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Irradiation toughening in a hierarchical structured alloy

Defeng Guo [a,b], Xiaohong Li [a,b], Ming Li [a], Yindong Shi [a], Guosheng Zhang [a], Kiminori Sato [c], Zhengjun Zhang [d], Xiangyi Zhang [a,*]

[a] State Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao 066004, People's Republic of China
[b] College of Science, Yanshan University, Qinhuangdao 066004, People's Republic of China
[c] Department of Environmental Sciences, Tokyo Gakugei University, Tokyo 184-8501, Japan
[d] Department of Materials Science and Engineering, Advanced Materials Laboratory, Tsinghua University, Beijing 100084, People's Republic of China

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ABSTRACT

The irradiation of high-energy particles always leads to the embrittlement of metallic materials. Here, we report an abnormal irradiation toughening phenomenon in a hierarchical structured ZrTi, which results from the irradiation-induced lattice relaxation that yields an enhanced ductility with a slight decrease of strength. Positron annihilation measurements reveal that the lattice relaxation dominantly originates from a recombination of irradiation-induced interstitials (i.e., Ti atoms) with preexisting dislocation-associated vacancies. This study provides an approach to suppress the irradiation-induced brittleness in metallic materials.

Metallic materials with high strength and good ductility are critical for keeping the safe, reliable and economic operation of nuclear plants, which provide about 13% of the world's supply of electricity today and may be the most feasible way to replace the polluting fossil fuels in the future. However, the irradiation of high-energy particles always leads to the embrittlement of materials – an enhanced strength with considerable decrease of ductility – because the irradiation-induced lattice defects, such as interstitials, vacancies and aggregated defect clusters, hinder the motion of dislocations in the materials [1]. To suppress the irradiation embrittlement, the strategy of using nanostructured materials has been proposed, due to their high-density grain boundaries and phase interfaces that act as sinks to point defects [2,3]. However, the application of nanostructured materials was limited by their poor ductility [2,4]. Different from conventional structural materials with microstructure features in a single scale, hierarchical structured (HS) materials with microstructure features in different scales (e.g., nanometer and micrometer scales) show a good combination of high strength and large ductility [4]. Moreover, these materials have high-density grain boundaries and phase interfaces, which may act as the sinks to irradiation-induced defects and thus have a potential of inhibiting the irradiation-induced embrittlement [2,5]. This may offer an opportunity to overcome the irradiation embrittlement in engineering materials. In this letter, we report an abnormal irradiation toughening phenomenon in a HS-ZrTi, and the physical mechanism governing irradiation toughening has been revealed using positron annihilation measurement techniques.

To produce HS-ZrTi materials, the deformed ZrTi (51.1Zr–40.2 Ti–4.5Al–4.2V wt.%) sheets with a strain of $\varepsilon = 2.3$ were subjected to recrystallization annealing (675 °C for 10 min) and subsequent two-step aging treatments (625 °C for 2 h + 300 °C for 3 h). Experiment details were given in Ref. [6]. The HS-ZrTi was then suffered from electron irradiation with a dose of 10^{16}–10^{17} cm^{-2} on the rolling plane using a 10 MeV electrostatic accelerator under ambient atmosphere. The temperature of the samples was kept below 100 °C during the irradiation processes using a water-cooling specimen holder. For comparison, the HS-ZrTi was also annealed at 250 °C for 2 h in a vacuum furnace to study the effect of temperature on structure and mechanical properties.

Uniaxial tensile tests of the HS-ZrTi were performed to measure the mechanical properties before and after irradiation. A transmission electron microscope (TEM) and an X-ray diffractometer (XRD) were used to characterize the microstructure of the HS-ZrTi. The positron lifetime spectrum and the coincident Doppler broadening (CDB) of the positron-electron annihilation photons were recorded to determine atomic detects and their chemical environments in the HS-ZrTi before and after electron irradiation. The details of

* Corresponding author.
E-mail address: xyzh66@ysu.edu.cn (X. Zhang).

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the positron annihilation experiments were given in our previous studies [7,8].

As-prepared HS-ZrTi has a lot of α and α'' lamellae (indicated with the symbols α and the arrows) with multimodal width distributions from several to three hundreds nanometers (see Fig. 1a and b). The phase composition of these lamellae was determined by selected area electron diffraction (SAED) analyses [6]. Many residual dislocations (indicated with the triangles in Fig. 1a) can be observed in coarse α lamellae and the residual β phase between the α lamellae.

Fig. 1c presents the tensile curves of HS-ZrTi before and after electron irradiation with different doses. The as-prepared HS-ZrTi shows an ultimate tensile strength of σ_b = 1580 MPa and an elongation to failure of ε_f = 7.0% (Curve A). Strikingly, after irradiation with a dose of 10^{16} and 10^{17} cm^{-2}, the ε_f increases to 8.1% and 9.8% while the σ_b decreases slightly to 1519 and 1466 MPa (see curves B and C), respectively, which demonstrates an irradiation-induced toughening in the HS-ZrTi. The annealed HS-ZrTi gives a stress–strain curve (curve D) similar to that of the as-prepared one, ambiguously excluding the possibility that the observed radiation toughening phenomenon was caused by thermal effect during the electron irradiation process (<100 °C).

