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Simultaneously enhancing the ductility and strength of cryorolled Zr via tailoring dislocation configurations

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ABSTRACT

Dislocation configurations, e.g. dislocation tangles, cells and lamellas, in pure Zr have been tailored by cryorolling in the strain rate range of $\dot{\varepsilon}=0.59–2.24\ s^{-1}$, demonstrating a significant effect on mechanical properties. A simultaneous enhancement in the ductility and strength is obtained in the Zr cryorolled at $\dot{\varepsilon}=2.24\ s^{-1}$. The enhancement of ductility is due to the motion of preexisting dislocations under high tensile stress in dislocation lamella configurations yielded at the high strain rate, and the enhancement of strength results from the increase of dislocation density in the cryorolled Zr.

1. Introduction

Plasticity in metals occurs by the motion of dislocations, which multiply in the course of plastic deformation. The accumulation and interaction of dislocations create obstacles that make the propagation of dislocations difficult, leading to enhanced strength but decreased ductility, i.e. work hardening [1–3]. A material may be strong or ductile, but rarely both simultaneously. Recent studies on mechanical behavior of nanostructured metals [4–7] show that preexisting dislocations in some specific configurations can move under external stress and contribute to plastic deformation of the metals, hinting that dislocation configurations may have a significant effect on mechanical properties of materials. Therefore, tailoring dislocation configuration may be an alternative approach to increase the plasticity of deformed metals while keeping their high strength. For this goal, rolling at liquid nitrogen temperature, i.e. cryorolling, is a potential technique due to its high storage of dislocations by the suppression of dynamic recovery in deformed materials [8,9]. Previous studies show that higher strain rate leads to higher dislocation storage in cryogenically deformed metals [10–12], which provides a great room for producing various dislocation configurations. In the present study, we succeeded in controlling dislocation configurations in cryorolled Zr by employing various strain rates and observing a simultaneous increase in the ductility and strength in the deformed metal.

2. Experimental details

Fully recrystallized pure Zr (99.95%) sheets were cryorolled in the strain rate range of $\dot{\varepsilon}=0.59–2.24\ s^{-1}$ from 3.0 to 0.25 mm in thickness, i.e. an accumulated strain of $\varepsilon=2.87$, with a reduction of ~2% per pass. The cryorolling was performed by immersing the samples into liquid nitrogen for 10 min before and after each rolling pass, and the liquid nitrogen was sprayed on the surface of rollers and samples. The rolling temperature was determined to be −160 to −90 °C before and after each rolling pass by using an adiabatic calorimeter [9].

Transmission electron microscopy (TEM) specimens were prepared via twin-jet electrochemical polishing in a solution containing 10% perchloric acid and 90% acetic acid at 25 V and about 18 °C. Microstructures of samples in rolling plane were characterized by using a JEM-2010 microscope and all selected area electron diffraction (SAED) patterns in the present study were made by employing the same selected area aperture and exposure time. X-ray diffraction (XRD) spectra were recorded from the rolled surface using a Rigaku D/MAX-2500 X-ray diffractometer with Cu Kα radiation. The step-scanning mode with a step of 0.02° was used and the time spent on per step was 4 s. According to the Variance method [13,14], available for the calculation of dislocation density in hcp-structured metals [15,16], the fourth order

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moment of the peak profile, $M_4(q)$, is related to average dislocation density $\langle\rho\rangle$

$$\frac{M_4(q)}{q^2} = \frac{1}{3\pi^2\varepsilon_F}q + \frac{\Lambda\langle\rho\rangle}{4\pi^2} + \frac{3\Lambda^2\langle\rho^2\rangle}{4\pi^2 q^2}\ln^2(q/q_1) \qquad (1)$$

where Λ is a geometrical constant describing the strength of dislocation contrast, ε_F the average column length or area weighted grain size, $q=2/\lambda[\sin(\theta)-\sin(\theta_0)]$, where θ is the diffraction angle and θ_0 is the Bragg angle, q_1 is a fitting parameter not interpreted physically. Average dislocation density can be determined by fitting a straight line to the asymptotic part of the M_4/q^2 curve.

Uniaxial tensile tests were performed on samples with a cross-section of 2.00×0.25 mm and a gauge length of 10.0 mm by means of an Instron 5848 Micro-Tester (2 kN) at a strain rate of $1\times10^{-3}\,\mathrm{s}^{-1}$ at room temperature. The tensile direction was parallel to the rolling direction of samples.

3. Results and discussion

The broadening of (102) and (103) XRD peaks of cryorolled Zr increases with increasing strain rate (see Fig. 1), and a similar trend is also observed on other peaks, e.g. (002) and (110). The dislocation density in the cryorolled Zr was estimated by using the Variance method, $\langle\rho\rangle\ \sim1.6\times10^{15}$, 3.6×10^{15} and $4.8\times10^{15}\,\mathrm{m}^{-2}$ for $\dot{\varepsilon}=0.59$, 1.89 and 2.24 s^{-1}, respectively. This indicates that the dislocation density of deformed Zr increases with strain rate, which can be attributed to the fact that the generation of dislocations is promoted and their dynamic recovery is suppressed at high strain rate [11,12].

