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Mechanical behavior of ultrafine-grained/nanocrystalline titanium synthesized by mechanical milling plus consolidation: Experiments, modeling and simulation

Jian Liu [a,*], Akhtar S. Khan [a], Laszlo Takacs [b], Christopher S. Meredith [a]

[a] Department of Mechanical Engineering, University of Maryland, Baltimore County, Baltimore, MD 21250, USA
[b] Department of Physics, University of Maryland, Baltimore County, Baltimore, MD 21250, USA

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High quality bulk ultrafine-grained/nanocrystalline titanium samples were prepared through room temperature mechanical milling and conventional consolidation processes. The prepared bulk samples showed high purity, very low porosity and high ductility under compression. The dependency of yield stress and post-yielding behavior on grain size, strain rate and temperature were comprehensively studied. The texture evolution of the ufg/nc samples under compression was measured by synchrotron XRD. On the macroscopic scale, the viscoplastic phenomenological Khan–Liang–Farrokh (KLF) model was used to correlate the experimental results of the ufg/nc Ti.

1. Introduction

Titanium and titanium alloys are used in many applications, ranging from aerospace engineering to biomedical technology. Unalloyed commercially pure (CP) Ti has advantages such as corrosion resistance, inertness and biocompatibility over commonly used Ti alloy. However, the use of CP Ti has been limited due to its relatively low strength. One of the approaches available for improving the strength of CP Ti is the refinement of the grain size. According the Hall–Petch relationship, decreasing grain size leads to increasing yield strength. Numerous experimental investigations have demonstrated that the strength and hardness of metals with ultra-fine grains (ufg, 100 nm < grain size < 1 μm) or nanocrystalline grains (nc, grain size < 100 nm) are significantly enhanced over those of their coarse-grained (cg) counterparts.

Since its introduction into modern science as a major field by Gleiter (1989), ufg/nc materials have been the subject of widespread research (Jia et al., 2001; Meyers et al., 2006; Valiev et al., 2008; Khan et al., 2008a, 2008b; Barai and Weng, 2009; Farrokh and Khan, 2009; Meredith and Khan, 2012; Feng et al., 2013; Kumar et al., 2013; Okamoto et al., 2014; Ivanisenko et al., 2014). Many methods have been developed to synthesize materials with ufg or nc grain size, including inert gas condensation, electro-deposition, crystallization from amorphous material, severe plastic deformation and mechanical milling (including cryomilling) (Meyers et al., 2006).

Although each method has its advantages and disadvantages, SPD and mechanical milling stand out as versatile techniques to synthesize ufg/nc materials. They are both capable of producing ufg/nc structures in almost any type of material and in large quantities. They also require relatively inexpensive equipment.

* Corresponding author.

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1.1. ufg/nc Ti in literature

Most reported studies on the preparation of ufg/nc pure Ti employed severe plastic deformation (SPD) techniques. SPD processes can refine the grain size without introducing porosity or extra impurities by subjecting the bulk samples to exceptionally high strains.

The Equal Channel Angular Pressing (ECAP) is the most promising and well-studied SPD technique. The advantage of ECAP is its capability to maintain the net dimensions of the workpiece. Based on the experimental data (Zhernakov et al., 2001; Stolyarov et al., 2001; Tabachnikova et al., 2005; Ko et al., 2006; Shpeĭzman et al., 2007; Korshunov et al., 2008; Hyun et al., 2010; Meredith and Khan, 2012), it can be concluded that after multiple passes of ECAP at different conditions, the grain size can be decreased to 200–300 nm which leads to a 1.2- to 2.6-fold increase of the yield stress and tensile ultimate strength. The tensile ductility after ECAP was in the 10–20% range, which was sufficient for structural application. The value of the enhanced yield and ultimate strength depends not only on the processing parameters but also on the purity of the material. The yield strength of ECAPed pure Ti was in the range of 450–800 MPa whereas the ultimate strength was in the range of 660–820 MPa. By comparison, the strength of ECAPed pure Ti was lower or barely equal to that of Ti–6Al–4V alloy. This limitation of strength enhancement correlates closely with the limited grain size refining capability of the ECAP process.

