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Investigations of orientation and length scale effects on micromechanical responses in polycrystalline zirconium using spherical nanoindentation

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ABSTRACT

Here we investigate the elastic and plastic anisotropy of hexagonal materials as a function of crystal orientation using a high-throughput approach (spherical nanoindentation). Using high purity zirconium as a specific example, we demonstrate the differences in indentation moduli, indentation yield strengths and indentation post-elastic hardening rates over multiple grain orientations. These results are validated against bulk single crystal measurements, as well as data from cubic materials. By varying the indenter size (radius), we are also able to demonstrate indentation size effects in hexagonal materials, including possible signatures of strain hardening due to twin formation in the nanoindentation stress–strain curves.

Nanomechanical testing techniques, such as nanoindentation [1–3], micropillar compression and tension [4,5], as well as three point bend and cantilever bend techniques [6–8], have been used for the past couple of decades to probe small volumes of material of the order of 1 μm^3 or smaller. In techniques other than nanoindentation, preparation of miniaturized tensile and compression samples is typically carried out in bulk materials using Focused Ion Beam (FIB) milling, a time consuming technique that runs the risk of ion beam damage to the sample [9,10]. Nanoindentation, on the other hand, provides copious amounts of mechanical data from extremely small volumes of material while only requiring a flat polished surface [11]. Much work has also been carried out over the past few years to obtain stress–strain curves using a spherical nanoindentation approach [2,11–13]. This technique can transform the raw load–displacement data into meaningful indentation stress–strain curves that capture the local loading and unloading elastic moduli, local indentation yield strengths, and post-yield strain hardening behavior. As such, spherical nanoindentation represents a high-throughput technique that can amass enormous amounts of grain-level data from one polycrystalline sample.

Early work utilizing this technique focused largely on orientation dependent local (grain-scale) mechanical responses in relatively low anisotropy cubic systems such as face centered cubic (fcc) Aluminum and body centered cubic (bcc) Tungsten [2,12,14]. More recent studies have explored more elastically anisotropic cubic materials such as Fe–3%Si steel [15,16], the work-hardening in grain-boundary regions in polycrystalline metallic samples [17–19], and the influence of ion implantation on damage gradients relative to the sample surface in Tungsten [13]. In this work we report on the use of our recently developed spherical nanoindentation stress–strain techniques on materials with non-cubic crystal structure that deform extensively via twinning. While nanoindentation of hexagonal materials have been previously reported using both pyramidal [20] and spherical indenters [21,22], the use of indentation stress–strain analysis is advantageous in that it allows us to study not only the commonly reported modulus and hardness values in these materials, but also their yield strengths under indentation, as well as their post-yield hardening response and how the indentation length scale (controlled by changing the indenter tip radius) affects their propensity of twinning under the indenter.

Hexagonal close packed (hcp) metals such as zirconium (Zr) represent increasing complexity in mechanical behavior when compared to the relatively symmetric slip inherent to cubic systems. Zirconium's mechanical behavior has been studied extensively, and is known to be highly anisotropic compared to cubic metals showing varying degrees of dislocation slip and twinning depending upon loading direction [23–26]. In this vein, polycrystalline Zr is the ideal test bed for a spherical nanoindentation investigation into microscale mechanical anisotropy. The current work leverages modern high-throughput nanomechanical test techniques to determine differently oriented single crystal stress–

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strain responses of Zr at different length scales (by varying the indenter tip radius; all length scales studied here are significantly smaller than the millimeter sized grains in the sample). Note that in the current study only three carefully polished samples of Zr will be required to explore the complete range of orientations in hcp Zr (see Fig. 1), in comparison to the dozens of painstakingly prepared single crystal samples needed by past researchers.

In this study, spherical nanoindentations with different indenter tip radii were conducted on ~50 grains distributed in three samples [27] (see Supporting Information section). Indentation stress–strain curves were computed from the corresponding raw load–displacement datasets following the procedures outlined in Eqs. S1 and S2 [11,12]. The results from four representative grains, noted here as Grains 1, 2, 3, and 4, which have sufficiently differing crystallographic orientations measured using electron backscatter diffraction (EBSD) that are spread over the fundamental orientation zone (depicted as the inverse pole figure (IPF) "triangles" in Fig. 1) are discussed in detail in this work. The orientation of Grain 1 provides for an indentation direction almost parallel to the crystallographic c-axis, and that of Grains 3 and 4 almost perpendicular to the c-axis, while Grain 2 lies in the center of the IPF triangle. Fig. 1d shows a relative comparison of the indentation load–displacement responses measured with a 10 μm indenter radius between these four grains. As expected, Grain 1 shows the strongest response, as it invokes the hardest deformation modes available in Zr.

