



Role of shear localization in nanocrystallisation of zircaloy-2 processed by wire rolling at cryo temperature

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ABSTRACT

Shear localization during wire rolling of zircaloy-2 at cryo temperature has been investigated in the present work. Bulk nanocrystalline zircaloy-2 has been obtained by optimized annealing of cryorolled alloy. Microstructural characterization and mechanical behavior of the processed alloy have been performed by X-ray diffraction (XRD), optical microscopy, Electron backscatter diffraction (EBSD), Transmission electron microscopy (TEM) and tensile testing. The presence of (ω) phase after wire rolling has been observed from XRD. The tensile strength of deformed zircaloy-2 (true strain = 2.77) was found to be 1015 MPa with a uniform ductility of 10.8%, while loss of tensile strength (837 MPa) and increase in ductility (14.8%) after annealing at 400 °C for 30 min were observed. Nanocrystalline structure with an average grain size of 30 nm has been formed after annealing at 400 °C for 10 min. The microstructure reveals the formation of shear bands during wire rolling at cryo temperature due to large applied strain in each pass. The possible mechanism of deformation and shear band formation has been discussed. The effect of strain rate on amount of work converted to heat (β) during shear band formation has been discussed. The calculation reveals that the rise in temperature during deformation was 831.6 °K on applying a strain of 2.77, during wire rolling at cryo temperature.

1. Introduction

Zirconium and its alloys are used as cladding material in nuclear reactors due to its low neutron absorption cross section, corrosion resistance, high melting point and better mechanical properties [1–3]. Especially, zirconium alloys, such as Zr-2, Zr-4, Zr-1 Nb, Zr-2.5 Nb, ZRLO (Zr-1.0Sn-1.0Nb-0.1Fe), E635 (Zr-1.2Sn-1.0Nb-0.4Fe), M5 (Zr-1.1Nb-0.12O), NDA (Zr-1.0Sn-0.1Nb-0.27Fe-0.16Cr), PCA (Zr-0.8Sn-0.3Fe-0.2Cr), HANA (Zr-Nb-Cu), N18 (Zr-Sn-Nb), are used in the field of nuclear power reactors worldwide [4]. Extensive study on the mechanical behavior [5–9], corrosion resistance [10,11], hydrogen absorption [12–14] and radiation damage [15,16] of zirconium and its alloys has been made by various researchers. Grain refinement to ultrafine (0.1–1 μ m) and nanocrystalline (less than 100 nm) regime can further improve the mechanical and corrosion behavior of zircaloy-2 without changing the composition of the alloy. In contrast, severe plastic Deformation (SPD) is a promising technique to produce ultrafine

and nanocrystalline materials, in which the material undergoes large plastic deformation [17–19]. Rolling at cryo temperature has been an effective SPD technique to produce ultrafine and nanocrystalline material with high strength and ductility [20].

The nanocrystalline materials exhibit superior strength, high ductility, enhanced super plastic formability and superior wear resistance [21]. The unique mechanical behavior is due to different deformation mechanism in nanocrystalline materials compared with their coarse grained alloys. A cross over from intragranular dislocation based plasticity to grain boundary mediated plasticity occurs when the grain size is > 10 nm, with the mechanism of grain boundary sliding [22,23], grain rotation [24–27] and grain boundary migration [28] dominating. Therefore, the nanometer size grained materials have comparable ductility with their coarse grain counterpart.

Due to the hexagonal structure and c/a ratio less than 1.63, limited slip planes exist in zircaloy-2 for deformation [29]. At low strain, $\langle a \rangle$ prismatic slip ($\{10\bar{1}0\}\langle 1\bar{1}10 \rangle$) and ($\{10\bar{1}2\}\langle 1011 \rangle$) extension twinning is

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most active deformation mode [30]. However, pyramidal slip in the first $\{10\bar{1}1\}$ plane and second order in $\{11\bar{2}2\}$ plane along $\langle 11\bar{2}3 \rangle$ (c + a) directions is also observed at low strain. The $\langle a \rangle$ basal slip $((0002)\langle 11\bar{2}0 \rangle)$ is active at high strain and at higher temperatures [30]. Apart from $\{10\bar{1}2\}\langle 10\bar{1}1 \rangle$ extension twinning, deformation through $\{11\bar{2}1\}\langle 11\bar{2}6 \rangle$ extension twinning, and contraction twins of the type $\{11\bar{2}2\}\langle 11\bar{2}3 \rangle$ and $\{10\bar{1}1\}\langle 10\bar{1}2 \rangle$ [30] are possible.

Localized heating during plastic deformation has been first reported by Tresca (1878) during forging of iron [31]. This localized heating can cause unstable highly localized plastic flow inside the material, also known as adiabatic shear band. Generally, adiabatic shear bands are always reported to be at high strain rate ($> 10^2 \text{ s}^{-1}$) loading conditions with local plastic strain inside the shear band can be as high as $10\text{--}10^2$ ($10^5 \text{ s}^{-1}\text{--}10^7 \text{ s}^{-1}$) [32]. Even the local strain rate inside the shear band also changes and observed in Ti–6Al–4V impacted at a global strain rate of 10^4 s^{-1} found to be $8 \times 10^4 \text{ s}^{-1}$ [33]. The local temperature distribution inside the band is highly transient, with short range spatial periodicity along the direction of growth [32]. The experimental studies showed the temperature difference at initiation to be from 50°C to 350°C and at propagation from 800°C to 1700°C in case of Ti64 alloy based on the results obtained by high speed infrared cameras [34]. There are two types of shear bands observed in Ti alloy [35,36], Al alloy [37] and Mg alloy [38], classified as transformed and deformed. The deformed bands have black etching characteristics with severely elongated grain (severe strain localization) while transformed bands have ultrafine and equiaxed recrystallised grains with white etching characteristics. Although, several studies on adiabatic shear band formation has been reported in the literature, but the mechanism still remains controversial. Hence, it becomes essential to investigate the role of shear band for the formation of ultrafine grain structures in the bulk materials. Therefore, in the present work, possible deformation mechanism at low strain and mechanism of shear localization observed during wire rolling of Zircaloy-2 at cryo temperature has been investigated. The microstructure and mechanical behavior of bulk nanocrystalline zircaloy-2 were investigated using EBSD, TEM and tensile testing.

