Heterogeneous deformation in single-phase Zircaloy 2

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Single-phase hexagonal Zircaloy 2 was subjected to near plane strain deformation. The deformed structure had two types of grains: (i) deforming and (ii) non-deforming. Typically grains of the second type were larger and remained nearly equiaxed even after 50% deformation. They also had low grain average misorientation and were estimated to be elastically harder. Residual stress developments were mostly concentrated in the near basal orientations – orientations approximately corresponding to the non-deforming grains.

Keywords: Zirconium; EBSD; Deformation; Residual stress; Texture

An exhaustive literature exists on heterogeneous deformation in single-phase metals and alloys [1–11]. Such studies are mostly concerned with differences in the extent of deformation, and the corresponding differences in microstructural developments.

Real heterogeneous deformation, where different grains/phases of the microstructure have remarkably different strains, has been reported [12–14] only in multi-phase alloys. For example, in any commercial alloy the hard constituent or dispersoid particles are often non-shearable and strain is accommodated mainly by the matrix phase [12]. In two-phase Zr [14], the softer second phase of only 15% of the total volume can effectively accommodate the entire strain.

Zircaloy 2, the chemical composition of which is given in Table 1, is used in nuclear power reactors for tubes and claddings [15]. Its typical microstructure involves equiaxed or elongated grains (based on prior processing) of a hexagonal close packed (hcp) structure, with submicron intermetallic precipitates. Such a Zircaloy 2, with equiaxed grain structure, was subjected to near plane strain deformation. The objective of the present study was to identify the extent of heterogeneous deformation in hcp zirconium.

Fully recrystallized single-phase Zircaloy 2, with nearly equiaxed grains (see Fig. 1a), was cold rolled in a laboratory rolling mill to 20% and 50% reductions. Samples from the mid-thickness sections of the rolled plates were electropolished using standard techniques [14] and then subjected to electron backscattered diffraction (EBSD) measurements and X-ray diffraction (XRD). The EBSD were performed using a TSL-OIM package on a Fei quanta-200 HV scanning electron microscope (SEM), while a panalytical MRD system was used for XRD measurements. Instead of measuring relative intensity, as in the case of conventional X-ray texture, the residual stress values were estimated using Reuss's model according to a methodology described elsewhere [16], at different goniometer angles. Using four different pole figures (of residual stress values) and the WMIV method [17], residual stress distribution functions were calculated.

Figure 1 shows the typical microstructures of the Zircaloy 2 before (Fig. 1a) and after (Fig. 1b) the cold rolling. The IQ (image quality of the EBSD patterns) maps clearly indicate the existence of two types of grains in the deformed structure (Fig. 1b): (i) grains which had undergone refinement in size and a drop in IQ and (ii) larger grains with superior IQ. A superior IQ qualitatively indicates [18] a smaller presence of defects/dislocations. It should be noted that the present grade of Zircaloy 2, during room temperature near plane strain deformation, does not exhibit deformation twinning.

Table 1. Chemical composition (in wt.%) of the alloying elements of Zircaloy 2

Sn	Fe	Cr	Ni	O	Zr
1.54	0.15	0.12	<0.05	0.12	Balance

Equiaxed type (ii) grains were observed, albeit to a lesser extent, even after 50% reduction and in all cross-sections (see Fig. 2). The equiaxed shape of many such grains indicates insignificant macroscopic strain.

As shown in Figure 3, the two types of grains had different orientations. Type (ii) grains were mostly near basal, while type (i) grains had a range of orientations (though developments of certain preferred orientations are visible). A distinction between the two types of grains could easily be established from their grain size. The starting grain size was between 2 and 40 μm, and the deformation (as shown in Figs. 1b and 2) refined the grain size for some of the grains. Grains below 3 μm in size were considered to be grains which had undergone such refinement – termed generically as "deforming" grains. The grains above 3 μm, on the other hand, were classified as "non-deforming" grains. Such a classification allowed the easy partitioning of EBSD data (data from several scans). Further analysis on the partitioned data is given in Figures 4 and 5.

As shown in Figure 4, the non-deforming grains had an insignificant average grain misorientation (see Fig. 4a), while the smaller deforming grains had a lower grain orientation spread (see Fig. 4b). Figure 5 extends the distinction between the two types of grains to orientation estimated [19] quantities of elastic modulus and Taylor factor. These can, respectively, be taken as resistance to elastic and plastic deformation. As shown in

