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Grain-scale measurement of slip resistances in aluminum polycrystals using spherical nanoindentation

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Abstract—In this work, we develop and demonstrate novel protocols based on spherical nanoindentation and orientation image mapping (OIM) for quantifying the local increases in slip resistances in the individual grains of a deformed (or strain hardened) polycrystalline sample. These new protocols utilize the recently developed data analyses methods for extracting indentation stress–strain (ISS) curves in conjunction with the measurements of the local crystal orientations at the indentation sites using the OIM technique. The proposed protocols involve two main steps. In the first step, spherical nanoindentation measurements are conducted on fully annealed samples of the material of interest to map out the functional dependence of the indentation yield strength (Y_{ind}) on the crystal lattice orientation in the annealed condition. In the second step, spherical nanoindentation and OIM measurements are conducted on the deformed samples of the same material and are analyzed rigorously to reliably estimate the increase in the local slip resistance at the indentation sites. The function established in the first step is utilized in the second step to properly account for the influence of the local crystal orientation on the measured Y_{ind} in the deformed sample. This novel measurement and data analysis protocol is demonstrated in this paper on polycrystalline samples of high purity aluminum. From this study, it was noted that the influence of the crystal lattice orientation on the measured Y_{ind} in Al crystals can be as high as 40%, with the lowest values corresponding to the [1 0 0] (cube) orientation and highest values corresponding to the [1 1 1] orientation. The measurements on the deformed samples showed a significant variation in the strain hardening rates in the individual grains of the polycrystalline sample. A positive correlation was observed between the percentage increase in the local slip resistance and the value of the Taylor factor computed for the local crystal orientation at the indentation site subjected to the macroscale imposed deformation.

Keywords: Spherical nanoindentation; Orientation imaging; Indentation yield; Taylor factor; Indentation stress–strain curves

1. Introduction

Over the last decade, nanoindentation [1–4] has emerged as a tool of choice for investigating mechanical behavior of hierarchical materials, since the interrogation volume under the indenter can be varied systematically by roughly three orders of magnitude in length scales in the range of 50 nms to 50 μms. While the indentation experiments are easy to perform and require minimal sample preparation (compared to other small scale mechanical testing options such as micro-pillar testing [5–7]), the data analysis and interpretation is quite complicated, mainly due to the complex continuously evolving stress state under the indenter tip. Traditionally, indentation experiments have been carried out with sharp tips [8–10], and the values of local elastic modulus and hardness were extracted mainly from an analysis of the unloading portion of the test segment [11–13]. However, recent advances in instrumentation (e.g., the availability of the continuous stiffness measurement (CSM) [14]) have now made it possible to convert the measured load–displacement data from spherical nanoindentation into highly reproducible and consistent indentation stress–strain (ISS) curves [15]. Examining the indentation data in the form of ISS curves provides better insights into the local mechanical response in the sample (although it still needs to be interpreted as an average over the indentation zone experiencing highly heterogeneous stress/strain fields). More specifically, it was demonstrated that these new protocols produce meaningful information such as the local indentation modulus and the local indentation yield strength (Y_{ind}). In recent works, the use of ISS curves for nanoindentation data analysis has demonstrated tremendous promise in providing new insights into material behavior, including the role of grain boundaries during macroscale deformation in metals [16], buckling behavior of carbon nanotube forests [17], and lamellar level properties in bone [18].

In this work, we build on prior work from our research group that focused on the characterization of the changes in the local slip resistance in deformed polycrystalline samples of cubic metals [19]. The main concept underlying these new protocols was already introduced in our earlier paper, and involved the utilization and combined analysis of

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measured data from both spherical nanoindentation and orientation imaging microscopy (OIM) [20,21]. This particular analysis protocol was prompted by the recognition that the measured Y_{ind} in deformed samples is influenced by both the local crystal lattice orientation and the local dislocation density in a multiplicative manner (details presented later). In order to isolate the contribution from the local dislocation density, it becomes essential to establish a protocol that correctly accounts for the effect of the crystal lattice orientation at the indentation site. Consequently, in our prior work, we establish a new protocol that approached the task in two distinct steps. In the first step, the focus was placed on the annealed samples of the material system of interest (even though our interest is really in the deformed samples), where there is negligible dislocation density in the samples. In this way, one can map out the functional dependence of the Y_{ind} on the local crystal orientation in the indentation zone. Subsequently, in the second step of the protocols, the focus is shifted to the deformed samples of actual interest. However, the information gathered from the first step (on the annealed samples) is critically important in relating the measured Y_{ind} to the local percentage increase in the slip resistance at the indentation site. It should be noted that this protocol provides a single value for the effective (or the averaged) slip resistance at the indentation site and is therefore most easily interpreted for cubic metals, where it is reasonable to assume that the slip resistance of the different slip systems at the material point of interest are roughly equal to each other. Extension of this protocol to hexagonal metals (with a multitude of slip and twin systems with large variations in their corresponding slip and twin resistances even at a single material point) needs a more detailed analysis.

