Acta Materialia 95 (2015) 399–410

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

A statistical analysis of the influence of microstructure and twin–twin junctions on twin nucleation and twin growth in Zr

P.-A. Juan [a,c], C. Pradalier [b], S. Berbenni [c], R.J. McCabe [d], C.N. Tomé [d], L. Capolungo [a,*]

[a] G.W.Woodruff School of Mechanical Engineering, Georgia Institute of Technology, UMI GT-CNRS 2958, France
[b] Georgia Tech Lorraine, UMI GT-CNRS 2958, France
[c] Laboratoire d'Etude des Microstructures et de Mécanique des Matériaux LEM3, UMR CNRS 7239, University of Lorraine, Metz, France
[d] MST, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

ARTICLE INFO

Article history:
Received 10 March 2015
Revised 12 May 2015
Accepted 12 May 2015
Available online 18 June 2015

Keywords:
Zirconium
Twinning
Twin–twin junction
Twin nucleation
Twin growth

ABSTRACT

The purpose of the present work is (1) to study the statistical relevance of twin–twin junctions and (2) to study statistically the influence of twin–twin junctions and microstructure on nucleation and growth of twins in h.c.p. materials. A new automated twin recognition technique has been developed and is used to extract statistics from EBSD scans of high purity clock-rolled zirconium specimens loaded along the through-thickness and one of the in-plane directions. The technique allows for recognition of tensile and compressive twin systems within each individual grain. The ten possible twin–twin junction types that may occur in Zr between first generation twins are introduced as well as their associated frequencies in cases of through-thickness and in-plane compression. The present study shows that twin–twin junctions between twins belonging to the most active twinning modes are statistically relevant. It is also shown that twin–twin junctions hinder twin growth. In agreement with previous studies, it is found that irrespective of the loading direction and twin mode, both grain size and crystallographic orientation largely influence the propensity of grains for twin activation. However, the study suggests large differences in nucleation and growth mechanisms for each twinning mode.

1. Introduction

High plastic anisotropy observed in hexagonal close-packed (h.c.p) metals arises from the activation of both twinning and slip systems [1–6]. In zirconium, twinning implies a reorientation of the lattice leading to a mirror image with respect to the twinning plane. $\{10\bar{1}2\}$ and $\{11\bar{2}1\}$ twins form when the c-axis is loaded in tension and $\{10\bar{1}1\}$ and $\{11\bar{2}2\}$ twins form when the c-axis is loaded in compression. Therefore, $\{10\bar{1}2\}$ and $\{11\bar{2}1\}$ twin modes are termed tensile while $\{10\bar{1}1\}$ and $\{11\bar{2}2\}$ are termed compressive [7–9]. Kaschner et al. [2] characterized experimentally the prevalent deformation mechanisms in Zr loaded along the through-thickness and one of the in-plane directions at liquid nitrogen and room temperatures. These mechanisms are prismatic slip, pyramidal slip and tensile twinning at room temperature and prismatic slip and tensile and compressive twinning at liquid nitrogen temperature. They show that the lattice reorientation induced by twinning softens the material when compression twins are activated or hardens the material when tensile twins nucleate and grow [2].

Crystallography of twinning [10,11,1,12,13] is well understood and recent developments in the case of $\{10\bar{1}2\}$ twins have largely refined our understanding of twin growth. However, the contributions to mechanical response of interactions between slip modes [14,15], between slip and twin modes [16–20,5,21,22] and between twin modes on the mechanical response of h.c.p. metals remain less understood. Regarding slip system interaction, dislocation dynamics simulations of plastic deformation in Zr single crystals revealed that no junction is formed between screw dislocations gliding on different prismatic planes at low temperature, resulting in low strain hardening [23]. More recently, Juan et al. [24] introduced a double inclusion elasto-plastic self consistent scheme to study the effect of twin and parent domain interactions on slip system activation and hardening in the AZ31 Mg alloy. The direct coupling between parent and twin phases leads to an increase in hardening and hardening rate of twinned grains. Regarding twin–twin interactions, El Kadiri et al. [25] compared nucleation and growth of $\{10\bar{1}2\}$ twins in Mg AM30 in one grain containing one twin variant and in another grain containing two twin variants with approximately the same Schmid factor. It was

---

[*] Corresponding author.

http://dx.doi.org/10.1016/j.actamat.2015.05.022
1359-6454/

suggested that twin intersections tend to increase the rate of nucleation events while decreasing the growth rate of individual twin lamella. Twin–twin intersections behaved as barriers counteracting further twin propagation and lead to higher strain hardening. Yu et al. [26,27] experimentally characterized three different $\{10\bar{1}2\}$ twin–twin junctions in Mg single crystals that had been cyclically loaded. These prior studies are all limited to $\{10\bar{1}2\}$ tensile twins.

Following an approach similar to the one used by [28,29], the purpose of the present work is to perform a complementary statistical study of the influence of twin–twin junctions, grain size and grain orientation of twinned grains on nucleation and growth of all twins. To this end, a new automated twin recognition technique [30], based on graph theory analysis, is developed and used on electron backscatter diffraction (EBSD) data obtained from clock-rolled high-purity Zr. First the experimental data collected in Capolungo et al. [28] and pertaining to in-plane loading at 76 K of clock rolled Zr is revisited. Second, to assess the generality of these results, additional experimental data are collected for the same material and loading direction. Finally, new experiments on Zr loaded in compression along the through thickness direction are performed and scans are studied in order to analyze compressive twins, their mutual interactions and their interactions with tensile twins.