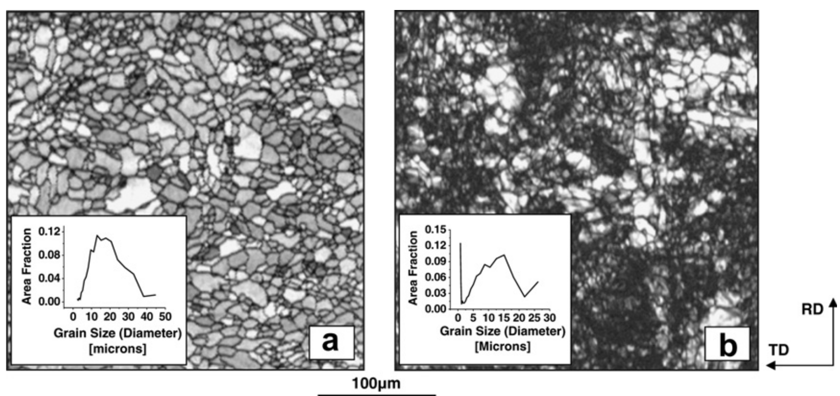

Figure 1. IQ maps of: (a) prior deformation microstructure and (b) structure after 20% cold deformation. Insets provide grain size distributions. The rolling (RD) and transverse (TD) directions are marked.

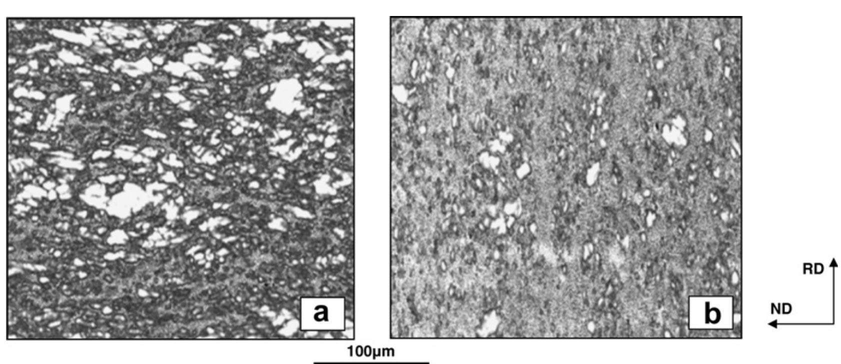

Figure 2. IQ maps of: (a) 20% and (b) 50% cold-deformed Zircaloy 2. The rolling (RD) and normal (ND) directions are marked. Even in (b), many (but not all) of the 'larger' grains remain equiaxed.

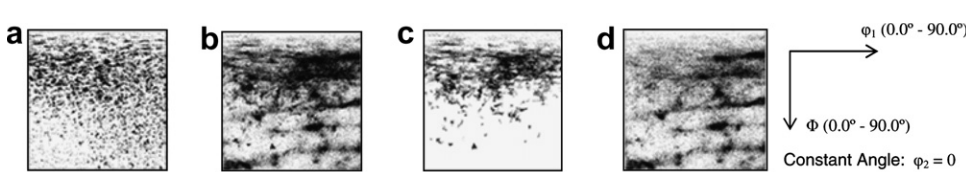

Figure 3. Euler space plots of $\phi_2 = 0°$ section representing the EBSD data points of: (a) the starting structure, (b) the structure in (a) after 20% cold reduction, (c) non-deforming grains in (b), and (d) deforming grains in (b). The distinction between (c) and (d) was made from grain size: (c) above 3 μm and (d) below 3 μm in size.

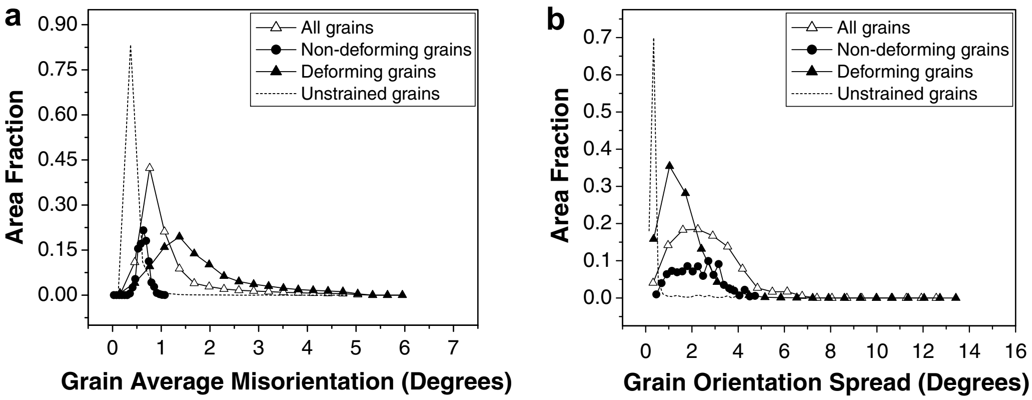

Figure 4. (a) Grain average misorientation (GAM) and (b) grain orientation spread (GOS). These were estimated by providing a criterion for the grain boundary (misorientation exceeding 15°) and then isolating the grains. From such "isolated" grains GAM (misorientation between neighboring points of a grain) and GOS (misorientation between all measurement points of a grain and the grain average orientation) were estimated. Corresponding values for deforming (below 3 μm), non-deforming (above 3 μm) and all (deforming + non-deforming) grains are listed. The dotted line, representing unstrained/undeformed alloy, shows the limits of measurement uncertainty.