Fig. 1d presents XRD patterns of as-prepared, irradiated and annealed HS-ZrTi. After electron irradiation, the XRD peaks (indicated with ER) are narrower than those of the as-prepared and annealed sample (indicated with As and An, respectively), indicating an irradiation-induced lattice relaxation. Similar phenomenon has also been observed in proton-irradiated 304 and 316 L stainless steels and ion-irradiated tungsten [9–11]. The lattice relaxation will decrease the stress to start and move dislocations, contributing to ductility enhancement [12].

To reveal the physical mechanism governing the irradiation-induced toughening, we employed positron annihilation measurement technique (Fig. 2). In as-prepared HS-ZrTi, positron lifetime spectrum can be fitted by two lifetime components, $\tau_1 = 126 \pm 3$ and $\tau_2 = 194 \pm 4$ ps with a relative intensity of $I_1 \sim 58\%$ and $I_2 \sim 42\%$ (Fig. 2b and c), respectively. With increasing irradiation dose to 10^{17} cm^{-2}, I_1 increases to ~71% and I_2 decreases to 0%, respectively. At the same time, a new component appears with a long positron lifetime of $\tau_3 = 305 \pm 8$ ps and a small relative intensity of $I_3 \sim 10\%$ (Fig. 2c).

The lifetime components τ_1 and τ_2 result from positrons annihilation in free delocalized (τ_f) and vacancy-trapped (τ_v) states, respectively, which are deduced from the relationship between positron lifetime (i.e., τ_f and τ_v) and valence electron density (ρ_{el}) in solid materials (Fig. 2d) [13,14]. However, the value of lifetime τ_2 is obviously below that of τ_v yielded from the fitting curve for lattice vacancies. This indicates that the lifetime component τ_2 likely results from the annihilation of positrons at *dislocation-associated* (DA) vacancies, where a short lifetime is predicted by molecular dynamics calculations [15]. The existence of DA vacancies with a short positron lifetime as compared with the value of τ_v has also been confirmed in previous studies of deformed Fe [16,17] and Ni [18]. In the HS-ZrTi, the electron irradiation reduces the amount of the DA vacancies significantly, which is indicated by the decrease of the relative intensity I_2. This may contribute to the lattice relaxation observed in XRD studies (see Fig. 1d). The lifetime component τ_3 likely results from the annihilation of positrons at

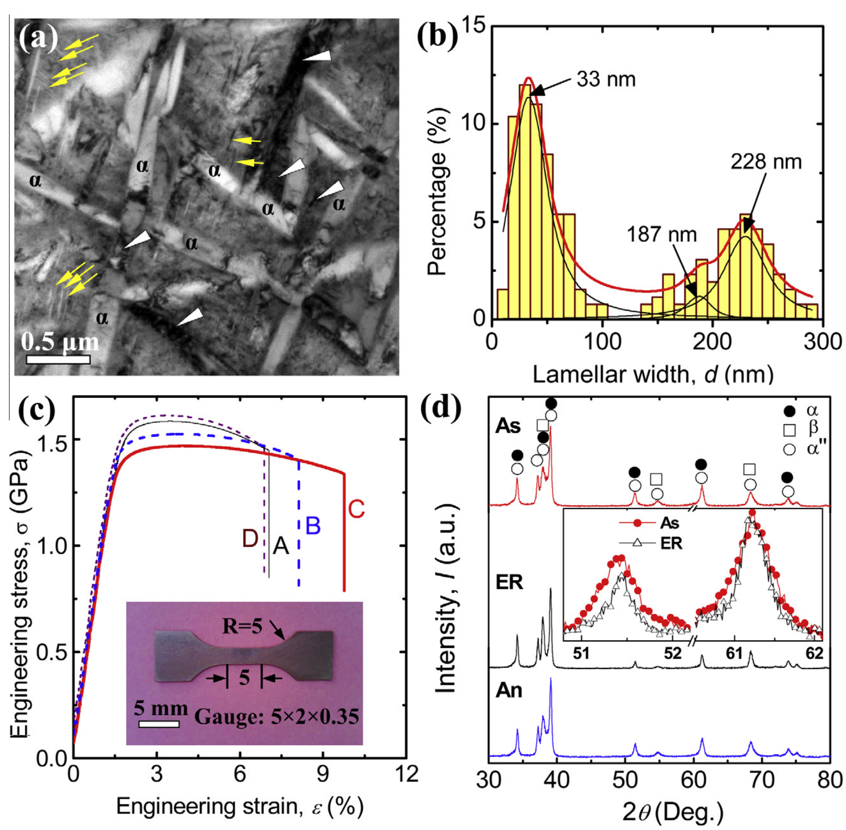

Fig. 1. (a) TEM image and (b) lamellar width distribution of as-prepared hierarchical structured (HS) ZrTi. The α lamellae, α'' lamellae and residual dislocations in (a) are indicated with the symbols α, arrows and triangles, respectively. Figure (c) shows engineering tensile stress–strain curves of the as-prepared (A), annealed (D) and irradiated HS-ZrTi with a dose of 10^{16} (B) and 10^{17} cm^{-2} (C), and the inset is the tensile specimen geometry. Figure (d) presents XRD patterns of the as-prepared (As), irradiated (ER) (with a dose of 10^{17} cm^{-2}) and annealed (AN) (250 °C) HS-ZrTi. The inset is the enlarged XRD patterns.