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Microstructural characterization of creep anisotropy at 673 K in the M5® alloy

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Abstract

Zirconium alloy tubing is used in pressurized water nuclear reactors in order to prevent fissile material from leaking into the coolant. It can be the first safety wall of nuclear fuel, and is submitted to complex thermomechanical loadings. In consequence, new Nb-modified alloys, such as the M5® alloy, and fine numerical models are being developed to guarantee a better and longer mechanical integrity of these tubes. To identify the physical mechanisms that could be considered in such models, an experimental approach, combining creep tests with electron backscattered diffraction and Transmission electron microscopy investigations, was carried out.

Tubular specimens were submitted to multiaxial creep tests at a temperature of 673 K. Seven ratios between the axial and hoop applied stresses were investigated. It enabled a macroscopic evidence of the creep anisotropy. Besides, EBSD analyses on a mesoscopic-sized non deformed area led to the characterization of the variation of the average Schmid factor with the direction of loading. Finally, TEM observations were done on seven crept samples, corresponding to the seven directions of loading tested mechanically. The variations of the different parameters investigated (activated slip systems, dislocation densities, curvatures of the dislocations) can be seen as the effects of the creep anisotropy at a microscopic scale. The correlation between results is then discussed in a multiscale frame.

Keywords: Zirconium (Zr); Creep; Anisotropy; Transmission electron microscope (TEM); Dislocation

1. Introduction

Zirconium alloys are widely used in the nuclear industry as a major core component in boiling water and pressurized water reactors. The thermal creep strength of these components is of prime importance to ensure the mechanical integrity of core structures during and after their use in the reactor. In consequence, thermal creep mechanisms have been widely studied during the past 50 years [1–5]. Although there is no consensus concerning the physical mechanisms involved, creep anisotropy of zirconium alloy tubing is generally accepted [6–11]. A better understanding of this phenomenon would lead to improvements in modeling and predictions of the creep behavior of core structures.

Creep anisotropy at 673 K for low strain levels is mainly due to a combination of two metallurgical properties. Firstly, the fabrication process of the tubes is at the origin of a strongly heterogeneous crystallographic texture [6,9]. Secondly, the limited number of possible slip systems and their orientation in the hexagonal close packed (hcp) lattice induce a localization of dislocation glide. Indeed, ab initio simulations [12,13] confirm that prismatic glide is more liable to occur than basal glide for alpha zirconium (αZr). These arguments may therefore explain a variation in the ease of dislocation glide with loading direction, which can be linked to the macroscopic anisotropy observed during tensile strengthening [10]. However, the consequences for

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creep behavior remain unclear. Moreover, developments of analytical descriptions of creep data, through computation using stress exponents [4] or Transmission electron microscopy (TEM) observations [5], suggest that dislocation climb could replace dislocation glide as a rate-controlling process at intermediate temperatures (673–1123 K). Thus, characterization of creep anisotropy needs new experimental data obtained at different length scales.

In the present work, we propose to investigate creep mechanisms on the M5® alloy at 673 K, using a multiscale approach to the effects of the loading direction. Thus, after a presentation of materials and characterization techniques, we will first focus on creep tests, which constitute a macroscopic characterization of creep anisotropy. Then, electron backscattered diffraction (EBSD) analyses, i.e. a mesoscopic scale characterization, are presented with an emphasis on the effect of crystallographic texture, presented through Schmid factor computations. These results led us to assume that strain could be carried mainly by prismatic and pyramidal dislocation glides along the $\langle a \rangle$ axis of the hcp lattice. Finally, this hypothesis is checked at a microscopic scale using TEM. At least 50 grains were observed on several specimens crept at 673 K under different multiaxial loadings (seven directions in stress space). This enabled a statistical quantification of the observed parameters: dislocation densities, activated slip systems, and curvature of dislocations. The evolution of these parameters with the direction of loading is discussed with regard to the creep test results and EBSD analysis. Our results suggest that cross-slip activation and long-range internal stresses can explain the anisotropy of creep in relation to the crystallographic texture.

