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The tensile properties and fracture behavior of gradient nano-grained/coarse-grained zirconium

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ABSTRACT

The tensile properties and fracture behavior of gradient nano-grained/coarse-grained zirconium produced by surface circulation rolling treatment were investigated. Experimental results indicated that gradient nano-grained zirconium foil exhibits high yield strength (~748 MPa) and reduced tensile elongation (~3.0%) relative to its coarse-grained counterpart. On the contrary, the gradient nano-grained/coarse-grained zirconium shows good mechanical properties which combine the strengthening from nanocrystalline zirconium with the strain hardening provided by coarse-grained zirconium. Localized plastic deformation is effectively suppressed, instead of a uniform and fish-scale overall deformation.

1. Introduction

Nanocrystalline materials exhibit many novel properties such as high hardness and strength, enhanced corrosion and fatigue resistance, as well as excellent tribological properties relative to their conventional coarse-grained (CG) counterparts [1–4]. Due to various limitations of present synthesis techniques, preparation of ideal bulk nano-grained (NG) samples (free of contamination and porosity, bulk in size, uniform and small in grain size) is still a challenge to material scientists [5]. In fact, most failures of materials occurring on surfaces (fatigue fracture, fretting fatigue, wear and corrosion etc.) are very sensitive to surface structure and properties, and the surface optimization may effectively enhance the global performances of materials. The overall properties and behaviors of materials can be greatly improved by generating a nanostructured layer on material surfaces. This kind of surface modification, referred as surface nanocrystallization, provides a novel approach to meet specific structure/property requirements on material surfaces without changing chemical composition [5–7].

With the in-depth investigation of surface nanocrystallization, various advanced processing techniques, e.g., surface mechanical attrition treatments [5,7,8], surface mechanical grinding treatment [9,10], have been developed for obtaining a gradient NG (GNG) layer on Fe, Ti, Zr and Cu. Investigations on mechanical properties show that the GNG materials exhibit enhanced strength–ductility [7,10]. However, little is known for the deformation and fracture behavior of the GNG structures. In this study, we chose Zr as a model metal and succeeded in introducing a GNG/CG structure, yielding a simultaneous enhancement of strength and ductility. The tensile properties and fracture behavior of GNG/CG Zr are investigated in details.

2. Experimental

A commercially pure Zr plate with a fully recrystallized microstructure was subjected to surface circulation rolling treatment (SCRT) to prepare a GNG Zr foil on CG Zr substrate. The details of this technique have been described elsewhere [11]. Microstructure features of surface layer were characterized by using a JEOL-2010 transmission electron microscopy (TEM) at a voltage of 200 kV. The tensile samples of GNG foil were prepared as follows. Firstly, the top surface about 5 μm thick was removed to eliminate surface roughness effect. Dog-bone-shaped tensile specimens were cut from top surface layer (about 100 μm thick) by using electro-discharging. Finally, they were mechanically polished from untreated side until the thickness was about 80 μm. The final tensile sample geometry has a gauge length of 6 mm, a cross-sectional area of $3 \times 0.08\ mm^2$. The tensile specimens of GNG/CG samples were prepared substantially the same as above. The final tensile sample geometry has a gauge length of 10 mm, a cross-sectional area of $3 \times 0.3\ mm^2$. At least four tensile tests were performed by an Instron-5848 micro-tester with a video non-contact extensometer at a strain rate of $10^{-3}\ s^{-1}$ at room temperature. For comparison, four CG tensile samples with the same geometry as GNG/CG Zr were also tested under the same conditions. The fracture surfaces and side flat faces after tensile tests

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were characterized by employing a HITACHI S-4800 scanning electron microscopy (SEM).

3. Results and discussion

In our previous investigation, the microstructural characterizations of Zr surface layer were systematically investigated. TEM measurements showed that the average grain sizes are less than 100 nm in the top 80-μm-thick layer; meanwhile, a GNG/CG structure is achieved with average grain sizes varying from about 8 nm in the topmost surface to micrometers at about 300 μm [11].

Tensile engineering stress–strain curves of GNG foil, GNG/CG Zr and CG Zr are shown in Fig. 1. For GNG foil, the yield strength (0.2% offset, $\sigma_{0.2}$) and ultimate tensile strength (σ_{UTS}) are 748 MPa and 856 MPa, respectively. Meanwhile, it shows a low ductility with a total plastic strain less than 3.0%. On the contrary, $\sigma_{0.2}$ and σ_{UTS} of GNG/CG Zr increase by about 68% and 49% respectively as compared with its CG counterpart together with acceptable ductility (the uniform elongation and elongation to fracture of GNG/CG Zr are about 64% and 82% of its CG counterpart). The comparison of tensile properties with previous studies on pure Zr indicates that an inverted relationship between strength and ductility in metal Zr is clearly observed (see the shaded area in Fig. 2). However, the data for GNG/CG Zr made in this study is far away from the shade, indicating a combination of high strength and good ductility.

