Materials and Design 55 (2014) 612–618

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Mechanical and microstructural characterizations of ultrafine grained Zircaloy-2 produced by room temperature rolling

Sunkulp Goel [a], R. Jayaganthan [a,*], I.V. Singh [b], D. Srivastava [c], G.K. Dey [c], N. Saibaba [d]

[a] Department of Metallurgical and Materials Engineering & Centre of Nanotechnology, IIT Roorkee, Roorkee 247667, India
[b] Department of Mechanical and Industrial Engineering, IIT Roorkee, Roorkee 247667, India
[c] Materials Science Division, Bhabha Atomic Research Center, Mumbai 40085, India
[d] Nuclear Fuel Complex Limited, Hyderabad 501301, India

ARTICLE INFO

Article history:
Received 14 July 2013
Accepted 16 September 2013
Available online 4 October 2013

Keywords:
Rolling
Zircaloy-2
Characterizations
Nanograins

ABSTRACT

The effect of deformation strain at room temperature on the microstructural and mechanical properties of Zircaloy-2 was investigated in the present work. The sample was initially heat treated at 800 °C in argon environment and quenched in mercury prior to rolling. The deformed alloys were characterized by using EBSD and TEM. It reveals the misorientation of incidental grain boundaries (IDBs) due to large plastic strain induced in the sample. The recovery of deformed alloy upon annealing leads to the formation of ultrafine and nanostructured grains in the alloy. The hardness achieved after 85% room temperature rolling (RTR) is found to be 269 HV, while the tensile strength is 679 MPa and 697 MPa in the rolling and transverse direction, respectively. The improvement in strength is due to generation of high dislocation density and ultrafine grains in the deformed alloy with 85% thickness reduction, during rolling. The deformed alloy subjected to annealing at 400 °C for 30 min sample shows increase in ductility (6% and 7.2%) in rolling and transverse direction, respectively, due to the annihilation of dislocations as evident from the TEM study.

1. Introduction

Zircaloy-2 is used as pressure tubes in power reactors and as structural material in light water reactors (LWR) [1]. Better corrosion resistance, mechanical properties and low neutron absorption cross section makes it favorable for its application in nuclear reactors [2,3]. In Zircaloy-2, tin, iron, chromium and nickel are used as alloying elements with their concentration less than 2% [4]. They provide high corrosion resistance in steam and hot water environment [5]. It exhibits two phase i.e. α phase (hexagonal closed pack (HCP)) structure, which is stable below 1080 K, while the β phase is body centered cubic (BCC) stable above 1250 K [6]. γ is the intermetallic phase generally known as secondary phase precipitates (SPP) observed in this alloy [7]. $Zr_2(Ni, Fe)$ with body center tetragonal structure [8,9] and $Zr(Fe, Cr)_2$ with hexagonal structure are the two types of SPP with size less than a micrometer were observed in Zircaloy-2 [10]. These precipitates are hard intermetallic particles and the formation of hydrides in Zircaloy-2 makes it brittle due to which generally cracks initiate from this brittle hydride phase [11,12]. To avoid formation of hydride, Zircaloy-2 is heat treated in argon environment at 800 °C and quenched in mercury as reported in the literature [13].

Very limited slip systems are active in the Zircaloy-2 due to is hcp α phase with c/a ratio less than 1.633. Twinning is active in Zircaloy-2 and the twinning modes are of two types such as tensile in $\{10\bar{1}2\}\langle\bar{1}011\rangle$ and $\{11\bar{2}1\}\langle\overline{11}26\rangle$ and compressive in $\{11\bar{2}2\}\langle\overline{11}23\rangle$ and $\{10\bar{1}1\}\langle\bar{1}012\rangle$ (at high temperature) [14]. It causes large orientation of lattices and thus rotating basal planes from the pole. Basal position from 0° to 50° from the normal direction indicates $\{11\bar{2}2\}$ twinning gets activated. During compressive loading, basal poles rotate by 64° from the center of the pole. $\{10\bar{1}2\}$ twinning gets activated when 50–90° orientation towards normal direction takes place and $\{11\bar{2}1\}$ twinning is operable, when the basal pole tilt from 0–90° occurs. Thus, twinning orients the basal pole $\{0002\}$ parallel to deformation direction [15].

During rolling, the Zircaloy-2 sheet undergoes compressive load in normal direction, while friction forces in the rolling direction due to which it elongates more in the rolling direction. There is small compressive force exerted in transverse direction due to which less elongation occurs in the transverse direction. The basal pole $\{0002\}$ prefers to align parallel to the compressive direction (normal direction) and spread 20–40° towards the transverse direction because of the small compressive force in the transverse direction [15]. Rolling was performed by quenching Zircaloy-2 at β temperature with Widmanstätten type of structure and process annealed during rolling [16,17]. Normally, the rolling temperature

---

* Corresponding author. Tel.: +91 133 2285869; fax: +91 133 2285243.
E-mail address: rjayafmt@iitr.ernet.in (R. Jayaganthan).

0261-3069/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.matdes.2013.09.039

Table 1
Chemical composition of Zircaloy-2.

