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Hot deformation behavior of Zr-1%Nb alloy: Flow curve analysis and microstructure observations

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ABSTRACT

The hot deformation behavior of Zr-1%Nb alloy has been studied at temperatures of 770–850 °C and strain rates of 10^{-2} to 1 s^{-1}. To minimize friction during the tests at high temperature, the ends of the specimens were coated with graphite powder and a thin layer of mica was placed between the face of the specimen and the anvils. In order to investigate the warm working behavior of Zr-1%Nb alloy, all data were analysed stepwise and the step-by-step procedure was provided. The occurrence of the dynamic recrystallization (DRX) phenomena at different working conditions was analysed by the work hardening rate analysis. The stress-strain curve of many samples was identical to the typical DRX stress-strain curve which consists of a single peak stress followed by a gradual decrement toward the steady-state stress. Microstructural observations showed that at low temperatures and high strain rates the flow curves can be explained by localized deformation as a consequence of shear band formation. The transmission electron microscopy (TEM) analysis showed that the DRX has taken places through a continuous mechanism. At high Zener-Hollomon (Z) parameter a drop in flow stress was detected as a consequence of adiabatic deformation heating. The warm working constants of this alloy were determined through the general constitutive equations. Furthermore, after analyzing the work hardening behavior of this alloy, the activation energy for deformation of Zr-1%Nb alloy was calculated as 514 kJ/mol.

1. Introduction

Zirconium in pure form exhibits a low temperature α-stable phase with hexagonal close-packed (hcp) structure and a body-centered cubic (bcc) β-phase stable at elevated temperatures. Alloying elements affect the relative stability of the phases at a given temperature, and can produce a two phase (α + β) regime at intermediate temperatures [1,2]. Zirconium is an irreplaceable structural material in fission reactors due to its low neutron capture cross-section, excellent corrosion resistance, reasonable mechanical properties and good heat conductive properties [3,4]. Zr–Nb alloys play an important role in the energy production, being the main material for the cladding of nuclear fuel in the nuclear power plants [5,6]. Industrial hot deformation processing such as forging for these Zr alloys is conducted in the temperature range where a duplex structure (α + β) occurs. The understanding of hot flow stress is quite important in metal forming processes such as rolling and forging. As a result, considerable research has been carried out to model the flow stress of metals and alloys. Most of these models divide the stress–strain curves into two regions: the first involving the effects of work hardening and dynamic recovery on flow stress and the second adding the softening caused by dynamic recrystallization (DRX) [6,7]. Some equations have also been proposed for modeling the flow curves [8]. Moreover, artificial neural networks (ANN) have been successfully used for the prediction of hot flow stress [9,10]. In the present work, the hot flow stress of a Zr-1%Nb alloy was modeled and predicted by constitutive equations for the first time. The constants of constitutive equations and the activation energy for the warm deformation of this alloy were also calculated. In addition, the work hardening studies were undertaken to identify the stress corresponding to the initiation of DRX precisely.

2. Materials and methods

2.1. Sample preparation

The base alloy was homogenized at a temperature of 1050 °C for 1 h (with water-quench microstructure). Cylindrical specimens were machined with height of 12 mm and diameter of 8 mm. In order to

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Fig. 1. left side: cylindrical sample before the hot compression test, right side: deformed sample after hot compression.

minimize the risk of inhomogeneous compression due to friction between the anvils and the specimen surface, the entire end face of the specimens was machined away to form small ring (Fig. 1 right side). Subsequently, the rings were filled with graphite powder as a lubricant material then a thin layer of mica was placed between the face of the specimen and the anvils so as to maintain uniform deformation and avoid sticking problems during quenching.

2.2. Equipment

The warm working investigations were carried out by a hot compression testing machine (Zwick Roell Z250). The equipment included a load frame rated for a maximum load of 250 kN, a hydraulic power supply and closed loop servo hydraulic and computerized outer loop systems. After finishing of each hot compression cycle, the load and displacement data were recorded by a data acquisition system.

