

Improvement of the fatigue performance of an ultrafine-grained Nb–Zr alloy by nano-sized precipitates formed by internal oxidation

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The formation of nano-sized precipitates in an ultrafine-grained Nb–Zr alloy was investigated. ZrO₂ precipitates induced by internal oxidation during heat treatment at low homologous temperatures significantly intensify the hardness of the surface layer without deterioration of the surface quality, and thus significantly improve the fatigue performance.

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Recently, refractory elements have proven successful in various applications with a range of loading conditions, mainly due to their very high melting temperatures. Niobium (Nb) falls into this group of elements, and is characterized by its high ductility, low yield strength and, consequently, good formability. It has been demonstrated that solid solution strengthening by alloying with zirconium (Zr) brings about a well-balanced combination of material properties [1], broadening the scope of the utility of Nb. The Nb–Zr alloy has been widely studied in order to optimize its properties for space nuclear power systems [2–5], yet it is also emerging as the new material of choice in other areas, such as biomedicine. Good corrosion properties of pure titanium (Ti), Zr and Nb along with their proven biocompatibility and osteogenesis [6,7] promote their use in biomedical implants, however, their low strength necessitates alloying prior to their use in application. Ti is mostly strengthened by adding vanadium and aluminum, which, however, have been shown to release ions that act as toxic reagents in animals' bodies [7,8], further strengthening the position of Nb–Zr as an alternative to Ti alloys in biomedical applications. This, in turn, warrants a deeper understanding of the role of pro-

cessing-induced microstructures on the mechanical properties of this alloy.

The current study was undertaken with this motivation, and a Nb–Zr alloy was subjected to equal channel angular extrusion (ECAE), an effective method for obtaining an optimum combination of high strength and ductility [9–11], and improved fatigue performance [12–14]. The Nb–Zr was received in its coarse-grained (CG) condition in the form of a rolled plate with a nominal composition of 1.0 wt.% Zr, 0.02 wt.% O, 0.006 wt.% N, 0.0003 wt.% H, 0.0035 wt.% Fe, 0.006 wt.% C, 0.06 wt.% Ta and balance Nb. Billets with a 25 × 25 mm² cross-section were extruded 16 times at room temperature following the ECAE route E at a rate of 2.5 mm s⁻¹ through a die with a sharp 90° angle [13]. ECAE route E is a hybrid route that can be described by 2C × 2C, where the × stands for an intermediate 90° rotation. This route was used as it brings about a homogeneous microstructure with excellent mechanical properties and a very high product yield [12,13]. One-hour heat treatments within the temperature range of 525–800 °C were conducted on samples with the dimensions of 8 × 5 × 1 mm³ extracted from the initial plate and the ECAE-processed billets, in order to investigate the effect of heat treatment on the mechanical response. The heat-treated samples were air-cooled prior to mechanical grinding down to 5 μm grid size for the removal of a thin

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non-adhesive Nb-oxide layer on the surface. Afterwards, hardness measurements were carried out using a Vickers microhardness tester with a load of 0.098 N (HV0.01). The effect of the heat treatment on the microstructure was observed with the aid of scanning electron microscopy (SEM) and transmission electron microscopy (TEM). For the latter investigations, the samples were taken from the surface layer, either by the focused ion beam technique or by standard electrochemical preparation (CG sample, see Ref. [13] for details).

The hardness experiments revealed that a 1 h heat treatment of the ultrafine-grained (UFG) Nb–Zr alloy at 600 °C, which corresponds to a homologous temperature of 0.32 for this alloy, brings about nearly a four-fold increase in the hardness of the surface layer (Fig. 1). The cross-section of the fatigued sample displays two separate regions. The 80- μm -thick surface layer reflects the characteristics of brittle fracture, while the traces of ductile fracture are evident in the core of the heat-treated sample (Fig. 1). The core of the heat-treated UFG Nb–Zr remains unchanged with a hardness of 175 HV0.01 throughout the cross-section, while the hardness at a distance of 10 μm to the surface of the heat-treated sample attains a value of about 650 HV0.01. Initially, the surface layer clearly suffered damage due to heat treatment at an elevated temperature, as evidenced by a rough structure (not shown), which poses a risk factor under cyclic deformation. The formation of pore-like features is attributed to the possible presence of an eutectic composition on the surface, but needs to be investigated intensively in the future. A systematic heat treatment study within the temperature range of 525–800 °C revealed that decreasing the heat treatment temperature to 560 °C and increasing the heat treatment

time to 6 h could eliminate the pores without significantly altering the hardness.