Element	Sn	Fe	Cr	Ni	N
Wt.%	1.3–1.6	0.07–0.20	0.05–0.16	0.03–0.08	.006

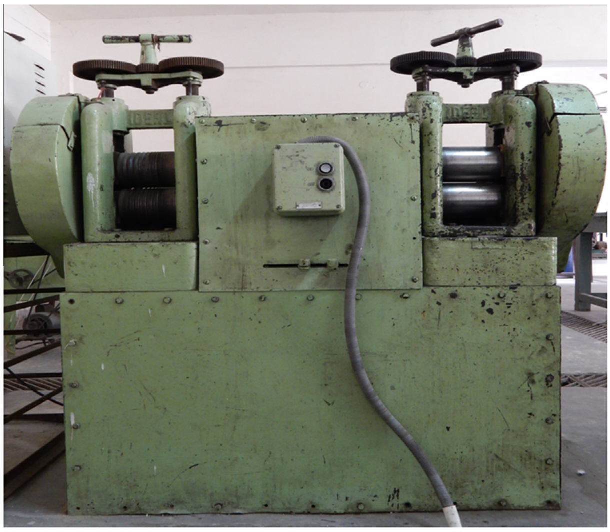

Fig. 1. Experimental setup used for RTR.

varied from 400 °C to 700 °C as reported in the literature [18,19]. Rolling reduction up to a maximum of 70% at room temperature was obtained by annealing Zircaloy-2 in a seal quartz tube for 10 h at 750 °C and cooling with a rate of 5 °C per minute. The dislocation density was investigated using Williamson Hall technique and it was found to be of the order of $10^{-15}/\mathrm{m}^2$ [20]. Zr subjected to cryorolling up to 2.87 strain followed by annealing exhibit a multimodal structure of nano and ultrafine grains [21,22]. The tensile properties and ductility of cryorolled Zr were investigated and the mechanisms governing the improvement in strength and ductility of the deformed alloy were substantiated [23,24]. The improvement in strength of cryorolled Zircaloy-2 up to 70% thickness reduction has also been reported [25].

It is well known fact that grain size influences physical and mechanical response of a structural material. Development of a material with desired properties include its grain size control and grain boundary engineering through optimized thermo mechanical processing conditions [26]. Ultrafine and nanocrystalline materials provide high strength, super plasticity at elevated temperature and good fracture strength properties [27]. These materials are produced by severe plastic deformation techniques (SPD) such as ECAP, Multiaxial forging and ARB [28,29]. The formation of

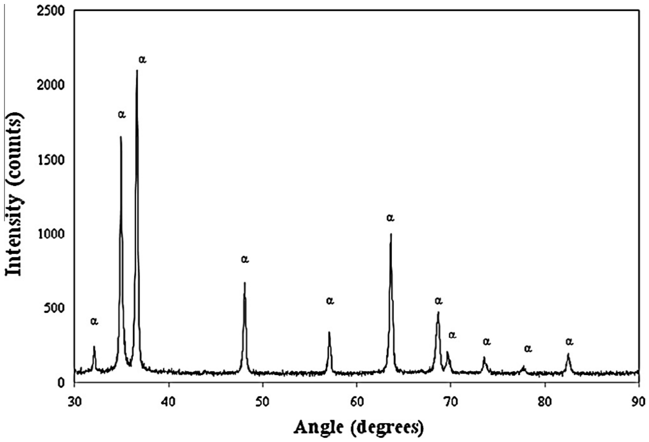

Fig. 3. X-ray diffraction (XRD) analysis of the 800 °C mercury quenched sample.

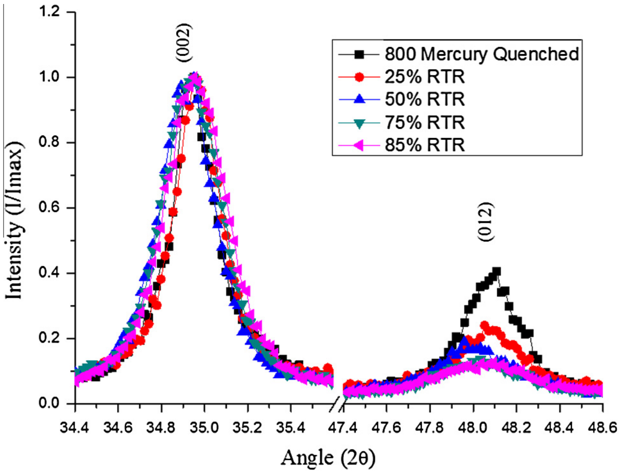

Fig. 4. XRD peaks of mercury quenched 25% RTR, 50% RTR, 75% RTR and 85% RTR Zircaloy-2. Showing the effect of rolling on the broadening of (002) and (012) peaks.

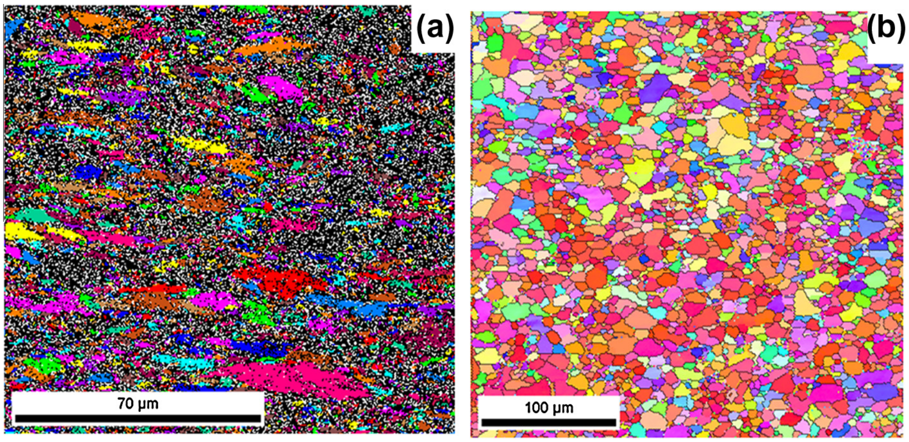

Fig. 2. (a and b) Showing the EBSD image microstructure of as received and 800 °C mercury quenched Zircaloy-2.