Microstructural characteristics of cold-rolled Zr-2.5Nb alloy annealed near the monotectoid temperature

CHAI LinJiang\textsuperscript{1}, WU Hao\textsuperscript{1}, WANG ShuYan\textsuperscript{1}, LUAN BaiFeng\textsuperscript{2*}, WU Yue\textsuperscript{1} & HUANG XiaoYu\textsuperscript{1}

\textsuperscript{1} College of Materials Science and Engineering, Chongqing University of Technology, Chongqing 400054, China; \textsuperscript{2} College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China

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A dual-phase Zr-2.5Nb alloy was rolled at room temperature to 50\% reduction and then annealed at two temperatures (560 and 580\degree C) near the monotectoid temperature. X-ray diffraction, electron channeling contrast imaging and electron backscatter diffraction techniques were jointly used to characterize microstructural characteristics developed in the as-rolled and annealed specimens. Results show that plastic deformation occurs in both bulk \(\alpha\)-Zr grains and thin \(\beta\)-Zr films during rolling, allowing large lattice strains to be accumulated in \(\beta\)-Zr and active dislocation slip (especially the prismatic \(\alpha\) slip) to be initiated in \(\alpha\)-Zr. During subsequent annealing at 580\degree C, the prior \(\beta\)-Zr films are transformed into submicron \(\beta\)-Zr particles, which lose coherency (the Burgers orientation relationship) with surrounding \(\alpha\) grains. In the specimen annealed at 560\degree C, however, the prior \(\beta\)-Zr films are found to be decomposed into nanoscale \(\beta\)-Nb particles. In both the annealed specimens, the \(\beta\)-Zr and the \(\beta\)-Nb particles appeared to be linearly distributed along the rolling direction. Two types of \(\alpha\) structures, i.e., small equiaxed crystallites formed by recovery of dislocation structures and coarse bamboo-like recrystallized grains, are revealed in the annealed specimens. Effective boundary pinning due to the dense \(\beta\)-phase particles is demonstrated to play a key role in forming such unusual bamboo-like grains.

\textbf{Zr alloy, dual-phase microstructure, rolling, recrystallization, electron backscatter diffraction}

\section{Introduction}

Thanks to many attractive properties like low neutron absorption, good corrosion resistance and biocompatibility, Zr alloys have been intensively used in nuclear, chemical and biomedical industries [1–3]. Among commercially available ones, most of them can be categorized as single-phase and dual-phase alloys in terms of the presence or not of metastable \(\beta\)-Zr after typical fabrications. The former includes a series of Zr alloys such as Zircaloy-2/4 and Zr-702 [4–7], while Zr-2.5Nb alloy stands as a representative of the latter [8–11]. For all these materials, before any products (for example pressure tubes in heavy water reactors) are finally manufactured, multiple rolling/drawing and annealing are usually employed. Control of microstructures produced by such thermo-mechanical procedures is found to be important for enhancing performance of the Zr alloy products. Thus, numerous attempts have been stimulated to understand how their microstructures evolved during deformation and annealing (e.g. [12–14]). However, related efforts have to date been mainly limited to the single-phase Zr alloys due possibly to their microstructural simplicity. In spite of equal (if not higher) importance, understanding on deformed microstructures and recovery/recrystallization behaviors of the dual-phase Zr alloys like Zr-2.5Nb remains rather limited [8,15]. In fact, accompanied with recovery/recrystallization, monotonous or unusual microstructures are demonstrated.
process during annealing, the metastable $\beta$-Zr presented in dual-phase Zr alloys could be decomposed under specific conditions [16]. This means that more complicated recovery/recrystallization may occur, in comparison to the case in single-phase alloys.

