrosion resistance and good biocompatibility [1]. Several strategies have been employed to enhance the mechanical behavior of this material. Alloying leads to increased strength but lower corrosion and biocompatibility [2]. Severe plastic deformation (SPD) techniques are utilized to fabricate very strong ultrafine-grained structures with yield stresses (σ_y) as high as 1.30 GPa [3–5]. Similar strength levels are reached in bimodal grain size microstructures fabricated by powder metallurgy routes [6]. Further increases in σ_y values to approximately 1.50 GPa can be achieved by the dispersion of second phase particles such as Y_2O_3 , TiC and TiB in the titanium matrix [7–10]. Finally, high strength values have also been measured in ω -Ti stabilized under ambient conditions by the simultaneous application of pressure and shear [11,12], for which an ultimate tensile strength (UTS) of 1.20 GPa has been reported. In this work we propose an alternative processing route to fabricate a very strong pure Ti that leverages on phase transformation phenomena as a microstructure design tool. The proposed method consists of the consolidation of a dual phase powder by field assisted hot pressing (FAHP). The resulting microstructures and their mechanical behavior are discussed.

2. Materials and methods

Hcp titanium powder with a purity of 99.8% was milled at a speed of 300 rpm with a ball to powder weight ratio of 10:1 in a Fritsch P-6 planetary mill using a WC pot and balls under an inert Ar atmosphere. Following milling the powder was packed in a graphite die with an inner diameter of 10 mm and was pressed using a Gleeble 3800 machine in vacuum (10^{-3} Pa) at 750, 840 and 1070 °C for 1 to 10 min with a heating rate of 100 °C/min and a pressure of 50 MPa. The density of the sintered disks (5 mm in thickness and 10 mm- \varnothing) was measured by the Archimedes method with water as a liquid medium. A value higher than 99% of the theoretical density was achieved in all samples. The phases present after milling and sintering were characterized by conventional X-ray diffraction (XRD). The amount of contamination in the powders was measured by mass spectrometry using LECO TC500 and LECO CS200 glow discharge spectrometers. Microstructural analysis was performed by scanning (SEM) and transmission electron microscopy (TEM). The sintered materials were tested in compression at room temperature (RT) and at a strain rate of 10^{-3} s $^{-1}$. Three samples were tested for each condition.

3. Results and discussion

Fig. 1(a) is a sequence of XRD patterns illustrating the evolution of the phases present in the pure Ti powder with increasing milling time. It can be seen that, after 10 h of milling, an fcc phase starts forming and that, after 35 h, the transformation is complete. This observation is consistent with the literature [13–15]. The formation

* Corresponding author. Fax: +34 915503047.

E-mail address: teresa.perez.prado@imdea.org (M.T. Pérez-Prado).

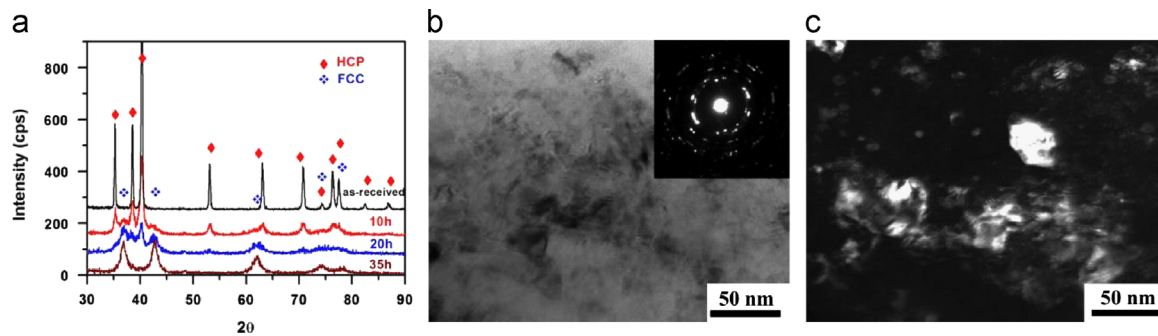


Fig. 1. (a) XRD patterns illustrating the transformation from hcp to fcc Ti with increasing milling time; (b) and (c) TEM bright field and dark field micrographs showing the grain size in the powder particles after 10 h of milling.