2.3. Experimental procedure

The specimens, starting with a β-quench microstructure, were kept at the deformation temperature for 5 min before the hot compression test. Thereafter, the specimens were deformed at a constant speed of 600, 60, 6, 0.6 mm/s up to a total strain of 0.87. The schematic of hot compression tests are shown in Fig. 2. In fact, all the hot compression tests were undertaken at temperatures of 770, 800, 820 and 850 °C, with strain rates of 10^{-2} to 1 s^1.

2.4. Friction effect consideration

In principle, the effect of friction between the sample surface and anvils during the hot compression test cannot be eliminated completely. Nevertheless, this residual friction affect the stress curves of samples and thus this effect should be taken into account in order to correct the raw stress data based on this effect. In this work, the upper-bound theory which is a simple theoretical analysis of the barrel compression test for determination of the constant friction factor (*m*) was used [11].

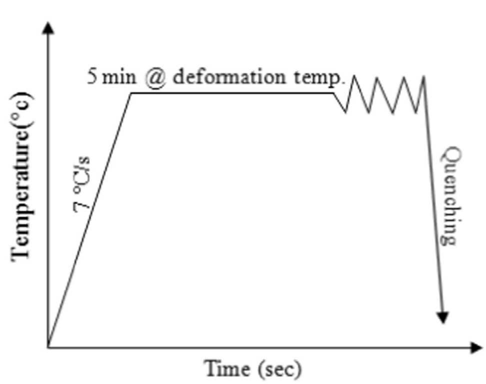

Fig. 2. The heating and deformation schedule used in the experiments.

2.5. Curve fitting

Traditionally, the dynamic recrystallization was detected through the presence of stress peaks in flow curves. However, it has been reported that some materials do not illustrate well-defined peaks related to the occurrence of DRX and further analysis are required to detect the onset of DRX. Poliak and Jonas [12–14] have shown that the onset of DRX can also be identified from inflections in plots of the strain hardening rate as a function of stress. Thereafter, this method was simplified by Najafizadeh and Jonas [15]. Several research have shown that this technique can be an appropriate way to identify the onset of DRX, especially in the case that the flow curves do not display the well-defined peaks [12–15]. The work hardening (θ) is calculated through the derivation of the true stress with respect to true strain. However, presence of short range noises in the flow curves make this calculation impossible and thus to address this problem, the stress-strain curves should be smoothed by fitting a high order polynomial to the curve [6,13,15]. In fact, through this fitting all the irregularities and fluctuations in the stress-strain curves would be removed and calculation of the work hardening can be feasible. In the current work, all the stress-strain curves were modified to eliminate the effect of friction and thereafter the corrected curves were smoothed through the fitting a sixth order polynomial. The shear modulus dependence on temperature for Zr–1%Nb in the temperature range under consideration, was taken as [16]:

$$\mu = 29.28 - 0.021T \tag{1}$$

Where μ is in GPa and T in K. The effect of the temperature on the shear modulus inherently present in experimentally measured stress is eliminated using the ratio σ/μ [16].

2.6. Microstructure characterization

Optical microscopy and Transmission electron microscopy (TEM) were employed to study the microstructure of cross sections of samples after warm deformation. For this reason, the samples were polished up to 1 µm grit diamond paste. An etchant with the composition of 10 ml HF, 45 ml HNO_3 and 45 ml H_2O was used to etch the samples [17]. TEM samples were prepared by means of a twin-jet electropolisher at 20 V using a solution of methanol, percholoric acid and n-butanol at −40 °C.

3. Result and discussion

3.1. Friction corrected curves

Table 1 demonstrates the friction coefficients (*m*) calculation

Table 1
Calculated friction coefficients at different deformation conditions.

Friction coefficient	Temperature,°C	Strain rate, s^{-1}
0.08	820	0.3
0.09	850	1
0.1	750	0.3
0.13	850	0.1
0.14	770	0.3
0.15	770	0.1
0.17	750	0.01
0.18	800	0.01
0.2	800	1
0.23	770	0.1
0.25	800	0.1
0.26	820	0.1