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New approach to achieve high strength powder metallurgy Ti-6Al-4V alloy through accelerated sintering at β-transus temperature and hydrogenation-dehydrogenation treatment

Fei Cao, K.S. Ravi Chandran *, Pankaj Kumar

Department of Metallurgical Engineering, The University of Utah, Salt Lake City, UT 84112, USA

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ABSTRACT

It is demonstrated that high density powder metallurgy (PM) Ti-6Al-4V alloy, with a finer prior-β grain size, can be achieved by vacuum sintering of TiH_2 compacts at the β-transus (1010 °C) temperature, taking advantage of the accelerated self-diffusion. The subsequent refinement of the coarse lamellar microstructure by hydrogenation-dehydrogenation treatment produces a high tensile strength (~1130 MPa) and ductile (~10%) alloy. The superior tensile strength and ductility result from the nanoscale Widmanstatten structure and the finer prior-β grain size, respectively, created by the two treatments. The results show a promising pathway to make high strength and ductile PM titanium alloys.

There is a large volume of work on PM titanium alloys, since the 1970s, aimed at near-net shape manufacturing of components. However, wide spread application has not been successful due to subpar densities, low mechanical properties, and high cost of manufacturing [1,2]. Previous works [3,4,5] on PM titanium focused on vacuum sintering of blended elemental (BE) powders in the β-phase field, typically >1200 °C. The major problem in this approach is that large prior-β grains and coarse lamellar colony structures are created, leading to a relatively low tensile and fatigue strength [2,3]. Therefore, approaches to accelerate densification, refine α + β microstructure, and improve mechanical properties without mechanical working, are critically needed to create competitive PM titanium alloys.

Recently, work from our group [6] showed that a very fine α + β microstructure in Ti-6Al-4V alloy can be produced by hydrogen-sintering-and-phase-transformation (HSPT) process. The alloy composition meets the ASTM-B348 composition specification for wrought Ti-6Al-4V, especially the oxygen content (~0.2 wt.%). This alloy had a tensile strength of 1033 MPa and an elongation of 13.7% [7]. After the heat treatment to produce a bimodal microstructure, the tensile strength and ductility levels were 1076 MPa and 11.9%, respectively [8]. These strength and ductility levels are higher than that required to meet the ASTM alloy specification. Jia et al. [9] claim that a tensile strength of 1422 MPa and a ductility of 7.2% elongation was achieved in Ti-6Al-4V alloy with a very high oxygen level (~0.52 wt.%) made by forging and heat treatment. This material cannot be classified as ASTM Ti-6Al-4V alloy for several reasons. Not only the oxygen content in this material had highly deviated from the ASTM standard, the ductility level claimed seems to be questionable. This is because extensive studies [10,11] on PM Ti-6Al-4V alloys, have shown, consistently, that ductility decreases to about 2% with O levels > ~0.4 wt.%. Regardless, for meaningful gains in strength and ductility relative to the ASTM standard levels, they must be achieved without any deviation from ASTM specification and without the use of additional mechanical working or heat treatment steps after PM consolidation of Ti-6Al-4V.

The objective of this work is to demonstrate a new, easily implementable, PM processing approach that can lead to high density PM titanium alloys with higher strength and ductility levels. It is shown that this can be achieved while still conforming to ASTM specification for oxygen level, and, without using any additional mechanical working or heat treatment. The technique uses, first, vacuum sintering of BE compacts at the β-transus temperature (1010 °C), followed by a hydrogenation-dehydrogenation (HDH) at a lower temperature, with the intent to keep the processing costs as low as possible. There are two hypotheses to be tested in this context: (i) the anomalously high self-diffusion in Ti should lead to accelerated sintering at the β-transus temperature and should result in finer prior-β grains, and, (ii) the subsequent refinement of microstructure in the HDH step should lead to a large increase in strength without sacrificing ductility too much.

