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# Understanding thermally activated plastic deformation behavior of Zircaloy-4

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# highlights are the control of

• The transition from thermally activated to athermal regime took place at ~673 K.

The athermal stress at 673 K was 115 MPa.

The uniform ductility increased in dynamic strain aging regime.

The activation volume was estimated using repeated stress relaxation technique.

 $\bullet$  In DSA regime, dislocation  $-$  oxygen interaction controls the plastic deformation.

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Understanding micromechanics of plastic deformation of existing materials is essential for improving their properties further and/or developing advanced materials for much more severe load bearing applications. The objective of the present work was to understand micromechanics of plastic deformation of Zircaloy-4, a zirconium-based alloy used as fuel cladding and channel (in BWRs) material in nuclear reactors. The Zircaloy-4 in recrystallized (at 973 K for 4 h) condition was subjected to uniaxial tensile testing at a constant cross-head velocity at temperatures in the range 293 K-1073 K and repeated stress relaxation tests at 293 K, 573 K, and 773 K. The minimum in the total elongation was indicative of dynamic strain aging phenomenon in this alloy in the intermediate temperature regime. The yield stress of the alloy was separated into effective and athermal components and the transition from thermally activated dislocation glide to athermal regime took place at around 673 K with the athermal stress estimated to be 115 MPa. The activation volume was found to be in the range of  $40 b<sup>3</sup>$  to 160 b<sup>3</sup>. The activation volume values and the data analyses using the solid-solution models in literature indicated dislocation-solute interaction to be a potential deformation mechanism in thermally activated regime. The activation energy calculated at 573 K was very close to that found for diffusivity of oxygen in  $\alpha$ -Zr that was suggestive of dislocations-oxygen interaction during plastic deformation. This type of information may be helpful in alloy design in selecting different elements to control the deformation behavior of the material and impart desired mechanical properties in those materials for specific applications.

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## 1. Introduction

Undoubtedly, materials have played an important role in providing safety, security, and advancement of human societies in the past and will continue to do so in the foreseeable future [[1\]](#page--1-0). For example, without ferrous alloys and aluminum alloys, the transportation industry could not have achieved its current status. The

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<https://doi.org/10.1016/j.jnucmat.2018.03.031> 0022-3115/© 2018 Elsevier B.V. All rights reserved. nuclear power generation industry is no exception to this when it comes to their reliance on advanced materials [\[2\]](#page--1-0). Advanced materials such as radiation resistant ferritic steels, stainless steels, and zirconium alloys have been enablers of various nuclear technologies [[3,4](#page--1-0)].

Generation-IV reactors have broadened the operating envelop of the nuclear reactors significantly and, therefore, the materials are expected to operate at relatively higher temperatures, higher neutron irradiation doses, and in much more corrosive environ-ment for longer duration [\[2](#page--1-0),[5,6](#page--1-0)]. It has resulted in current fleet of Corresponding author.<br>
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either in terms of unsatisfactory performance at the operating conditions or unknown materials performance in the new operating regime. Fuel cladding tubes are used in the nuclear reactor core to contain radioactive fuel. At present, in light water reactors, zirconium-base alloys are used for this purpose. In older reactors, other materials such as aluminum and magnesium alloys have been used in the past, and for future reactors new zirconium alloys with improved properties or ferrous-base alloys are being considered [[3,4](#page--1-0)].

From alloy design perspective for load-bearing applications, so far, we have largely relied on trial and error method aided by experience gained and knowledge developed over several decades. Moreover, scientists and engineers have largely tried connecting role of processing and alloying elements to microstructure and macroscopic mechanical properties. However, the need of the hour is to use the tools and techniques available in scientific database to tie processing and alloying to the microscopic deformation related parameters, which in turn govern mechanical properties of materials. This kind of approach will serve two purposes  $-$  first, it will bring a shift in our experimental approach to alloy development and second, the microscopic parameters derived from such approach may be used for validating computational tools being developed for accelerating pace of new material discovery [\[7\]](#page--1-0).

In the present work, we have studied micro-mechanisms of plastic deformation of Zircaloy- $4 - a$  Zr-base alloy. Although a number of studies in the past has focused on high temperature plastic deformation behavior with some studies covering a wide range of temperature  $[8-22]$  $[8-22]$  $[8-22]$  $[8-22]$ , very few have focused on understanding micromechanics of plastic deformation of zirconium alloys  $[20,23-27]$  $[20,23-27]$  $[20,23-27]$  $[20,23-27]$  $[20,23-27]$  $[20,23-27]$  $[20,23-27]$ . For example, Boratto et al.  $[9]$  $[9]$  $[9]$  studied deformation behavior of a hot-worked bar of Zircaloy-4 in dynamic strain aging regime and have reported a minimum in ductility at about 673 K (400 $\degree$ C. Talia and Povolo have studied influence of tensile test parameters on the strength and ductility of an annealed Zircaloy-4 and reported that test parameters such as heating rate, hold time, and vacuum condition having strong influence on the strength of the alloy  $[11]$  $[11]$  $[11]$ . In another study by Lee et al.  $[18]$  $[18]$  $[18]$ , it was shown that ductility decreased in the temperature range  $523 K-673 K$  $(250-400 \degree C)$  and it was attributed to a decrease in strain-rate sensitivity of the alloy in this temperature range.