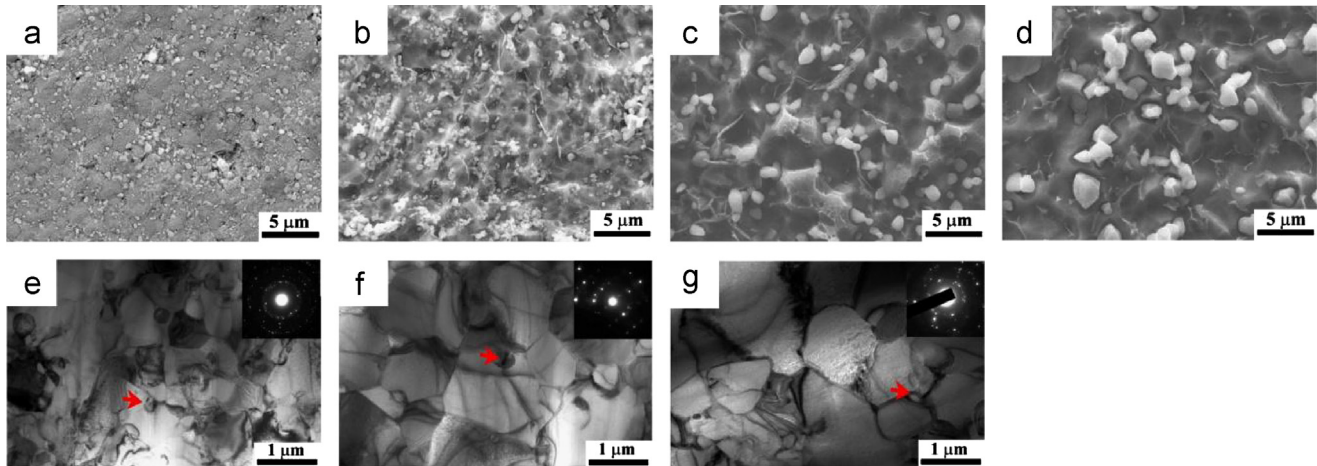


Fig. 2. (a)–(d) SEM micrographs illustrating the microstructure of the samples consolidated at 750 °C for 1 min (a), 750 °C for 10 min (b), 840 °C for 8 min (c), and 1070 °C for 10 min (d). (e)–(g) Bright field TEM micrographs corresponding to the samples consolidated at 750 °C for 1 min (e), 750 °C for 10 min (f), and 840 °C for 8 min (g).

of the fcc phase was attributed both to the negative hydrostatic pressure arising from grain refinement during milling [13] and to contamination with interstitials of C, O and N, as the mentioned phase transformation was not observed during processing under an ultrahigh purity environment [14]. The average amounts of C, O and N present in the powders of the current study are 1.008, 0.668 and 0.386 at%, respectively. In agreement with the mentioned studies, Fig. 1(a) shows no evidence of any compounds containing O, C, and N, suggesting that these elements are mostly in solid solution. The fcc-Ti phase formed during milling is very hard ($HV1 \sim 800$). Pressing of single-phase fcc powders (milled during 35 h) at 1000 °C for 10 min gave rise to compacts with a σ_y of 2 GPa but no plasticity. Thus, the powders milled for 10 h were selected for further processing as it was presumed that the dual phase (hcp + fcc) mixture could lead to a good combination of strength and ductility. The grain size of the powder particles after 10 h of milling is in the range of 10 to 50 nm, as can be observed in Fig. 1 (b) and (c).

Fig. 2 is a collection of SEM (Fig. 2(a)–(d)) and TEM (Fig. 2(e)–(g)) micrographs corresponding to the samples consolidated at 750 °C for 1 min (Fig. 2(a) and (e)), 750 °C for 10 min (Fig. 2(b) and (f)), 840 °C for 8 min (Fig. 2(c) and (g)), and 1070 °C for 10 min (Fig. 2(d)), respectively. At low sintering temperatures and times the microstructure consists of second phase particles dispersed in a α -Ti matrix, containing both coarse ($d > 1 \mu\text{m}$) and ultrafine grains ($200 \text{ nm} < d < 1 \mu\text{m}$). The particles (indicated with red arrows in Fig. 2(e)–(g)) are mostly located at the grain boundaries. With increasing sintering temperatures and/or times both the grain size of the α -Ti matrix and the size of second phase particles increase, but it is still possible to observe some ultrafine grains in the samples consolidated at 840 °C for 8 min.

Fig. 3(a) illustrates several XRD patterns corresponding to the samples pressed at temperatures ranging from 750 to 1070 °C for 1–10 min. The intensity of the hcp peaks increases with increasing pressing temperature. Additionally, some new peaks become apparent after sintering at 750 °C for 1 min, suggesting that the fcc phase transforms into a new phase. This is consistent with the presence of the second phase particles, evident in Fig. 2. Phasha et al. [15] observed the same phase transition when heating fcc-Ti at high temperatures and they interpreted it as a decomposition of this phase into a mixture of hcp-Ti and a rhombohedral Ti phase having the space group R-3m #166 and lattice parameters of $a=b=c=3.021^\circ$ and $\alpha=\beta=\gamma=59.992^\circ$. The new peaks appearing in the present study upon consolidation are located at the same 2θ values as those reported by Phasha et al. [15]. A close look at XRD databases, however, suggests that these maxima may also correspond to a TiC compound. In order to distinguish between these two phases, further examination of the particles was carried out by x-ray energy dispersive spectroscopy in the SEM. This analysis confirmed that the particles contain Ti and a significant amount of C. A representative C line profile plot is shown in Fig. 3(b). The amount of C approaches a value of 27 at%. The presence of other impurities such as N and O in the particles was found to be negligible. It is true that the amount of C present is lower than the one present in a TiC compound (50 at%). This might be due to an overestimation of the relative amount of Ti as it is impossible to isolate the signal from the particle from that of the matrix.

The formation of the TiC particles reflects that the amount of C in solid solution in the fcc phase exceeds the solubility limit of the hcp phase. The diffusion of this element may be facilitated by the presence of a large boundary area both in the nanocrystalline fcc parent phase and in the resulting hcp-Ti with different volume

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