In this study, the PM Ti-6Al-4V alloy samples were sintered starting from cold-isostatic-pressed (350 MPa) cylindrical bars made from the blended mixture of TiH_2 powder (−325 mesh, Reading Alloys, Inc., PA) and 60Al-40V (wt.%) master alloy powder (−400 mesh, Reading alloys Inc., PA). The compacts were vacuum sintered (VS) either at the β-

* Corresponding author.
E-mail address: ravi.chandran@utah.edu (K.S.R. Chandran).

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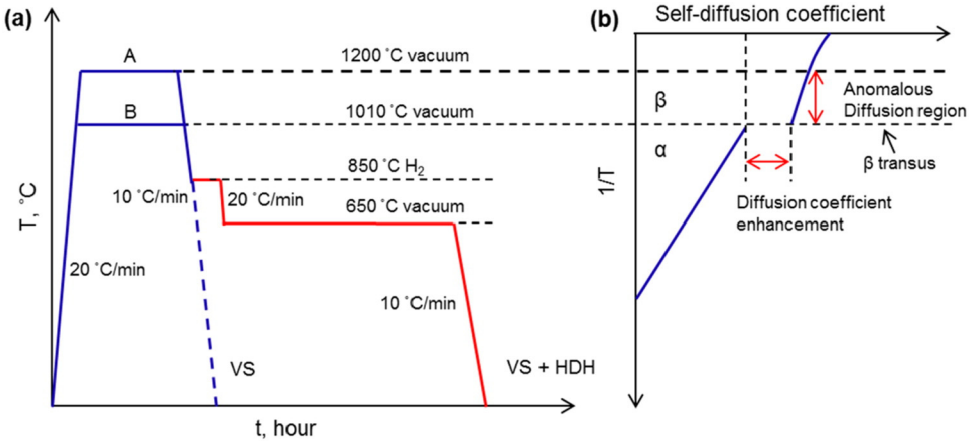

Fig. 1. (a) Temperature-time profile for vacuum sintering and hydrogenation-dehydrogenation treatments in PM Ti-6Al-4V alloy processing, and (b) schematic of the enhancement of self-diffusion and the anomalous diffusion region in titanium near α/β phase transition temperature [14,15].

transus temperature of 1010 °C (named 1010VS) or at 1200 °C (named 1200VS) for 8 h under vacuum (<0.001 Pa). Some of the VS samples were given hydrogenation-dehydrogenation treatment (HDH) to refine the microstructure. In this process, the samples, after vacuum sintering, were cooled at a rate of 10 °C/min to the hydrogenation temperature (Fig. 1(a)). Then the hydrogenation was done (1 atm. pressure of H) at 850 °C for 2 h, followed by dehydrogenation at 650 °C in vacuum for 15 h. It is to be noted that these temperatures are near the lowest possible hydrogenation and dehydrogenation temperatures and were selected on the basis of Ti-H phase diagram by Kerr et al. [12,13]. The hydrogenation at 850 °C for 2 h was found to lead to about 0.7 wt.% H in solution in β titanium, which is slightly larger than the minimum (~0.6 wt.%) required for eutectoid transformation [13].

The 1010VS and 1200VS samples that received HDH treatment are hereafter referred to as 1010VSHDH and 1200VSHDH, respectively. Microstructures and elemental distributions were imaged using optical, scanning electron (Quanta 600F) and transmission electron microscopes (JEM-2800). ASTM E-8 standard tensile bars (6.35 mm diameter, 38.1 mm gage length) were machined from the sintered bars. The tensile tests were performed at room temperature with a strain rate of 10^{-3} S^{-1} and strains were measured using extensometer. At least three tests were performed for each treatment condition and the stress-strain curves were highly reproducible.

