Ultrafine-grained titanium of high interstitial contents with a good combination of strength and ductility

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Ultrafine-grained titanium of high interstitial contents with a good combination of strength and ductility

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A dehydrided Ti powder of very high oxygen content was successfully consolidated using back pressure equal channel angular processing into a fully dense bulk ultrafine-grained Ti showing apparent compressive ductility as well as high true yield and ultimate strengths of 1350 and 1780 MPa, respectively. Interstitial solid solution strengthening contributed to the majority of the increase in strength with additional contribution from ultrafine grains. Significantly, the material also exhibited much improved ductility for such a high interstitial content, thanks probably to the nonequilibrium grain boundaries and bimodal grain structure introduced during severe plastic deformation. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2832657]

Ultrafine-grained/nanostructured (UFG/NS) metallic materials have been the subject of extensive research thanks to their superior mechanical properties compared to their coarse-grained counterparts.[1–3] Due to the high strength-to-density ratio and excellent biocompatibility for structural and implant applications, high performance titanium and its alloys have been synthesized by controlling at least one microstructural length scale down to the submicron/nanometer regime.[4–8] Ti-based UFG/NS composites, such as Ti–Cu–Ni–Sn–Nb (Ref. 5) and Ti–Fe–Sn (Ref. 8) alloys, exhibited very promising combined mechanical properties with ultimate compressive strength over 2200 MPa and plastic strain higher than 8%. However, most Ti-based UFG/NS composites are multicomponent alloys containing high content of cytotoxic alloying elements such as Ni, Cu, or Fe, which are not biocompatible and, therefore, not desirable for implant applications.[9] Recently, increasing interests in substantial grain refinement using equal channel angular pressing have opened a promising route to producing bulk UFG/NS materials with a good combination of mechanical properties.[3,4] Especially, some of the intrinsically brittle intermetallics exhibited certain ductility after severe plastic deformation (SPD).[1] In this letter, back pressure (BP) equal channel angular (ECA) processing was employed to consolidate a dehydrided (DH) Ti powder of very high oxygen content into a fully dense bulk UFG-Ti exhibiting enhanced strength as well as apparent compressive ductility.

A dehydrided Ti powder (1.15 wt. % O and 0.09 wt. % N) was produced through dehydrogenation of commercial TiH_2 powder (purity of 98%, Sigma-Aldrich) under vacuum at 650 °C for 10 h followed by ring milling for 2 min at room temperature. The average crystallite size of the DH-Ti powder was determined to be 130 nm using x-ray diffraction. Consolidation of the DH-Ti powder was carried out using BP-ECA processing in air for one pass at 630 °C with a back pressure of 200 MPa. For comparison, a micrometer-sized commercially pure (CP) Ti powder of lower interstitial contents (0.71 wt. % O and 0.04 wt. % N) was consolidated for one pass at 600 °C with a back pressure of 50 MPa. The contents of oxygen and nitrogen were analyzed using a carrier gas hot extraction (O/N analyzer/Leco) for consolidated DH-Ti (1.34 wt. % O and 0.3 wt. % N) and CP-Ti (0.73 wt. % O and 0.07 wt. % N). For DH-Ti, the increases in oxygen and nitrogen contents were believed to originate from the entrapped air during BP-ECA processing of ultrafine particles.[10] Density of the consolidated samples was measured using the Archimedes' method. Room-temperature mechanical properties were characterized under quasistatic uniaxial compression loading using a MTS testing machine at a strain rate of 3.0×10^{-3} s^{-1}. The Vickers microhardness (HV) was measured at room temperature under a static load of 100 g for 25 s. Microstructural and fractographic analyses were conducted using x-ray diffractometry (XRD) (Cu $K\alpha$), optical microscopy, scanning electron microscopy (FEI Quanta, 30 kV) and transmission electron microscopy (TEM) (FEI Tecnai F20, 200 kV).

