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Effect of annealing on microstructure and tensile property of a novel ZrB alloy

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ABSTRACT

With the aim to develop new Zr-based alloys with excellent mechanical properties, a novel Zr-0.8B (wt%) alloy was manufactured through the following steps: casting, forging, hot-rolling and annealing treatment. The microstructure and mechanical properties of the hot-rolled and annealed Zr-0.8B alloys were investigated. The microstructure of all specimens was comprised by α and ZrB_2 phases. The coarse α laths and ZrB_2 whiskers were significantly refined by hot rolling. Also, a high volume of dislocations and many subgrains were formed during the hot-rolling process. A high degree of recrystallization of α phase was obtained during the annealing treatment, due to the substantially stored energy in the Zr matrix and the presence of a significant amount of subgrains. The tensile strength of the annealed Zr-0.8B alloys was decreased with the increase of annealing temperature, until 800 °C, and then increased as the heat treatment temperature was further increased to 900 °C. Many factors, such as the volume fraction of ZrB_2 whiskers, grain size, dislocation density, the amount of subgrains and solution strengthening, have a significant effect on the strength. However, the elongation-to-failure showed a reverse tendency compared to the tensile strength. The reasons caused fracture of the annealed Zr-0.8B alloys are primarily attributed to dislocation pile-ups and the presence of microvoids.

1. Introduction

Due to their excellent irradiation resistance, small thermal neutron absorption cross-section, moderate strength and ductility and superior corrosion resistance, Zirconium (Zr) and Zr alloys have been widely employed in nuclear and chemical industries. For example, Zircaloy-4 and Zr–Nb alloys have been employed as nuclear fuel cladding materials for several decades [1]. Several newly developed Zr alloys, such as M5 (Zr-1 Nb–O) [2], Zirlo (Zr-1.0 Nb–1.0Sn–0.1Fe) [3] and NDA (Zr-0.1 Nb-1.0Sn-0.27Fe-0.16Cr) [4], are being tested in reactor. Zr705 is being increasingly employed into acetic acid plants [5]. This is attributed to the outstanding chemical and physical properties of Zr; the corrosion behavior of Zr and its alloys has been the subject of work in many studies [6–8]. By comparison, the mechanical properties of Zr alloys have received much less attention during the last decades. However, a wider variety of advanced Zr alloys has been lately required, due to the increase applications in more severe operating conditions, such as higher operation pressure, higher pH operation and increased operation temperature [9]. The relatively low strength of the conventional Zr alloys (approximately 300 Mpa–600 Mpa) limits the areas where Zr alloys can be used, such as the petrochemical, aeronautical, marine, chemical and desalination industries. Hence, the development of new Zr-based alloys with higher strength is of significant importance.

Boron (B) containing Zr alloys have attracted considerable interest during the recent years, due to their several advantages over conventional Zr alloys, such as superior fatigue properties, excellent wear properties, increased strength with comparable ductility and relatively easier processing capability [10–12]. Recently, a series of ZrB alloys that exhibit higher strength, as compared to conventional Zr alloys, has been developed [13]. The effect of boron concentration on microstructure and mechanical properties of hot-rolled plates has been reported [13]. Although the tensile testing results showed that the yield and ultimate tensile strength of Zr-B alloys was increased with the increase of B content, the poor elongation was not as well as we had expected. Therefore, further research on the ZrB alloys, particularly fouced on their plastic properties, is necessary. The refinement of α laths and the presence of hard and brittle ZrB_2 whiskers are responsible for the unsatisfied elongation values. In the dispersion-hardened system, the relatively small α laths acted as hard agents and the amount of the small α lath had an important impact on the macroscopic ductility [14]. Compared to the fine α laths, alloys

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with a microstructure composed by equiaxed grains may demonstrate enhanced ductility and higher strength. Moreover, crack formations are often accompanied by the ZrB_2 whiskers. Heat treatment is a common way to control and adjust structure and properties of Zr alloys [15–17]. Therefore, investigating the microstructure evolution and corresponding mechanical properties of alloy following various heat treatments, has a great promoting effect on the ZrB alloy.

The aim of the present work is first to investigate the microstructure evolution of the Zr-0.8B alloys following various annealing heat treatments. Then, the effect of annealing temperature on mechanical properties was studied and discussed, in relation to the evolution of the microstructure.

2. Experimental procedure

Sponge Zr ($\geq$99.5% purity) and B powder ($\geq$99.9% purity) were used to prepare the experimental material with the following composition: Zr-0.8B (in wt%). The raw materials were blended and then melted via electromagnetic induction furnace using a water cooled copper crucible under an argon atmosphere. For ensuring compositional homogeneity, the ingot was remelted three times. Following melting and cooling, the cast ingot was hot-forged into a round bar (Φ 45 mm) in order to eliminate any casting defects. According to the DSC curves, as shown in Fig. 1, the heat forging was performed after holding at β phase region (1100 °C) for 1.5 h. Then, a process for relieving forging stress was conducted in a tubular vacuum heat treatment furnace. The treatment was performed at 1100 °C for 12 h, under a protective argon atmosphere. Following the stress relieving process, the microstructure of the round bar was obtained and presented in Fig. 2.

