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## Deformation and fracture behavior of commercially pure titanium with gradient nano-to-micron-grained surface layer

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Abstract: Titanium with gradient nano-to-micron scale grains from surface to matrix was fabricated by surface mechanical grinding treatment (SMGT) at room temperature. The SMGT-treated titanium shows higher strength than that of as-received one, but moderate ductility between those of ultra-fine grained (UFG) and coarse-grained titanium. Tensile stress-strain curves of SMGT-treated titanium show double strain hardening regimes. The strain hardening rate  $(d\sigma/d\varepsilon)$  decreases with increasing strain in tensile deformation. The high strain hardening rate at initial yielding is attributed to nano-to-micron-grained surface layer. The low strain hardening rate at large plastic strain regime primarily results from coarse-grained matrix. The SMGT-treated titanium shows a ductile fracture mode with a large number of dimples. The small size of dimples in the treated surface layer is due to the combination of the high strength and strain hardening exponent. The difference between dimple size in nano-to-micron-grained surface layer and coarse-grained matrix is discussed in terms of plastic zone size at the tip of crack in the SMGT-treated titanium.

Key words: surface mechanical grinding treatment; commercially pure titanium; gradient nano-to-micron grain; strain hardening; dimple

#### **1** Introduction

Grain refinement to submicron and nanometer scales can significantly enhance the strength of metals by introducing a great number of grain boundaries and crystal defects [1-10]. However, the uniform plastic deformation capability is dramatically restricted in the nano-grained (NG) and ultra-fine grained (UFG) metals [11–15]. Consequently, the brittleness, which has been attributed to the absence of strain hardening, is displayed since tiny NG/UFG grains have very low dislocation storage efficiency [16,17]. Dislocation slip in NG and UFG metals is more severely restricted than the coarse-grained ones due to the following two reasons. Firstly, nanoscale grains suppress dislocation slip but facilitate grain boundary sliding. However, the amount of plastic deformation in the latter is not large enough to accommodate the large plastic strain. Secondly, high density of dislocations or lattice distortions due to severe plastic deformation becomes strong barriers to dislocation slip. As a result, strain localization (necking instability) quickly occurs after yielding and little

uniform elongation exhibits during tension. The brittleness of nano-grained and ultrafine-grained metals prepared by severe plastic deformation (SPD), which significantly restricts their wide application, has been considered to be intrinsic property [4].

WANG et al [18] proposed that a proper population of coarse grains embedded in fine grains could substantially enhance the strain hardening capacity and ductility of NG/UFG metals. The metals with bimodal grain size distribution can be expected to have an excellent combination of strength and ductility. Strain hardening induced by inhomogeneous microstructures can stabilize the tensile plastic deformation and result in a large tensile ductility (~65%) and uniform elongation (~30%) at a slight cost of yielding stress. The similar results were also obtained by RAJU et al [19] in Cu-Ag alloys. On the other hand, LU et al [20-22] found that NG copper film with a spatial gradient grains in a bulk coarse-grained (CG) copper exhibited superior mechanical properties as well. They proposed that coarse-grained substrate could suppress strain localization of nano-grained surface layer. Consequently, nano-grained surface layer, which has high strength,

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shows a comparable ductility with coarse-grained matrix of copper. The formation of nano-to-micron-grained surface layer in the severe deformation process was also discussed in detail [23,24]. The tensile properties of pure titanium after surface mechanical grinding treatment have been reported to be higher than those of coarsegrained titanium [25]. However, more experimental results are needed to reveal the effect of gradient surface nano-to-micron-structured laver on the deformation and fracture behavior of pure titanium, and the cooperation of nano-grained surface and coarsegrained matrix in strain hardening of titanium with such inhomogeneous microstructure. The objective of this work is to ascertain mechanical properties, strain hardening mechanism and fracture character on the severe deformed surface and undeformed matrix so as to obtain a better understanding on mechanical behavior of metals with nano-to-micron-grained surface layer.