The first section is dedicated to the experimental procedure and material characteristics. The second section introduces a nomenclature to describe all possible twin–twin junctions in Zr and presents their associated statistics. In the last section, the influence of grain size, crystallographic orientation and twin–twin junctions on nucleation and growth of twins is discussed.

2. Experimental procedure and material characteristics

2.1. Experimental procedure

The material used comes from a high-purity crystal bar Zr (<100 ppm) which was arc-melted, cast and clock-rolled at room temperature. Cuboidal samples were machined from the rolled plate and annealed at 823 K for 1 h. In the as-annealed state, grains are free of twins, equiaxed and have an average diameter equal to 17 μm. Specimens display a strong axisymmetric texture where basal poles are aligned within approximately 30 degrees of the through-thickness direction (Fig. 1). Samples were deformed in an equilibrium liquid nitrogen bath at 76 K in order to facilitate twin nucleation and loaded in compression along one of the in-plane directions to 5% strain (IP05) and along the through-thickness direction to 3% strain (TT03). Fig. 2 shows the macroscopic stress–strain curves of cubes compressed along the through-thickness (TT) and in-plane (IP) directions.

Experimental data were collected from 10 and 4 (240 μm × 120 μm) scans at different locations on the same cross sectional area of the TT03 and IP05 samples (Fig. 3), respectively. The section plane for TT03 analysis contains both the TT direction and IP direction, and the section plane for IP05 analysis contains the TT direction and the IP compression direction. Statistical data were obtained using the automated EBSD technique developed by Pradalier et al. [30]. The total analyzed area for TT03 and IP05 specimens is 205,736 μm^2 and 73,122 μm^2, respectively. Twins represent 9.1% and 5.7% of the total scanned area in TT03 and IP05 samples, respectively. Incomplete grains bounded by scan edges are not considered in the statistical analyses.

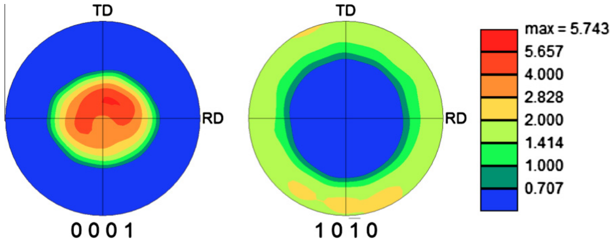

Fig. 1. Initial basal (0001) and prismatic (10–10) pole Figures of the clock-rolled high-purity zirconium studied in this work. The 3-axis is the through thickness direction of the plate.

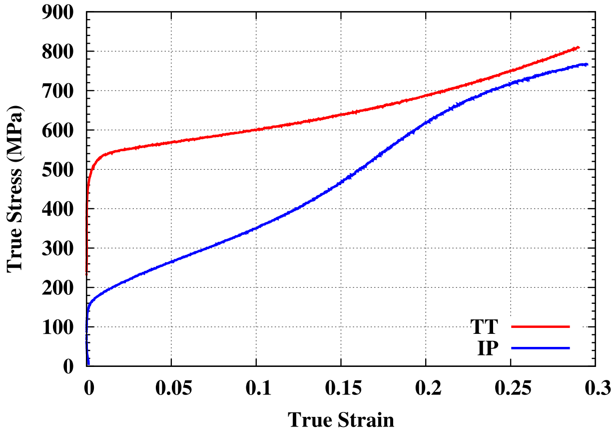

Fig. 2. Macroscopic stress–strain curves of high purity Zr samples loaded in compression along through-thickness (TT) and in-plane (IP) directions at 76 K and 300 K.

Computing misorientations between measurement points and relying on graph theory analysis, the twin recognition EBSD software [30] is able to identify the four twin modes present in Zr (Table 1). As highlighted by recent studies [2,28], $\{11\bar{2}2\}$ compressive (C_1) twins and $\{10\bar{1}2\}$ tensile (T_1) twins are the most commonly observed twins in TT03 and IP05 scans, with 74.4% and 81.7% respectively (Table 2). Table 2 also reveals that the second most active twinning modes are $\{10\bar{1}2\}$ (T_1) and $\{11\bar{2}1\}$ (T_2) in TT03 and IP05 samples, respectively. In both cases, the second most active twin modes represent about 17% of the total number of twins. However, no $\{10\bar{1}1\}$ (C_2) twin has been observed in 14 scans.

Grain areas are directly calculated from the number of experimental points of the same orientation with a step size, equal to 0.2 μm. As a result of the annealing treatment the grains are equiaxed. Grain area is computed by multiplying the number of measurement points that the grain contains by the area associated with a pixel, i.e. 0.1 μm^2. Grain diameter is estimated assuming a spherical grain. The software developed by Pradalier et al. [30] fits an ellipse to each twin. The measured twin thickness is defined as the minor axis of the ellipse. The true twin thickness is then estimated by multiplying the measured twin thickness by the cosine of the angle formed by the twin plane, K_1, and the normal to the sample surface [31,29]. Because EBSD scans do not provide access to local stresses before unloading and sectioning, for classification purposes the geometric Schmid factors (SF) are computed from the inner product of the symmetric Schmid tensor and the normalized macroscopic stress tensor, such that $\|\Sigma\|^2 = \sum_{i=1}^{3}\sum_{j=1}^{3}\Sigma_{ij}^2 = 1$. In addition, similar statistics using the macroscopic stress for computations of distributions can be produced from the use of either full-field or mean field models. The symmetric Schmid tensor is defined as the symmetric part of the dyadic product between the Burgers vector and the normal vector to the deformation plane. For each twinned grain, the six possible twin variants of each twinning mode are classified in order of decreasing SF.