Densities of both consolidated CP-Ti (4.48 g/cm^3) and DH-Ti (4.53 g/cm^3) were very close to the value (4.51–4.54 g/cm^3) of commercially pure Ti,[11] indicating a high efficiency of producing dense materials by BP-ECA processing. The Rietveld refinement[12] of XRD patterns yielded an average crystallite size of 80 nm for both consolidated CP-Ti and DH-Ti. CP-Ti exhibited a microstructure consisting of elongated particles in both the longitudinal [Fig. 1(a)] and cross [Fig. 1(b)] sections with a substantial amount of mechanical twins. Figures 1(c) and 1(d) show TEM images and corresponding selected-area diffraction (SAD) patterns in the cross section. Finer elongated grains [Fig. 1(c)] were revealed in localized regions, possibly derived from deformation twinning.[7] The SAD pattern displayed elongated and clustered diffraction spots, implying the presence of low-angle grain boundaries and lattice distortions. Most of the elongated grains in Fig. 1(c) were, therefore, viewed as subgrains with low-angle misorientations. Figure 1(d) shows a region comprising domains subdivided by high-density dislocations. The SAD pattern revealed that

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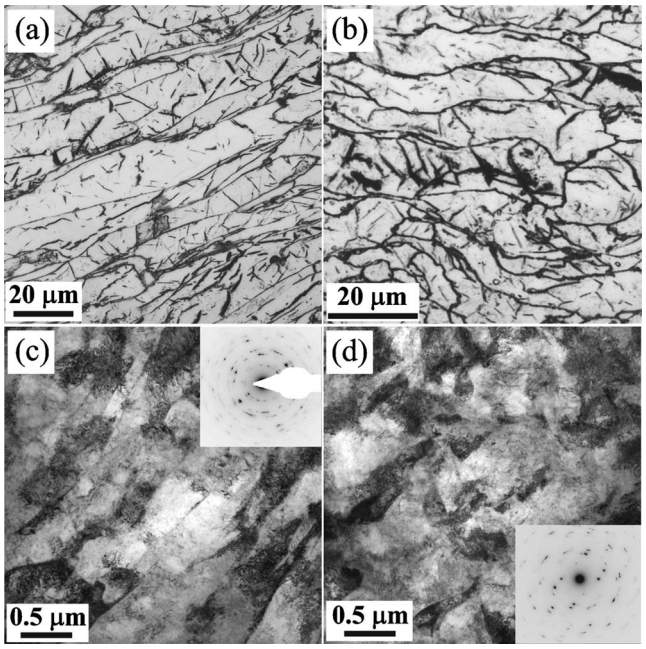

FIG. 1. [(a) and (b)] Optical micrographs in the longitudinal and cross sections, respectively. [(c) and (d)] TEM bright-field images in the cross section and corresponding SAD patterns (the insets) of the BP-ECA consolidated CP-Ti.

the less sharp domain boundaries had characteristics of low-angle grain boundaries.

In contrast, although elongated particles were also observed in the longitudinal section of DH-Ti [Fig. 2(a)], these particles had a finer interparticle spacing and a shorter particle length owing to smaller powder size. Most of the particles in the cross section [Fig. 2(b)] were irregular but near equiaxed and particularly very fine particles filled in the interparticle regions of coarse ones, forming a homogeneous bimodal structure. Unlike CP-Ti, deformation twins were seldom observed in DH-Ti except in several big particles. The TEM examination [Fig. 2(c)] revealed the presence of equiaxed grains of several micrometers in size. The SAD dotted pattern implied most grains had high-angle boundaries. It is noteworthy that ultrafine grains were found to uniformly distribute mostly at the triple grain junctions [Figs. 2(c)–2(e)], reflecting a distinct difference in grain size of the DH-Ti powder. The spreading of boundary thickness extinction contours of some ultrafine grains [Fig. 2(d)] was characteristic of nonequilibrium grain boundaries and was suggested to correlate with a high level of elastic strain and lattice distortion.[1] The volume fraction of the ultrafine grains was estimated to be about 30%. Figure 2(f) shows the HRTEM image of an ultrafine grain and corresponding fast Fourier transformation (FFT) pattern from which the ultrafine grain was identified as α-Ti with the [121] zone axis.