Our prior work [19] explored these new concepts on polycrystalline Fe–Si samples that exhibit a body-centered cubic (bcc) structure. Although the earlier work established a strong foundation, much additional development is needed to improve the fidelity and robustness of these novel protocols. In this study, we have undertaken a much more extensive effort with the following salient distinctions from the earlier work: (i) The present study was performed on polycrystalline samples of high purity Aluminum (Al), which exhibits a face-centered cubic (fcc) structure. The main reasons for the selection of Aluminum were the relatively simple set of well-defined slips systems (the bcc metals exhibit pencil glide where multiple planes possessing the (1 1 1) directions could serve as potential slip systems) and very low elastic anisotropy. Both of these factors are expected to simplify significantly the subsequent analyses of the data presented here. (ii) The samples selected for the present study have been much more systematically processed, corresponding to 0%, 10%, and 20% reductions in plane strain compression. In the earlier work, the samples corresponded to 0%, 30%, and 80% reductions, which made it very difficult to correlate the increases in slip resistances to the amount of cold work imposed on the sample. (iii) The number of measurements performed within a single orientation as well as the number of orientations tested in both annealed and deformed conditions are both substantially larger in this study. Consequently, the present study allowed for a much more meaningful statistical investigation of the correlations sought in the study. (iv) A number of the steps involved in the protocols have been refined to increase the reliability and robustness of the method. For example, it was discovered that the use of a 100 μm radius indenter tip in the measurements on the annealed sample helped minimize the uncertainty introduced into the analyses as a consequence of the unavoidable pop-ins in these tests. In a similar vein, the protocols for extracting the Y_{ind} and for capturing mathematically the functional dependence of the Y_{ind} on the crystal lattice orientation were both substantially refined in the present study.

The overall study presented here is aimed at gaining quantitative insights into the strain hardening rates in the individual grains of a polycrystalline sample. These measurements are critical for maturing physics-based crystal plasticity models [22–25]. Toward this goal, we present our measurements from the study and our analyses of these results. The measurements are provided as tables in this paper to allow other researchers to conduct their own analyses of the main experimental results obtained here using different assumptions, approximations, and crystal plasticity models.

2. Indentation stress–strain (ISS) curves

The procedures used in this study to convert the raw load–displacement data in spherical nanoindentation into ISS curves have been detailed in prior publications [15,26], and are briefly reviewed here. The procedure, based on Hertz theory [27,28], involves the accurate determination of the effective zero point and the computation of the indentation stress and strain values. The effective point of initial contact (may or may not correspond to the actual point of contact) [15] is identified such that the initial elastic loading data segment immediately following this point provides the best agreement with Hertz theory for all three measured signals: the load ($\tilde{P}$), the displacement ($\tilde{h}$), and the contact stiffness (S). For spherical nanoindentation, this search for the effective initial point can be accomplished using the following relationship based on Hertz theory:

$$S = \frac{3P}{2h_e} = \frac{3(\tilde{P} - P^*)}{(\tilde{h} - h^*)} \tag{1}$$

where P^* and h^* denote the load and displacement values respectively at the point of initial contact. Linear regression can be used to determine the effective point of initial contact or the so-called effective zero-point (i.e., the values of P^* and h^*).

The radius of contact (a) can be estimated as:

$$a = \frac{S}{2E_{eff}}, \quad \frac{1}{E_{eff}} = \frac{1-\nu_s^2}{E_s} + \frac{1-\nu_i^2}{E_i} \tag{2}$$

where S is the harmonic contact stiffness. E_{eff} is the effective stiffness of the sample-indenter system, E and ν are Young's modulus and Poisson's ratio, and the subscripts i and s denote the indenter and sample, respectively. The term $\frac{E_s}{1-\nu_s^2}$ is generally referred to as the sample indentation modulus, denoted by E_{ind}. The value of E_{ind} is estimated from an analyses of the initial loading segment (identified in the zero-point analyses described above) using Hertz theory. The values of the indentation stress (σ_{ind}) and the indentation strain (ε_{ind}) for the initial elastic loading segment are then computed as:

$$\sigma_{ind} = \frac{P}{\pi a^2}, \qquad \varepsilon_{ind} = \frac{4}{3\pi}\frac{h_e}{a} \approx \frac{h_e}{2.4a}, \qquad \sigma_{ind} = E_{ind}\varepsilon_{ind}, \tag{3}$$