TEM observations (Fig. 2) present the evolution of dislocation configurations in cryorolled Zr at various strain rates. At $\dot{\varepsilon}=0.59\,\mathrm{s}^{-1}$ (Fig. 2a), there are some dislocation tangles (indicated by white arrows) and dislocation cells (DCs) (indicated by black triangles) in the deformed sample. The formation of a lot of DCs demonstrates the occurrence of dynamic recovery during the plastic deformation at low strain rate, which is confirmed by obvious misorientations characterized by elongated electron diffraction (ED) spots (indicated by arrows) and discontinuous ED arcs (indicated by the triangle) in corresponding SAED patterns (Fig. 2b), similar to previous observations of DCs in severe deformed Cu [17]. Increasing $\dot{\varepsilon}$ to $1.89\,\mathrm{s}^{-1}$ (Fig. 2c), more dislocation tangles and less DCs appear, indicating an effective suppression of dynamic recovery at a high strain rate. The suppression of dynamic recovery is further confirmed by SAED studies (Fig. 2d), in which the ED spots (indicated by arrows) are less elongated and discontinuous ED arcs almost disappear as compared with those at $\dot{\varepsilon}=0.59\,\mathrm{s}^{-1}$. Moreover, some dislocations are arrayed in band structures (indicated by triangles in Fig. 2c). Further increasing $\dot{\varepsilon}$ to $2.24\,\mathrm{s}^{-1}$, more irregular dislocation tangles (Fig. 2a) change into parallel dislocation lamellas (indicated by triangles in Fig. 2e), and SAED patterns with slightly elongated spots (Fig. 2f), which can be indexed as the ED patterns of single crystal Zr, which indicate a high internal stress in grains due to the existence of high-density dislocations. This shows a strong suppression of dynamic recovery at the high strain rate. These results indicate that higher strain rate results in an ordering of dislocation configurations, e.g., dislocation lamellas in the cryorolled Zr, which may be attributed to the interactions of dislocations at different strain rates [12,17], as observed in previous studies of cryorolled Al [18].

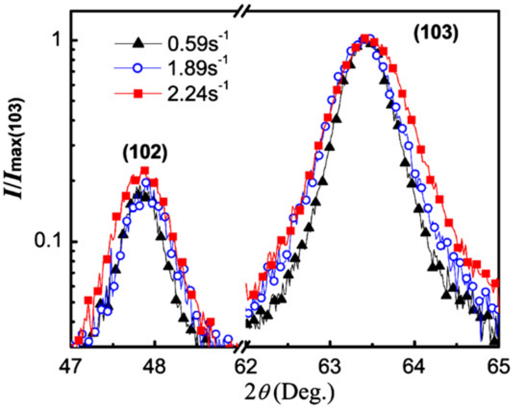

Fig. 1. XRD profiles of (102) and (103) peaks of cryorolled Zr at different strain rates.

Engineering stress–strain curves of cryorolled Zr are shown in Fig. 3, indicating an abnormal trend, i.e. the strength and ductility increase simultaneously with strain rate, as compared with the conventional work hardening. This unexpected result has been confirmed in multiple tensile tests. The variation in mechanical properties of cryorolled Zr can be attributed to the difference in dislocation density and dislocation configurations that are yielded at different strain rates. The high strength and low ductility of curve-A may result from dislocation forest strengthening and the limited motion of tangled dislocations [1,8].

To reveal the mechanism for a simultaneous increase in both strength and ductility in cryorolled Zr at high strain rate, in-depth TEM observations on the fractured tensile sample cryorolled at $\dot{\varepsilon}=2.24\,\mathrm{s}^{-1}$ have been performed (Fig. 4). The tensile true strain ε of different locations indicated with B, C, D and E in Fig. 4(a) has been estimated by the formula [19]

$$\varepsilon=\ln(d_0^2/d^2) \qquad (2)$$

where d_0 is the original thickness of tensile sample and d is the final thickness after tensile test, yielding a value of $\varepsilon=0$, 0.08, 0.14 and 0.21, respectively. At location B, parallel dislocation lamellas with dense dislocations are shown in the bright-field TEM image (Fig. 4b), and the corresponding SAED patterns with slightly elongated ED spots (inset in Fig.4b), which can be satisfactorily indexed as the ED patterns of single crystal Zr, indicate a high internal stress in the cryorolled Zr. The dark-field image made using the reflection circled in the SAED patterns (see the inset of Fig. 4b) further confirms the lamella characteristic of dislocations in the deformed sample (Fig. 4b′). At location C (Fig. 4c), most of dislocation lamellas disappear and a lot of DCs (indicated by triangles) form, which are indicated by more elongated ED spots (indicated by arrows in the inset in Fig. 4c) that show increased misorientations across DCs. At location D (Fig. 4d), there are no dislocation lamellas, and most of DCs evolve into subgrains (indicated by triangles). This is further supported by the SAED studies, in which discontinuous ED *rings* (indicated by the arrow in the inset in Fig. 4d) suggest the appearance of subgrains with low-angle boundaries because these patterns were taken using the same selected area aperture and exposure time as those used in Fig. 4(b and c) (the insets), where slightly and more elongated ED *spots* are shown respectively. At location E, many nanoscale subgrains (indicated by the triangles marked with the number 1, 2, 3 and 4) appear (Fig. 4e), which is supported by the corresponding dark-field TEM image (Fig. 4e′) of these subgrains indicated. The dark-field TEM image was made using the encircled reflections in the SAED patterns (see the inset in Fig. 4e). The formation of nanoscale subgrains is further confirmed by the continuous SAED rings (see the inset in Fig. 4e) at this location.

Above TEM observations reveal that preexisting dislocations in cryorolled Zr at $\dot{\varepsilon}=2.24\,\mathrm{s}^{-1}$ are movable under high stress *during tensile deformation*, similar to previous studies on nanostructured