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⟨a⟩ Prismatic, ⟨a⟩ basal, and ⟨c+a⟩ slip strengths of commercially pure Zr by micro-cantilever tests

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ABSTRACT

Slip strengths of ⟨a⟩ basal, ⟨a⟩ prism, and ⟨c+a⟩ pyramidal systems in commercially pure zirconium have been determined using micro-cantilever testing. A range of single crystal cantilevers 0.5 μm to 10 μm wide, oriented for single slip were prepared using focused ion beam (FIB) machining and subsequently deflected using a nanoindenter. The critical resolved shear stress (τ_{crss}) was found by fitting a crystal plasticity finite element model to the experimental load–displacement data for these micro-bending tests. All the three slip systems in α-Zr show a marked size effect in bending described well by $\tau_{CRSS}(W) = \tau_0 + AW^n$, where W is the cantilever width, τ_0 is the CRSS at the macro scale and $n = \sim -1$. The exponent, n, of near -1 is in good accord with hardening caused by the back stress generated by dislocations piling up at a diffuse barrier caused by the reduction of stress near the neutral axis. The macro scale CRSS values were used to successfully simulate deformation of a conventional macroscopic compression test.

1. Introduction

Zirconium is widely used in the nuclear industry as nuclear fuel rod cladding in water reactors, due to its low neutron cross section, good corrosion resistance and reasonable mechanical properties. To provide an adequate safety case and ensure safety during operation and shutdown, the mechanical performance of these polycrystalline components must be well known. Zirconium alloys for this application typically contain predominately the α-Zr (hcp) phase which exhibits pronounced anisotropy in both its elastic and plastic properties at the single crystal level. During processing strong and complex crystallographic textures are generated [1–3] which result in macroscopically anisotropic mechanical properties. Design and operation of a reactor must involve selection of appropriate materials, processing routes and textures to provide adequate strength for in-service operation. One such property is the critical resolved shear stress (CRSS) which describes the critical stress for plastic deformation to occur on a given slip system.

A physical understanding of conditions required for crystallographic slip is of clear importance when discussing the processes involved in deforming a polycrystalline aggregate. Unfortunately the reverse problem, extracting these physical properties from macroscopic tests is difficult due to competition between externally applied stresses and local constraint, due to networks of grains and other microstructural features which must deform together in order to accommodate plasticity. This problem is especially exacerbated when there are many 'soft' grains (i.e. ⟨c⟩ axis perpendicular to applied stress) which deform extensively while neighbouring 'hard' grains (⟨c⟩ axis parallel to applied stress) develop higher stresses but limited plastic strains. In these situations there is strong heterogeneity in the stress and plastic strain fields at the granular and intra-granular length scales. This heterogeneity or strain localisation has a significant role on the early stages of material failure processes.

The most direct route to determining CRSS values is to grow large single crystals that can be oriented so as to preferentially slip on the desired slip system during conventional tensile or compressive testing. This is a technically difficult undertaking especially for alloys and most results are from quite early work and are limited to pure Zr. In an extensive testing campaign Soo and Higgins [4] used strain rate jump tests to study the effects of oxygen content and temperature on the plastic flow of α-Zr oriented for prism slip. Near room temperature they found that increasing the oxygen content from 135 ppm to 2000 ppm caused the CRSS to increase from ~24 MPa to ~120 MPa, with a corresponding decrease in the activation volume from $312b^3$ to $40b^3$. Akhtar and Teghtsoonian [5] also made CRSS measurements for prism slip in high purity 100–200 ppm oxygen Zr single crystals. Further measurements on samples oriented for basal slip [6] were only able to generate CRSS values above ~850 K since at temperatures

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below this slip on prism planes or deformation twining was prevalent before activation of basal slip. At this low oxygen content the CRSS for basal slip was only slightly larger than for prism slip at temperatures above 1000 K but the CRSS increased on lowering temperature much more significantly for basal slip than prism slip so that basal slip was twice as hard as prism slip at ~900 K and the divergence continued to lower temperatures. Akhtar also conducted compression tests of ⟨c⟩ axis aligned Zr single crystals and obtained much higher CRSS values for ⟨c+a⟩ pyramidal slip while using slip traces to establish the slip plane was $\{10\bar{1}1\}$. Growing large single crystals for alloys is rarely possible and alternative strategies in which a combination of mechanical testing of a polycrystal while recording X-ray or neutron diffraction patterns coupled with modelling has typically been adopted. For example Skippon et al. [7] use a genetic algorithm to fit a Voce hardening law to neutron diffraction data obtained for in situ compression testing along different macroscopic axes of highly textured Zircaloy-2 alloy. The validity of the material property parameters obtained is dependent on the fidelity of the simulation used to model the deformation process.