In the present work, therefore, careful microstructural characterization was conducted for a Zr-2.5Nb alloy subjected to rolling and annealing treatments, with the objective to well reveal deformation and recovery/recrystallization features of the dual-phase Zr alloy. The decomposition of the metastable $\beta$-Zr in Zr-2.5Nb alloy requires the annealing temperature to be lower than its monotectoid temperature that slightly varies with impurities contained. A recent work [17] suggested the monotectoid temperature of commercial grade Zr-xNb alloys to be \(~585^\circ\text{C}\), which was referenced to select annealing temperatures (560 and 580°C) in this research. Results documented and discussed in this study are believed to be able to facilitate better understanding on major microstructural characteristics of dual-phase Zr alloys processed with critical parameters (near the monotectoid temperature).

2 Experimental procedure

The as-received material was a Zr-2.5Nb alloy that was hot-forged in the $\alpha<\beta$ region. A sheet with the dimension of 60 mm×20 mm×3 mm in rolling, transverse and normal directions (denoted as RD, TD and ND, respectively) was cut from the as-received material and subjected to rolling at room temperature with a reduction of 50% in the ND. Specimens with the dimension of 11 mm×9 mm×1.5 mm in RD, TD and ND were then machined from the as-rolled sheet. Annealings at 560°C for 50 h and 580°C for 20 h were then conducted using a box furnace for the as-rolled specimens after they were sealed into quartz tubes under vacuum to avoid oxidation during the heat treatment. The annealed specimens were hereinafter named after their annealing temperatures.

Phase constituents in various specimens were first examined by use of a DX-2500 X-ray diffractometer (XRD) using Cu Kα. Their microstructures were then well characterized by electron channeling contrast (ECC) imaging technique in a Zeiss Sigma HD field emission gun scanning electron microscope (FEGSEM). The backscattered electron-based ECC technique has been demonstrated to enable crystallographic orientation-based structures to be clearly imaged [18]. Subsequently, an automated electron backscatter diffraction (EBSD) analysis system attached to the FEGSEM was utilized to acquire a large amount of quantitative orientation data corresponding to specific microstructures. The EBSD system was composed of a NordlysMax2 detector (Oxford Instruments) with AZtec 2.4 and HKL Channel 5 software packages used for data acquisition and post-processing, respectively. Before these examinations, the to-be-analyzed surfaces (RD-TD plane for XRD and RD-ND plane for ECC and EBSD) were mechanically ground and then electro-polished in a mixture of 70 mL methanol, 20 mL butyl cellosolve and 10 mL perchloric acid at \(-30^\circ\text{C}\) and 20 V for 30–60 s.

3 Results

3.1 XRD pattern

Figure 1 presents XRD patterns of various specimens with different peaks indexed. In all specimens, $\alpha$-Zr with a hexagonal close packed (hcp) structure is found to be the major phase. In spite of greatly reduced intensities, a few peaks corresponding to $\beta$-Zr with a body centered cubic (bcc) structure can be noticed for the as-received specimen, clearly suggesting a dual-phase structure. After 50% rolling, widths of all diffraction peaks are evidently broadened, which could be attributed to large lattice strains induced by the plastic deformation [19]. Such lattice strains may be accumulated severer for the $\beta$-Zr since only faint intensities are left for their peaks. The $\beta$-Zr peaks are re-intensified after the as-rolled specimen is annealed at 580°C for 20 h due probably to relaxation of the lattice strains. This also suggests that $\beta$-Zr is not decomposed at all but well retained during and after the 580°C annealing. For the specimen annealed at 560°C for 50 h, however, the $\beta$-Zr peaks seem to completely disappear. Instead, a few peaks corresponding to $\beta$-Nb with the bcc structure are detected, which should be associated with the decomposition reaction of $\beta$-Zr $\rightarrow$ $\alpha$-Zr+$\beta$-Nb [16]. One can thus confirm from the XRD results that the monotectoid temperature of the present Zr-2.5Nb alloy is between 560 and 580°C.

3.2 ECC observation

Direct microstructural observations by ECC for various specimens are presented in Figures 2 and 3. For the as-received

![Figure 1](Color online) XRD patterns of various specimens.