#### 2 Experimental

Commercially pure titanium bars (TA2) with 16 mm in diameter and 100 mm in length were chosen. The bars were annealed at 973 K for 1 h to obtain an average grain size of  $20-30 \ \mu\text{m}$ . The annealed bars were mechanically machined to dog-bone shaped tensile bars with 30 mm in gauge length and 6 mm in diameter. The surface mechanical grinding treatment processing, which was reported by FANG et al [22], was performed at room temperature on titanium with the sample rotation speed of 600 r/min on a lathe. The penetration depth of one pass is 50 µm. The treating pass is an important parameter for formation of nano-to-micron scale grains in surface layer. Ten passes are found to be the maximum pass because treatment with more than 10 passes causes surface damage. And 5 passes are taken as a middle parameter to show the effect of passes on the formation of surface layer and the properties as well. The deformed microstructures were observed with an optical microscopy (OM) and a JEM-200CX transmission electron microscope (TEM). Tensile tests were carried out on the INSTRON1195 electron-tensile tester at a displacement rate of 1 mm/min. Fracture surfaces were observed with a Hitachi S-2700 type scanning electron microscope (SEM). Microhardness at different depths was measured using the Tukon-R2100B microhardness tester with a load of 20 g and the holding time of 10 s. The distance between two measuring points is around 30 µm.

### **3 Results**

### 3.1 Microstructural characterization of SMGTtreated titanium

Figure 1 shows the microstructures from surface to the center on the cross section of a SMGT-treated

titanium (titanium bars with surface mechanical grinding treatment). Figure 1(a) shows a typical gradient NG-UFG-CG microstructure. The original grain boundaries are obscure and hardly identified due to severe deformation in the surface layer with depth of 200 µm. Beyond 200 µm from surface, deformed grains with mechanical twins were observed. The effect of surface mechanical grinding treatment becomes little beyond 300 µm depth from surface. TEM images show the details of twins, subgrains or dislocation cells in deformed area at depth from 20 to 110 µm from the surface, as shown in Figs. 1(b)-(e). Twins were observed in the surface layer, as shown in Fig. 1(b) (110 µm from surface). On a close examination, subgrain embryo was formed inside twins, as indicated by an arrow of A in Fig. 1(b). Some twins were broken due to severe strain, as indicated by circle in Fig. 1(b). However, twins were seldom observed in NG and UFG areas closer to surface layer, as shown in Figs. 1(c)-(e). The elongated subgrains formed in this area, as indicated by arrow B in Fig. 1(c). Selected-area electron diffraction analysis showed nano-to-micron-grains in treated surface layer (Fig. 1(d)). The equiaxed nanograins and submicron grains were observed apparently. In the depth of 20 µm, the lower dislocation density and nano-sized grains with random crystallographic orientations were observed, due to dynamic recovery and dynamic recrystallization in the outmost layer as reported in Refs. [26-28]. Consequently, inhomogeneous microstructure in SMGT-treated titanium bar can be divided into four regions from surface to center: NG region, UFG region, strain-affected region and strain-free matrix.

The results show that the treating passes have apparent effect on the thickness of deformed surface layer. The thickness is about 320 and 150  $\mu$ m for samples with 10 and 5 passes, respectively. In outmost surface layer, the deformation is observed much severer in the sample with 10 passes than 5 passes, as seen in Fig. 2.

Based on the measurement of a number of TEM images, statistic distribution of grain (subgrain or cell) size at depths of 20, 50, 80  $\mu$ m from surface is shown in Figs. 3(a)–(c), respectively. Grain size variation is observed in the range from 20 to 320 nm in the treated surface layer. Grain size with the highest frequency decreases from 160–180 to 60–70 nm with depth from 80 to 50  $\mu$ m. The average grain size is about 100 nm in the layer of 20  $\mu$ m under the surface, and variation of average grain size versus depth from the surface is shown in Fig. 3(d). It reveals a gradient nano-submicron-structured surface layer with thickness of ~320  $\mu$ m, while the original coarse grains with size of 20–30  $\mu$ m remain beyond ~320  $\mu$ m to the center of the SMGT-treated titanium bars.

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