Figure 3(a) exhibits the room-temperature compressive true stress-strain curves for DH-Ti and CP-Ti. CP-Ti showed yield strength ($\sigma_{0.2\%}$ offset) of 630 MPa and ultimate strength of 952 MPa. In contrast, remarkably enhanced strength was achieved in DH-Ti with yield strength of 1350 MPa and ultimate strength reaching 1780 MPa. Both DH-Ti and CP-Ti showed apparent compressive ductility. Fractography indicated a mixed mode of brittle and ductile fracture for DH-Ti. Although cleavage and intergranular (not shown) were predominant, very fine

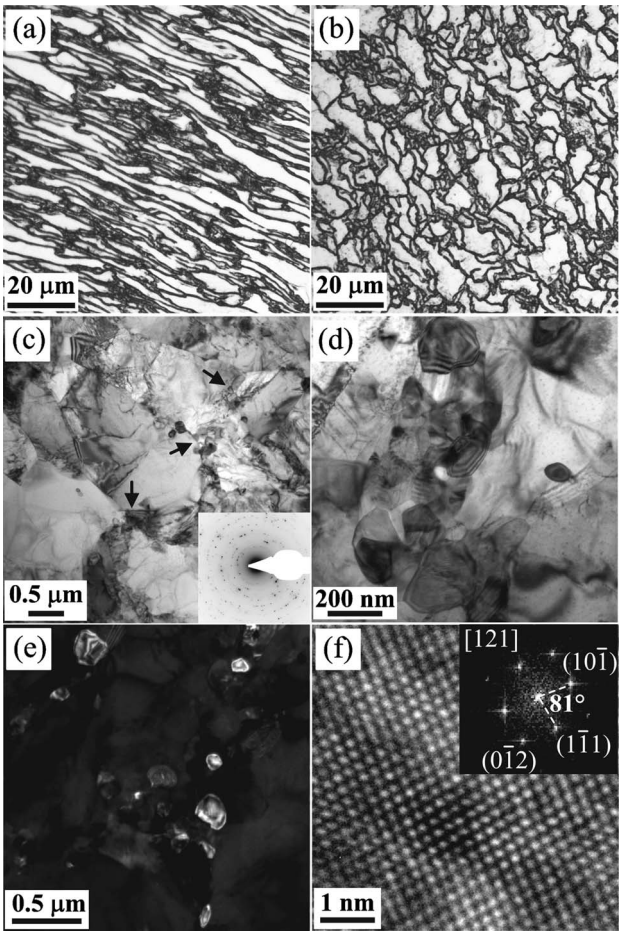

FIG. 2. [(a) and (b)] Optical micrographs in the longitudinal and cross sections, respectively. [(c) and (d)] TEM bright-field and (e) dark-field images in the cross section and corresponding SAD pattern (inset). (f) HRTEM image of one ultrafine grain and corresponding FFT pattern (inset) of the BP-ECA consolidated DH-Ti.

flutes connecting cleavage planes [Fig. 3(b)] were prominent throughout the fracture surface together with frequently observed shallow dimples [Fig. 3(c)]. The fluted fracture, considered as a result of low-energy ductile rupture, was reported to occur under mechanical overload in α-Ti of high oxygen content that hindered cross slip and promoted planar slip.[13] The presence of flutes and shallow dimples implied that localized plastic deformation happened in DH-Ti, leading to some limited ductility. In contrast, the rupture surface of CP-Ti showed both cleavages and elongated dimples (not shown) without visible fluted areas. HV values were measured to be 3207±137 MPa for CP-Ti and 5129±216 MPa for DH-Ti, in agreement with yield strengths.