In order to further increase the strength, a large number of investigations have applied additional cold working or SPD processes after the ECAP processing on CP Ti (Jia et al., 2001; Stolyarov et al., 2003; Zhu et al., 2003; Sadikova et al., 2005; Latysh et al., 2006; Salimgareeva et al., 2006; Semenova et al., 2008; Valiev et al., 2008). These processes can further refine the grain size/microstructure and/or introduce more dislocations to improve the strength. Strengths above 1 GPa can be obtained, which are higher than the strength of common Ti–6Al–4V alloys.

Other SPD techniques such as high-pressure torsion (HPT) (Popov et al., 1997; Sergueeva et al., 2001; Edalati et al., 2009), accumulative roll-bonding (ARB) (Terada et al., 2007), hydrostatic extrusion (HE) (Garbacz et al., 2006; Pachla et al., 2008; Topolski et al., 2010), and cryorolling (Moskalenko et al., 2009) were also used (without ECAP) to refine the grain size and enhance the strength. A combination of high strength and sufficient ductility was achieved in those cases. However, unlike ECAP, the other SPD techniques, used either by themselves or in combination with ECAP, do not preserve the dimensions of the workpiece. One or more dimensions of the workpiece are continuously reduced, which eventually reduces the workpiece to a final geometry of foil/plate or small diameter rod/wire, limiting their structural application.

Although there were several studies on the ball milling of Ti powder and the characterization of the milled powder (Wang et al., 1993; Panigrahi and Godkhindi, 2006; Dabhade et al., 2001; Dabhade et al., 2006; Sun et al., 2006a, 2006b), very few researchers produced bulk ufg/nc Ti samples using ball-milling plus consolidation. Among those, only the research group of Enrique J. Lavernia (Ertorer et al., 2008, 2009, 2010) obtained bulk fully dense samples of large enough sizes for experimental measurements of the plastic behavior. They used cryomilling as the milling process and hot quasi-isostatic forging (QIF) (Ertorer et al., 2008, 2009) or spark plasma sintering (SPS) (Ertorer et al., 2010) as the consolidation method. These techniques require more expensive equipment and are harder to scale-up to industrial applications compared to the room temperature milling and conventional hot compaction used in this study. Also, to maintain the ductility, they intentionally mixed the milled nano Ti powder with un-milled powder to achieve a bimodal grain size distribution for most of their samples. As a result, the yield strength and ultimate tensile strength are only comparable to those obtainable by ECAP and are lower than those obtained by other SPD techniques.

In summary, ECAP could refine the grain size of Ti without reducing the dimensions of the samples. However, due to the limitation of grain size refinement (200–300 nm) by ECAP, the increase of strength of Ti is limited (below 1 GPa). Other SPD techniques could further refine the grain size and/or the microstructure and increase the strength (above 1 GPa). However, one or more dimensions of the workpiece are continuously reduced, limiting their structural applications.

In the rather limited literature on ufg/nc Ti by mechanically milling and consolidation, no researchers have used room temperature milling and conventional hot compaction to produce fully dense bulk samples. Also, mechanical milling's ability of grain size refinement (into nano range, below 100 nm) and strengthening (above 1 GPa) are not fully exploited. This study fills these voids in the existing research.

1.2. Deformation mechanisms of Ti

Unalloyed commercially pure (CP) titanium is a HCP metal with a c/a ratio of 1.588 which is lower than the ideal value of 1.633. For Ti, the easiest deformation mode (mechanism) is prismatic slip which means prismatic slip will be the first to be activated when the random textured material enters plastic deformation. With further straining and increasing stress, other deformation modes such as basal slip, pyramidal $\langle a \rangle$ slip, pyramidal $\langle c+a \rangle$ slip, tensile and compressive twinning could also be activated. Especially when there is a strain component along the c-axis of a grain, at least one deformation mode—the pyramidal $\langle c+a \rangle$ slip and/or twining—needs to be active to accommodate it. Although the prismatic slip is the easiest to activate, the plastic deformation of Ti is the result of the competition among prismatic, basal, pyramidal $\langle a \rangle$, pyramidal $\langle c+a \rangle$ slip and twinning. At the grain level, the relative contribution from each deformation mode is determined by the relative strength of each slip/twining system and the orientation of the grains with respect to the loading direction.

Generally, the strength of each deformation mode is affected by the temperature, strain rate, solute concentration, grain size, etc. An increase of solute concentration and a decrease of grain size are expected in Ti processed by mechanical milling