Spectroscopic analysis indicated that samples met the ASTM B-348 specification for the PM Ti-6Al-4V alloy. The levels of O, H and N (analyzed by LECO TCH600) in the sintered samples were determined to be (in wt.%) 0.221, 0.00073 and 0.0193, respectively. Table 1 reports the average densities and tensile properties. All the samples achieved densities ≥99%. First, it is interesting to see that the 1010VS sample, sintered at the β-transition temperature, achieved a density of ~99%. This is relatively high, compared to the densities [3] obtained in vacuum sintering of BE compacts at 1260 °C, which are in the range of 95–99%. This high degree of densification, at the lowest possible temperature in β-phase field, is due to the enhanced self-diffusivity in Ti near the phase transition temperature, as illustrated in Fig. 1(b). Titanium exhibits anomalous, elevated self-diffusion behavior [14,15] near the β-transus and on the side of the β-phase. The self-diffusion co-efficient of pure titanium is 6.5×10^{-10} cm^2/s at the β-transus, which is quite close to that (9.6×10^{-9} cm^2/s) at 1200 °C [15]. The diffusivity at the β-transus temperature is also significantly higher than that extrapolated from the data of diffusion in α-phase (1.3×10^{-12} cm^2/s at 880 °C) [15]. Sanchez and De Fontaine [16] explained the diffusion enhancement as due to phase fluctuations at the crystallographic level associated with the formation of omega (ω) precursor phases. In addition, Bokshteyn et al. [14], argue that the local diffusion along β-ω interphase boundaries is 3–4 orders of magnitude higher than the diffusion in the β-phase. Thus, the high density achieved at by β-transus sintering is attributed to the accelerated diffusion arising from both effects.

Microstructures of the VS and VSHDH samples are shown in Fig. 2. The X-ray diffraction data (Supplementary section), indicates that the microstructures have fully transformed after the treatments. Fig. 2(a) and (b) are the optical micrographs of 1010VS and 1200VS samples, respectively, showing the coarse lamellar microstructures. A significant difference in the average size of prior-β grain size is seen—about 67 μm in 1010VS whereas it is 213 μm in 1200VS. The lower prior-β grain size is clearly due to the lower sintering temperature of 1010VS. It is to be note that an average prior-β grain size <100 μm, with 99% density, has not been achieved before by vacuum sintering. The prior-β grain sizes obtained by VS of PM Ti-6Al-4V alloys are normally >100 μm, at densities of 95–99% [2,3].

The microstructures of HDH treated samples, 1010VSHDH and 1200VSHDH, are presented in Fig. 2(c) and (d), respectively. The average sizes of the prior-β grains in 1010VSHDH and 1200VSHDH samples are 71 μm and 221 μm, respectively. This indicates that the HDH treatment did not alter the prior-β grain size much. The SEM imaging of both samples showed the presence of very fine Widmanstatten microstructure (shown in Fig. 2(e) for 1010VSHDH as example), as a result of the eutectoid transformation [12,13] ($\beta_H \rightarrow \alpha +$ hydride). The TEM image (Fig. 2(f)) indicates that the widths of α grains are in hundreds of nanometers. The microscale distribution Al and V corresponding to Fig. 2(f) are shown in Fig. 2(g) and (h), respectively. The elemental map indicates that Al and V is largely segregated in α and β phases,

Table 1
Tensile properties of Ti-6Al-4V alloy on the basis of average values from 3 to 6 samples for each treatment. Standard deviations are given in parentheses.

Treatment ID	Density (%)	Prior β grain size (μm)	Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation (%)	Reduction area (%)
1010VS	98.96	67	821 (±3)	908 (±2)	15.8 (±0.5)	26.9 (±0.9)
1010VSHDH	99.02	71	1049 (±8)	1128 (±5)	10.5 (±0.3)	21.8 (±1.2)
1200VS	99.80	213	841 (±2)	935 (±1)	16.6 (±1.0)	30.6 (±0.3)
1200VSHDH	99.81	221	1053 (±3)	1134 (±4)	7.7 (±0.6)	13.6 (±0.4)
Mill-annealed	100	5.6[a]	912 (±2)	1011(±3)	16.2 (±0.1)	41.9 (±0.2)

[a] Average α grain size.