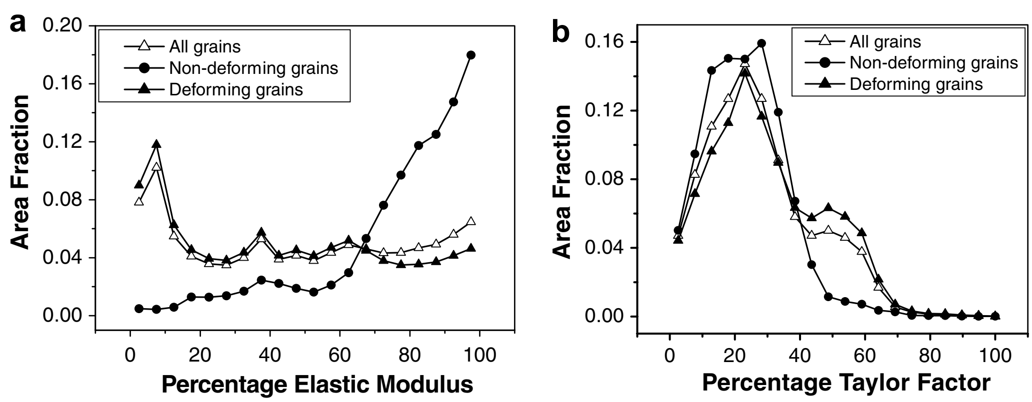

Figure 5. Orientation estimated [19] values of (a) elastic modulus and (b) Taylor factor for deforming (below 3 μm), non-deforming (above 3 μm) and all (deforming + non-deforming) grains. Both (a) and (b) are given in percentages, so that an easy basis for comparison is made.

Figure 5, though no distinction could be made in terms of Taylor factor, clearly the non-deforming grains were elastically harder.

To expand the picture of heterogeneous deformation further, residual stress values were measured for different goniometer angles and for the respective poles of (0004), $(01\bar{1}4)$, $(01\bar{1}5)$ and $(11\bar{2}4)$ in an Eulerian texture cradle [16,20–22]. In general, near basal orientations had more residual stress (five times or more) and Figure 6 plots the compressive residual stress distribution in the $\phi_2 = 0°$ section. A similar trend (though with lesser preference for basal) was observed for tensile residual stress. The trend in Figure 6 is clear – residual stress was concentrated on near basal orientations.

Heterogeneous deformation of polycrystalline metallic materials is a subject of considerable applied and academic interest. An exhaustive range of the published literature [1–14] exists on this subject. Typically in single-phase polycrystalline metals, differences in strain are observed between different grains and also inside the same grain. The other extreme example is in the case of multi-phase alloys, where strain can be fully accommodated in one phase – keeping the other phase free from macroscopic strain [12,14]. Such an extreme is, however, unheard of in single-phase alloys. The present study did stumble onto this unexpectedly.

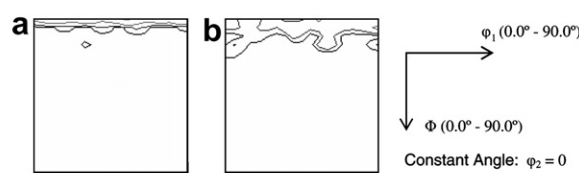

Figure 6. Plots showing compressive residual stress distribution at $\phi_2 = 0°$ section of the Euler space for (a) 20% deformed and (b) 50% deformed material. Contour levels are drawn at 1, 3, 7, 15 and 20 times the average residual stress values.

Deformed Zircaloy 2, the material used in the present study, clearly has two types of grains: deforming and non-deforming. This classification, though simplified in approach, is based on refinement in size. The classification can also be extended to changes in grain shape or aspect ratio and to developments in grain average misorientation (see Figs. 1, 2 and 4a). For example, near basal grains, $(01\bar{1}5)$ $\langle uvtw \rangle$ being the average orientation, did not become fragmented or changed their shape – they acted more like non-shearable particles. Difference in elastic stiffness, and not in plasticity, appears to be the source of this "extreme" heterogeneous deformation, and its immediate effect was in the heterogeneous distribution of residual stress. In the coming days more experimental and theoretical inputs are expected on this phenomenon; the present report will perhaps precipitate such studies.

The deformed microstructures could easily be distinguished as deforming and non-deforming grains. Non-deforming grains were of larger size and with insignificant grain average misorientation. Even after 50% reduction in thickness, they remained nearly equiaxed. Residual stress developments were largely concentrated on the near basal orientations, the approximate band of orientations corresponding to the non-deforming grains.

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