In Fig. 2, the extracted indentation stress–strain curves for Grain 1 are shown for spherical indenters ranging in radii from 10 μm to 100 μm. For the larger indenter sizes (of radius 100 μm), no pop-ins are evident, and the indentation yield stress (Y_{ind}, measured as a 0.2% offset stress) is 1279.7 ± 151 MPa (see Table 1). For the smaller 10 μm radius indenter, there is typically a large pop-in event (sometimes followed by several smaller pop-ins), and a subsequent steady-state work hardening at a rate of 11.2 ± 0.7 GPa. The work hardening rate was calculated as the linear slope of the indentation stress–strain curve between offset strains of 1 and 2%.

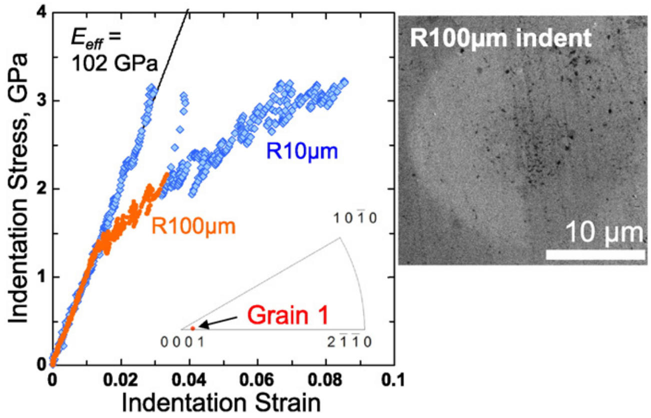

Fig. 2. Spherical nano-indentation stress strain response of Grain 1, measured with two indenters of radii 10 and 100 μm. Inset. Orientation of Grain 1 on the IPF triangle. Right image: residual imprint from a 100 μm radius indenter on Grain 1.

Fig. 3 shows the corresponding indentation stress–strain responses for Grains 2, 3 and 4 measured with five different indenters of 1, 5, 10, 100 and 150 μm radii. These grain orientations, which range from indentation perpendicular to the c-axis (Grains 3, 4) to another off-axis orientation (Grain 2) exhibit very different behavior than that found in the tests parallel to the c-axis (Grain 1). In contrast to the c-axis grains, Grains 2, 3 and 4 show distinct length scale effects, which can be broadly grouped in three general patterns. (i) Tests with the smallest sized indenter (1 μm radius) show a series of pop-ins that continue for the entire duration of the test (until a strain of over 0.25, green curves

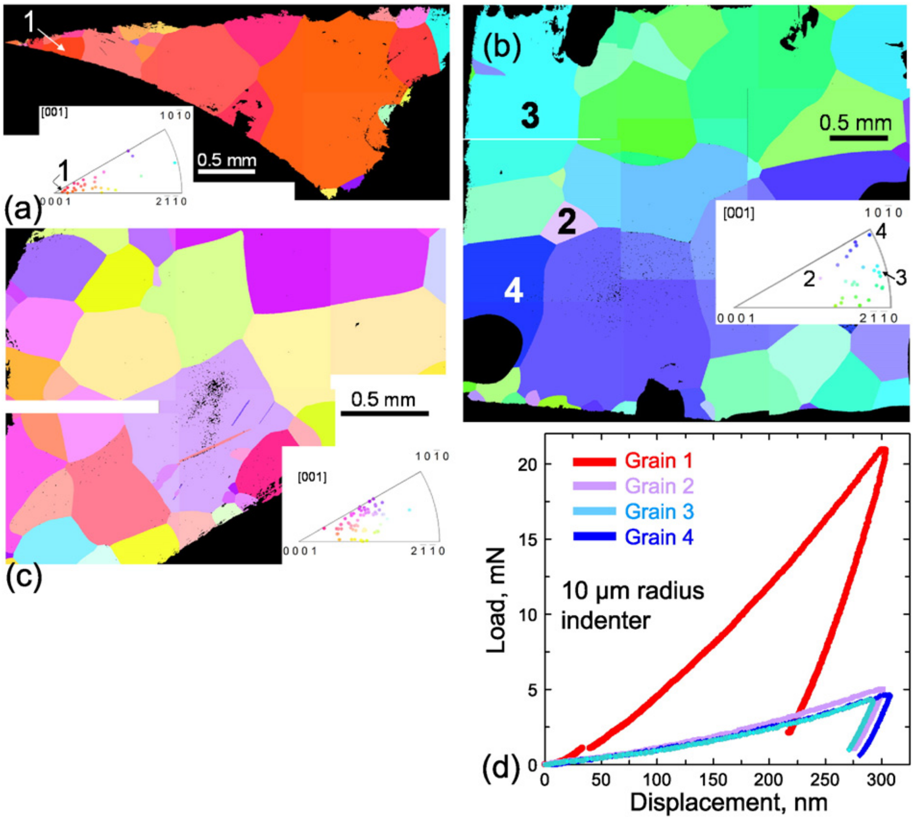

Fig. 1. (a–c) Montage of assorted EBSD micrographs of annealed zirconium. (d) Comparison of the load–displacement responses between Grains 1 to 4 using a 10 μm radius spherical indenter.