Incorporating interstitials in the lattice of Ti may result in a substantial increase in strength but a drastic reduction in ductility.[14] Interstitially dissolved oxygen of 0.8 wt. % or nitrogen of 0.4 wt. % caused severe embrittlement of Ti without any sign of plastic deformation.[14] However, the ECA consolidated DH-Ti exhibited enhanced strength and hardness and at the same time retained a certain degree of ductility in the presence of very high interstitial content. Based on the correlation between Vickers hardness and equivalent oxygen content,[14] the hardness of DH-Ti of 1.34 wt. % O and 0.3 wt. % N was estimated to be 4872 MPa, slightly lower than the measured one. It suggests that strength enhancement is largely attributable to interstitial solid solution

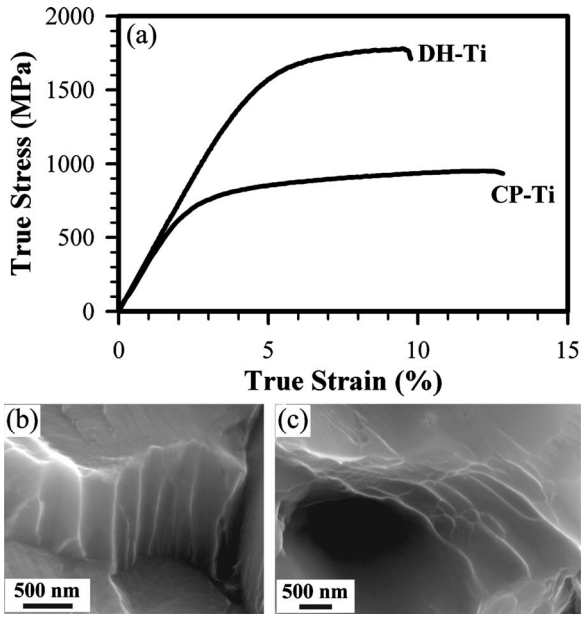

FIG. 3. (a) Room-temperature compressive true stress-strain curves for the BP-ECA consolidated DH-Ti and CP-Ti. [(b) and (c)] fracture surfaces showing flutes and shallow dimples, respectively, in the BP-ECA consolidated DH-Ti.

strengthening although the ultrafine grains also contribute. The attained ductility, on the other hand, was probably due to the nonequilibrium grain boundaries introduced by SPD as well as the bimodal grain structure. It was suggested that these grain boundaries were in a high-energy nonequilibrium configuration comprising an excess of dislocations and other defects.[1,3] Grain boundary sliding was proposed to be the possible deformation mode in SPD processed UFG/NS materials,[15,16] resulting in a certain level of ductility even in some intrinsically brittle intermetallics.[1] In the present DH-Ti, grain boundary sliding might be influenced by two factors. Grain boundaries may act as potential sinks for interstitials and a large amount of interstitials accumulating at grain boundaries were found to retard grain boundary sliding.[17] On the other hand, high-density dislocations near those nonequilibrium grain boundaries were suggested to significantly facilitate grain boundary sliding[15] to produce certain ductility. With respect to the attained ductility, it seems that the second factor predominates. However, the actual deformation mechanisms are likely to be complicated and experimental verifications of grain boundary sliding are imperative.

In summary, a dehydrided Ti powder of very high oxygen content (1.15 wt. %) was consolidated using BP-ECA processing into a fully dense bulk UFG-Ti with a bimodal grain structure, showing significantly enhanced strength together with apparent ductility in the presence of high oxygen (1.34 wt. %) and nitrogen (0.3 wt. %). The strength enhancement was mainly derived from interstitial solid solution strengthening although ultrafine grains also contributed. The ductility attained was likely to originate from the nonequilibrium grain boundary configuration and the bimodal grain structure. The good combination of high strength and improved ductility makes BP-ECA processing very promising in producing low-cost Ti and its alloys of high interstitial content.

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