2. Experimental features

2.1. Material

The M5® tubes used for this study were provided by AREVA. They were formed by a cold pilgering rolling process and then subjected to a final recrystallization heat treatment. These processes induce a fully recrystallized hcp alloy microstructure, with a strongly anisotropic crystallographic texture (Fig. 1). Grain sizes vary between 3 and 5 μm, and the zirconium content of the studied material is higher than 98.5%. The chemical composition of the M5® [14] is given in Table 1. Several second phase particles, due to alloying elements, are present in an α-matrix. To check those data, and to question the effects of the sample preparation process on the microstructure, we observed by TEM a thin foil taken on a non-deformed sample. No dislocations were observed inside the material before creep tests.

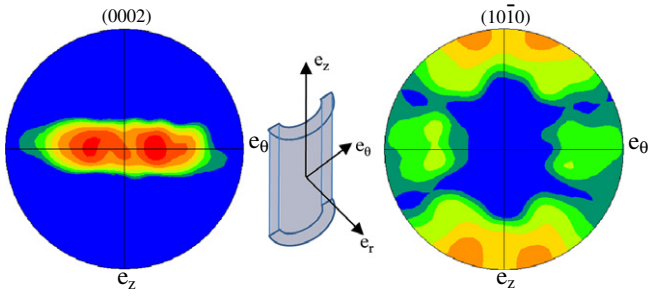

Fig. 1. (0002) and {10−10} pole figures of the recrystallized zirconium alloy (EBSD analysis performed on 20,000 grains).

Table 1
Chemical composition (in weight) of the M5®.

Nb (%)	O (ppm)	Fe (ppm)	Cr (ppm)	Zr
1	1450	370	41	Bal.

2.2. Mechanical tests

The specimens used for creep tests are 130 mm long, 0.5 mm thick cylinders cut from M5® tubes. The dimensional measurements and temperature regulation were made on a gauge of 20 mm long, centered on the middle of the specimens. Specimens are sealed by a clamping set before being subjected to creep tests with a specific device [15]. A constant temperature of 673 ± 2 K is maintained on the gauge using a radiative furnace. An axial load up to 5 kN can be applied simultaneously with an internal pressure up to 280 bars. As the specimens are thin-walled tubes, the radial stress can be ignored. We define the biaxial stress ratio $\beta = \sigma_{zz}/\sigma_{\theta\theta}$, with $\sigma_{\theta\theta}$ and σ_{zz} being respectively the applied axial and hoop stresses. The angle of the total applied stress with the axial direction is called α and we can set $\alpha = \arctan(1/\beta)$. Creep tests were stopped when stationary creep strain rate stabilized, leading to final equivalent creep strains between 1% and 3%.

2.3. Transmission electron microscopy method

Thin foils were cut parallel to the outer surface of the tested specimens, in the zone of the gauge. Thin foils were first mechanically ground to a thickness of 150 μm. They were then electrolytically polished with a Struers TenuPol®, at a voltage of 15 V and a temperature of 243 K in a 10% perchloric acid–20% 2-butoxyethanol–70% methanol solution. In order to relate the crystallographic orientations to the loading directions, a notch corresponding to the axial direction was placed on each thin foil and orientated parallel to the tilt axis of the sample holder. The observations were carried out at a temperature of 293 K in a Jeol 2011 Transmission electron microscope working at 200 kV (tilt ± 25°). For each of the seven directions explored under mechanical testing defined by α or β, at least 50 grains were randomly selected and observed under several diffraction conditions, to enable an accurate identification of their crystallographic orientations. Several electron diffraction patterns of each grain were used to build stereographic projections using the CaRIne Crystallography software [16]. The positions in a $\{e_\theta; e_z\}$ system of basal planes determined with this method (Fig. 2) are in