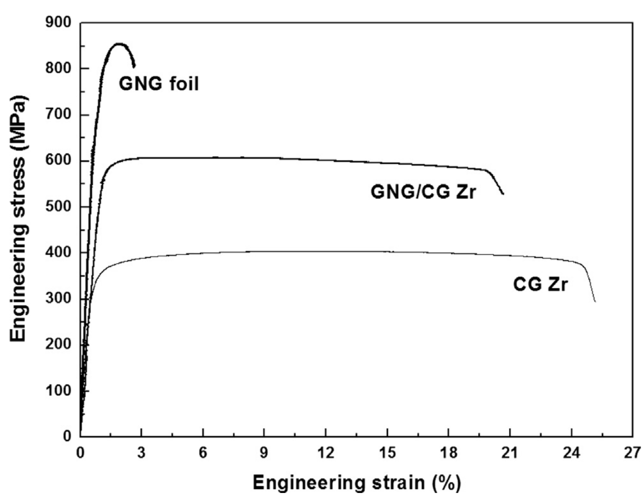

Fig. 1. Tensile engineering stress–strain curves of GNG foil, GNG/CG Zr and CG Zr.

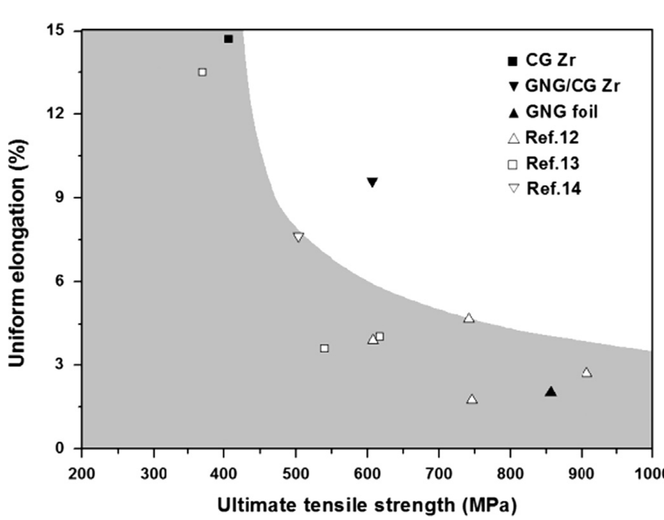

Fig. 2. Representative tensile properties of pure Zr. Hollow symbols are reported data [12–14]. Solid symbols are obtained from corresponding curves in Fig. 1.

The GNG foil exhibits some strain hardening during the plastic deformation prior to fracture. The strain hardening exponent (n) for samples can be derived by fitting the equation $\sigma = K\varepsilon^n$ to the uniform plastic deformation section of the true stress–strain curve. The n values for GNG foil, GNG/CG Zr and CG Zr are 0.012, 0.045 and 0.088, respectively. The GNG foil exhibits a strong-and-brittle tensile behavior. However, the GNG/CG Zr shows enhanced strength while maintaining sufficient strain hardening capacity as compared with GNG foil, which may result from that nanograins provide high strength while coarse-grains supply the strain hardening capability that prevents localized deformation and premature fracture [7].

Fracture surface morphologies of GNG foil, GNG/CG Zr and CG Zr are shown in Fig. 3. The GNG foil and GNG/CG Zr show clear ductile features. The mean dimple size for GNG foil is about 1–2 μm (Fig. 3a), and it is also obvious that there exist some large dimples of 6–10 μm which may be caused by coalescence of some small dimples during straining. The GNG/CG Zr shows a gradient structured dimple size which varies from 2 μm to 10 μm from treated surface to matrix (Fig. 3b). As a contrast, the CG Zr displays quasi-cleavage fracture with big cleavage facets and a few dimples (Fig. 3c).

SEM images of side flat faces of the fractured samples are shown in Fig. 4. Within the narrow region close to the fracture surface of GNG foil (Fig. 4a), there are many localized plastic deformation zones (black arrow) and microcracks (white arrow). The corresponding enlarged view of the labeled region in Fig. 4a displays a noticeable plastic zone (Fig. 4a′). Within this plastic zone, deformation bands (arrows) can be clearly seen. The deformation band scale is much larger than grain size, which indicates that the localized deformation extends over many grains across grain boundaries. The shear localization has also been observed in other nanocrystalline metals [15,16]. In contrast to the brittle fracture of GNG foil, the GNG/CG Zr exhibits a uniform and fish-scale overall plastic deformation (Fig. 4b). The cracked GNG surface layer deforms coherently with CG substrate after necking without any delamination. Meanwhile, we can clearly see that there is a significant plastic deformation indicated by arrows in Fig. 4b′ underneath the cracked GNG surface layer. In comparison, the side flat face of CG Zr appears many line-shaped deformation markings, which are about 0.5–1.0 μm in width and tens of micrometers in length (Fig. 4c and c′). These line markings may be shear bands representing localized plastic deformation [17].

The GNG/CG Zr exhibits significantly different fracture behavior in comparison to GNG foil and its CG counterpart. The localized plastic deformation is effectively suppressed. The distinguishing fracture behavior can be described as follows. At the initial stage of tension, the GNG surface layer deforms coherently with CG substrate. When the applied stress exceeds the yield strength of CG substrate but lower than that of GNG layer, microvoids and microcracks will emerge on the joint of CG substrate and GNG layer. Further straining results in more cracks and stress concentration at the crack tip. As a result, the fracture strength of GNG layer is exceeded, and cracks propagate toward the GNG side across the specimen. With continuous straining, the cracks propagate forward by connecting the adjacent microcracks, and hence leaving fish-scale morphology on the side flat face of the fractured sample. Subsequently, the CG substrate deforms with the fish-scale GNG layer until the sample is eventually torn apart when the fracture strength of material is exceeded. A similar phenomenon was observed in a 316 stainless steel. The analysis of fracture mechanism revealed that the nanostructured surface layer can block slip bands developed in CG matrix. So crack nucleation will occur in the subsurface layer due to the enhanced NG layer [7].