Five rectangular specimens (15 mm × 15 mm × 100 mm) were cut by electric discharge machining (EDM) from the round bar. According to the DSC curves, as shown in Fig. 1, the 15 mm thick specimens were heated to 800 °C (α+β phase region) for 30 min and then rolled into 6 mm-thick plated samples, at a strain rate of 0.6 s^{-1}. The total deformation was 60%. All the specimens were rolled under the same conditions and then cooled to room temperature (RT) in air. After rolling, annealing heat treatments were performed at 600, 700, 800 and 900 °C for 90 min and then cooled within the furnace.

Phase identification was carried out via X-ray diffraction employing a Cu Kα X-radiation (XRD, Rigaku D/max-2500PC) and matching each characteristic peak with JCPDS files. The microstructures were assesses via optical microscopy (Zeiss Axiovert 200 MAT), transmission electron microscopy (TEM, JEOL-2010) and scanning electron microscopy (SEM, Hitachi S-3400), coupled with energy-dispersive spectroscopy (EDS). The microstructural parameters were evaluated by image analysis software. The samples for metallographic observations were mounted and mechanically polished through standard metallographic procedures. The polished samples were etched in an etchant with composition of water: HF (hydrofluoric acid): HNO_3 (nitric acid)=85:5:15 (vol%). The TEM specimens were prepared through twin-jet electrochemical polishing in etching solution containing methanol (CH_3OH) and perchloric acid ($HClO_4$) (90:10 vol%), at 14 V and –35 °C.

The specimens for tensile testing were cut by EDM from the annealed alloys along the rolling direction (RD). Bone-shaped plate tensile specimens with a cross-sectional dimension of 2 mm × 3 mm (height × width) and an original gauge length of 21 mm were used for the tensile tests. Tensile testing was performed using an Instron 5982 universal mechanical testing machine, at a strain rate of 5×10^{-4} s^{-1}. Three specimens were tested for each annealed alloy, to ensure repeatability and get the average values. The fracture surfaces of the specimens, following tensile testing, were investigated by SEM.

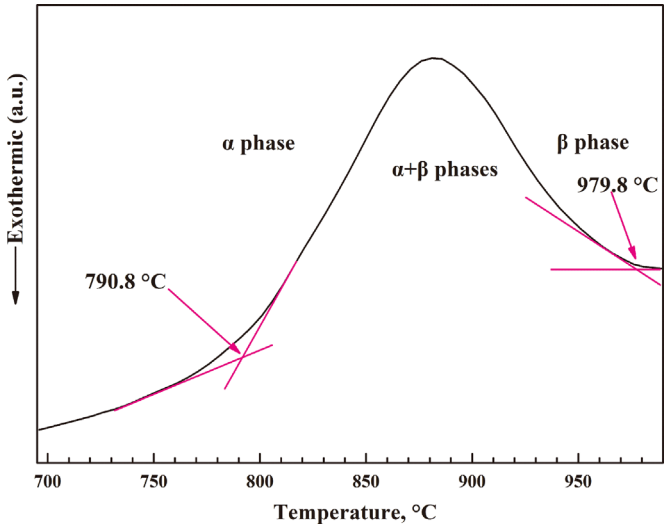

Fig. 1. DSC curve of the Zr-0.8B alloy near α+β phase region.

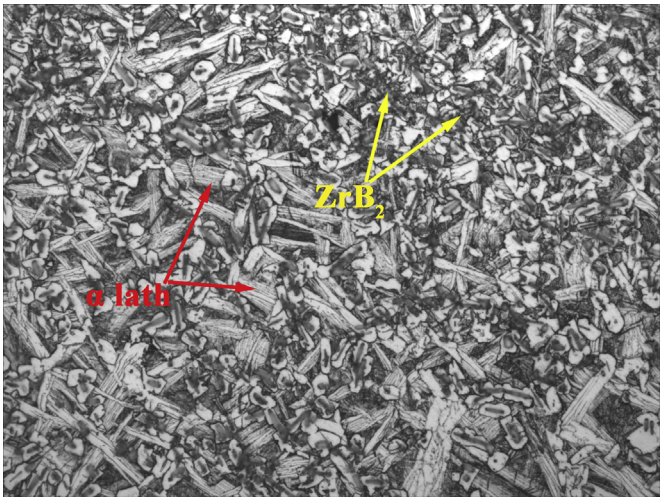

Fig. 2. Microstructure of the round bar.

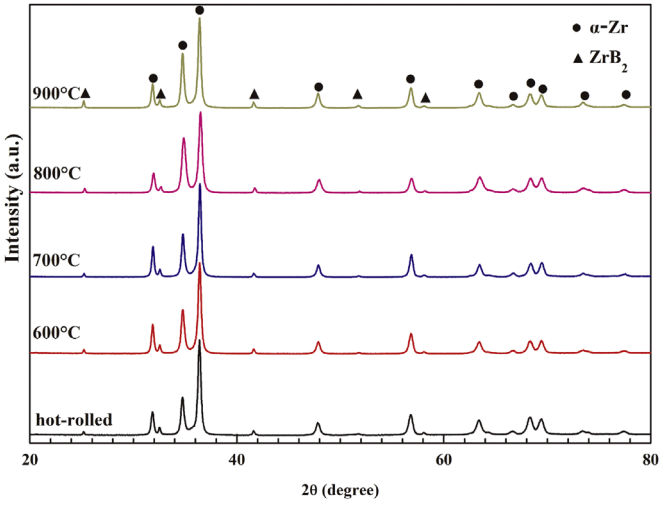

Fig. 3. XRD patterns of the Zr-0.8B alloys with varying annealing temperatures.