This significantly hard surface layer is indeed desired for certain applications that demand higher durability under wear, and it could be obtained by diffusing nitrogen [15] or carbon into the surface layer, or by forming precipitates. The latter can be achieved by internal oxidation to the extent permitted by the alloying system. Specifically, alloys, such as copper–beryllium, Nb–Zr, and Nb–hafnium, feature one element highly reactive with oxygen embedded in the matrix consisting of the other element with much lower reactivity with oxygen [16–18]. The current UFG Nb–Zr alloy exhibits a significantly large density of dislocations and grain boundaries in its as-ECAE-processed state, which leads to the hardness increase in the samples' interior (Figs. 1 and 2). This microstructure does not affect the diffusion rate of oxygen in Nb due to the bulk diffusion process in the Nb–Zr system, as evidenced by the similar depths of hardened surface layers (Fig. 1) of CG and UFG Nb–Zr. Consequently, it can be concluded that there is no significant contribution of short-circuit diffusion to the hardness in the UFG microstructure in the investigated system. The higher hardness values in the surface layer of the UFG alloy as compared with its CG counterpart indicate the presence of smaller precipitates, which in fact can be attributed to the higher number of nucleation sites in the highly distorted microstructure. The error bar in Figure 1 is representative of the error of individual hardness measurements carried out in the interior of the sample. However, owing to the steep hardness gradient in the near-surface layer, small variations in surface distance result in a substantial scatter of the hardness data. Thus, hardness profiles were furnished more than once in each case, and the data shown represent the averages of at least four individual profiles. For the same reason, data obtained at a distance <10 μm to the surface are not included, as scatter becomes unacceptable. In fact, the maximum hardness measured directly on the surface of the specimens could reach 950 HV0.01.

Consequently, oxygen reacting with Zr forms ZrO_2 nano-sized precipitates with a size <10 nm in the UFG

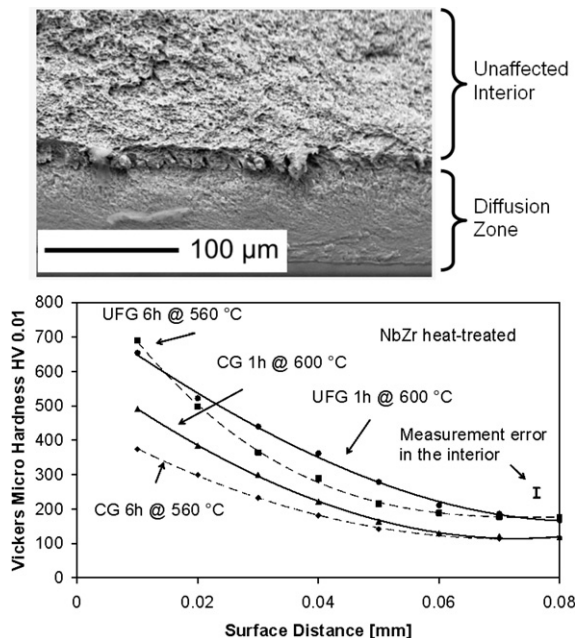


Figure 1. Hardness measurements carried out on various CG Nb–Zr and UFG Nb–Zr specimens. The SEM image displays part of the fracture surface of a fatigued UFG Nb–Zr specimen (1 h/600 °C). See text for details.

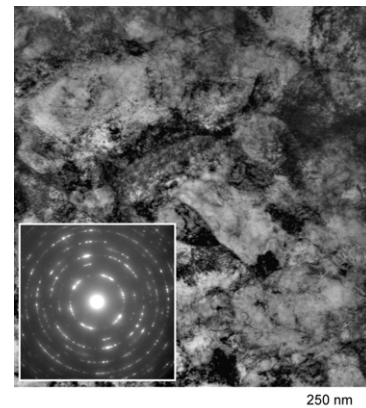


Figure 2. A bright-field TEM image and the corresponding SAD pattern of the route-16E UFG Nb–Zr microstructure prior to heat treatment [13].

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