2. Experimental procedure

The material composition, microstructure and method of mercury quenching (S.T.) has been discussed in our earlier work [39]. After quenching, the material has been cut in to $4 \times 4 \times 80 \text{ mm}^3$ sample. The samples are then wire rolled in the rolling mill with 8 rpm speed and 110 mm roller diameter. The assembly is shown in Fig. 1(a–b). Prior to every rolling pass, the samples were dipped in liquid nitrogen for 10 min. Due to large strain induced in every pass, temperature of the samples increases with every pass and for avoiding the heating, the samples were immediately quenched in liquid nitrogen. Three reductions with 0.69, 1.32 and 2.77 true strains were selected to perform further characterization as shown in Fig. 1(c). A true strain of 2.77 has been given to the billet in a total 8 passes.

The wire rolled zircaloy-2 has been further characterized by transmission electron microscopy (Tecnai-20 machine at 200 kV), Electron back scatter diffraction (FEI Quanta 200 FEG-SEM/EBSD at 20 V with a step size of $0.01 \mu\text{m}$), X-Ray diffraction and tensile test to study the effect of deformation on the microstructure and mechanical properties. The tensile tests were performed using dumb bell shaped 10 mm gauge length wire, machined using electro discharge machining. Twin jet polishing at 20 V and -20°C temperature using a solution of 20:80 perchloric acid and methanol has been used for TEM sample preparation, while the same solution temperature and voltage is used for electro polishing of sample used in EBSD Analysis. XRD of the samples was carried out by Pan analytical MRD system instrument using Cu K α radiation.

3. Results and discussion

3.1. Microstructure

The microstructure and α -phase of zircaloy-2 with an average grain size of $10 \mu\text{m}$ after mercury quenching (S.T.) has been discussed in our earlier work [39]. The optical microstructure obtained after imparting true strain of 0.69, 1.32 and 2.77 is shown in Fig. 2(a–c). The black arrow shows the rolling direction. The microstructure after 0.69 strains clearly shows the elongated grains as the load is applied, the length of the sample increases. There are some grains which are not deformed completely shown by marked circles in Fig. 2(a). As reported in the literature, the harder grains are basal oriented [40]. These are undeformed prismatic grains, possibly due to different loading in wire rolling, as confirmed by the EBSD maps. Upon further increasing the reduction with 1.32 true strain, the grains get further elongated as seen in Fig. 2(b). It is also noticeable that the whitish area has decreased. The grains get further elongated, when the strain reaches to 2.77 and almost appears like metallic threads or lamellas. Dark regions can also be observed between thread with $0.5\text{--}2 \mu\text{m}$ width. Also, the temperature of the wire during wire rolling at cryo temperature, reaches to nearly 100°C after every pass. These results are clear indication of shear localization also observed in TEM studies discussed later. During the processing, the 80 mm long billet is transformed in to 1.28 m long wire with $1 \times 1 \text{ mm}^2$ cross sectional area. The XRD pattern in Fig. 3 shows the presence of α phase as well as (ω) phase after deformation at cryo temperature [41]. It has been reported that the phase transformation of zirconium takes place when the compressive load reaches above 2 GPa, however depending on shear strain and texture [41].

3.2. EBSD

The elongated grains with large misorientation in the microstructure (Fig. 4(a)) after applying 0.69 true strain along with tensile twins, which is also marked by circle in the misorientation profile, can be seen from Fig. 4(b). kernel average misorientation map showing large dislocation density can be seen from Fig. 4(c). The heterogeneity is also observed from the grain profile shown in Fig. 4(d). Due to wire rolling at cryo temperature, grains are elongated with misorientations inside and low fraction of $\{10\bar{1}2\}$ twins are observed. There are some grain divisions between 15° to 25° boundary misorientation within the grains. These subdivisions are formed due to shear localization aligned to the rolling direction. In literature, these are defined as micro bands, which are formed due to alignment of dislocation cell structure [42]. However, they have not recorded any microband to cross through several grains [42]. The bands formed in the present work are investigated further by magnifying some sections as marked by square box in image 4(a). The sections are referred as p, q, r and s as marked in Fig. 4(a). On magnifying the section p, localized shearing can be seen with an angle of 33° intersecting two grains. On plotting the line misorientation (black line) of the band, the misorientation (Fig. 5(p1)) between the band and grain is less than 20° . The section contains parallel bands of this type having misorientations inside them. Other sections (Fig. 5(q, r and s)) are also magnified showing almost same misorientation ($15^\circ\text{--}25^\circ$) behavior (Fig. 5 (q1, r1 and s1)). The angles of the shearing planes (white lines) are different, may be due to grain rotation because of repeated rolling passes.

Twins $\{10\bar{1}2\}$ type are also observed in Fig. 5(s), and are responsible for reduction in grain size and hardening. Mostly, complete twins are observed in sections where grain fragmentation took place as marked by square in Fig. 4(a). Since, $\{10\bar{1}2\}$ twin orients the crystal from prism to basal plane, independent of temperature; it is the easiest deformation system in zirconium crystal [30]. Also, basal is the hardest orientation in the zirconium [40]. The twin causes maximum strain hardening during deformation. However, in the present case, twinning is not observed as much as reported by various researchers in earlier studies

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