Developments in focussed ion beam machining and micro-mechanical testing offer unique insight into deformation processes, as this approach enables micron and sub-micron scale single crystal specimens to be readily fabricated from polycrystalline samples. Unfortunately significant physical issues exist with testing on the small scale, as often the physics of local boundary conditions (i.e. high strain gradients [8,9]) and the scarcity of dislocation sources lead to increased strength and stochastic plastic flow [10]. This makes careful extraction of the properties more difficult, but provided adequate testing is performed and the physical basis of the size effect is understood then this is clearly possible [11–13]. Previously, we have implemented micro-cantilever tests to investigate the anisotropic elastic [14] and plastic properties [15] of titanium single crystals and devised suitable modelling routes for back calculation of key mechanical constants. In this study, we applied the method to study slip properties of single crystal commercially pure α-Zr. ⟨a⟩ Prismatic, ⟨a⟩ basal and ⟨c+a⟩ 1st order pyramidal slip systems were selectively activated in various sizes of micro-cantilevers. Crystal plasticity finite element analysis (CP-FEA) procedures were developed to extract CRSS values for different slip systems from the micro-tests. The size effect in micro-cantilever tests was determined for all the three slip systems. CRSS values extrapolated to the bulk were extracted by subtracting the size effect terms. These bulk CRSS values were then used within a CP-FEA simulation to predict the stress–strain response of a polycrystal deformed in compression, which was found to be in good agreement with experiment.

2. Experiments

A large bar of commercially pure Zr (99.2 wt.%) was purchased from Goodfellow (Goodfellow Cambridge Ltd, http://www.goodfellow.com). The specific chemical composition is given in Table 1. This material was heated to 750 °C, which is just below the α/β transus temperature, for 24 h in a vacuum furnace and then furnace cooled to grow grains, reduce defect populations and relieve residual stresses. After the annealing, the grain size increased to ~70 μm as shown in Fig. 1a. Micro-cantilevers were prepared within single Zr grains using focussed ion beam (FIB) machining.

Table 1
Chemical composition of the commercially pure Zr sample.

Zr	C (ppm)	Hf (ppm)	Fe (ppm)	Cr (ppm)	N (ppm)	O (ppm)	H (ppm)
Balance	250	2500	200	200	100	1000	10

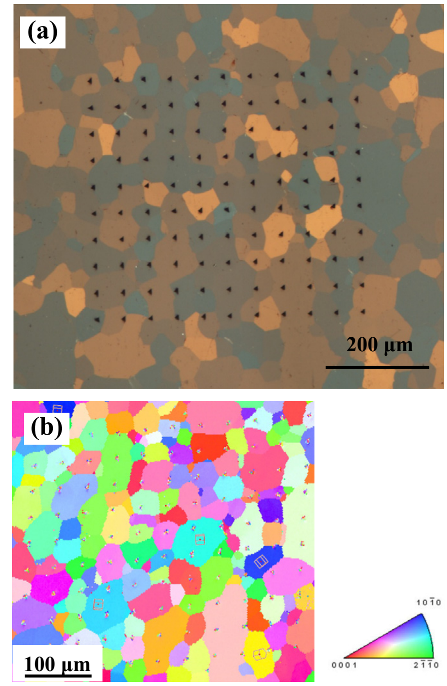

Fig. 1. (a) Polarised light optical image of Commercial pure Zr with indentation marks (b) crystal orientation map of the area shown in (a) indicating the orientation of the crystal with respect to the surface normal.

An indentation array was made to mark the area of interest. Cantilevers were fabricated far enough away from indents to exclude any residual effects associated with the indented volume. EBSD mapping was performed to characterise the local grain orientations, as shown in Fig. 1b. Individual grains, selected for single slip orientations, were chosen from EBSD grain orientation maps such as those in Fig. 1a and b.

Micro-cantilevers were prepared on a Zeiss NVison 40 dual beam FIB system, using a final step of 40 pA at 30 kV to minimize the FIB damage. All cantilevers tested in this work have triangular cross-section, which provides the flexibility to prepare micro-testing pieces in an arbitrary grain with the long axis of the beam pointing in any direction with the sample surface plane (i.e. two degrees of freedom). A series of tests were performed for each slip system with cantilever widths ranging from 0.5 μm to 10 μm. Care was taken to maintain the cantilever beam length to cantilever beam width ratio at 6:1 and to maintain this aspect ratio for all beam sizes. The 6:1 ratio was used so that the beams were neither too short to deviate strongly from simple beam theory [16] but nor were they too long so as to require prohibitive time for FIB machining. Fig. 2a is an example of 5 μm wide, 30 μm long micro-cantilever. Cantilever beam dimensions were carefully measured using the FEG SEM column of the NVision instrument.

Hexagonal materials have a number of slip systems, and there is considerable anisotropy in the single crystal plastic behaviour. Selective activation of each slip system was achieved by testing particular crystal orientations and micro-cantilever alignments within the α-Zr grains [15]. The basic rule for such a test is to maximise the Schmidt factor on the target slip system while minimizing the resolved shear stress for all the others. The precise geometry adopted for each slip system was as follows:

2.1. ⟨a⟩ type

$\langle 11\bar{2}0\rangle\{10\bar{1}0\}$ – prismatic slip: micro-cantilevers were prepared with the ⟨c⟩ axis in the surface plane perpendicular to the