All these studies were important to develop an overall understanding of the deformation behavior of zirconium alloys at macroscopic scale. However, as stated earlier, these studies were not sufficient to glean an insight into micromechanics of plastic deformation in zirconium alloys. In this context, the work carried out by Trojanova et al. [[16\]](#page--1-0), Heritier et al. [\[23\]](#page--1-0), and Derep et al. [[24\]](#page--1-0) are important. Trojanova et al. [[16\]](#page--1-0) have studied the thermally activated mechanism controlling the slip in Zr-Sn alloys and concluded that non-conservative motion of jogs in screw dislocations might be controlling the plastic deformation at elevated temperature. Similarly, the work carried out by Heritier et al. [\[23\]](#page--1-0) on  $\alpha$ -Zr in temperature range 648–898 K as a function of strainrates revealed that the athermal range was being controlled by Orowan stress needed for bowing-out of Frank network of dislocations. The work of Derep et al. [\[24\]](#page--1-0) on Zircaloy-4 in the temperature range 77–900 K found a number of mechanisms controlling the deformation behavior of the alloy in different temperature ranges within  $77-900$  K.

In this work, using uniaxial tensile testing and repeated stress relaxation tests as a function of temperature, we have investigated macroscopic mechanical properties and evaluated associated microscopic thermally activated parameters of Zircaloy-4 [\[28,29](#page--1-0)]. The use of repeated stress relaxation test allows computation of true activation parameters related to plastic deformation at constant microstructure which is not possible in the cases where different samples are tested at different strain-rates or during strain-rate jump tests on the same sample for the determination of activation parameters  $[28]$  $[28]$  $[28]$ . It is hoped that the knowledge gained from this work may serve as a basis for modifying the mechanical properties of such alloys for future use.

### 2. Experimental methods

#### 2.1. Materials

The Zircaloy-4 sheet was received in cold-rolled condition and was recrystallized at 973 K (700 $\degree$ C) for 4 h in argon atmosphere before investigating its mechanical properties in this study. The alloy consisted of 1.62 (wt.%) Sn, 0.23 Fe, 0.11 Cr, 1158 ppm  $O<sub>2</sub>$  and the rest Zr. The oxygen content was measured using inert gas fusion technique and the contents of the rest of the elements were estimated using PerkinElmer ICP-optical emission spectrometer [\[27](#page--1-0)].

#### 2.2. Microstructural characterization

The microstructural characterization of the alloy was carried out using electron backscattered diffraction (EBSD) technique. The samples for EBSD were prepared using mechanical polishing followed by electropolishing. During mechanical polishing step, the final polishing was done using 50 nm colloidal silica solution. The electropolishing was performed in 10 vol% perchloric acid and 90 vol% methanol solution at ~233 K ( $-40^{\circ}$ C) for 10 s followed by cleaning in liquid nitrogen cooled methanol.

#### 2.3. Mechanical testing

#### 2.3.1. Uniaxial tensile testing and stress relaxation testing

The geometry of the specimen used in the present study for mechanical properties evaluation is shown in Fig. 1. The tensile tests were carried out at temperatures from 293 to 1073 K  $(20-800 \degree C)$  at a constant cross-head velocity (initial strain-rate:  $10^{-3}$  s<sup>-1</sup>). For stress relaxation study, each specimen was loaded up to a pre-determined level of stress following which the crosshead was stopped for 30 s during which stress relaxation took place. At the end of 30 s, the specimen was reloaded to the same level of stress and the process was repeated four times. The stress relaxation studies were carried out at room temperature (20 $\degree$ C), 573 K (300 $\degree$ C), and 773 K (500 $\degree$ C). In each case, the tensile sample was oriented along the rolling direction of the sheet, i.e., the loading axis was parallel to the rolling direction.

#### 3. Results

#### 3.1. Microstructure

[Fig. 2](#page--1-0)(a) shows the orientation image micrograph (OIM) of the Zircaloy-4 in recrystallized condition. The color associated with each grain in the OIM micrograph represents a crystallographic direction perpendicular to the plane of the paper. The information



Fig. 1. Tensile test specimen geometry; all dimensions are in mm.

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