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Studies of grain boundary regions in deformed polycrystalline aluminum using spherical nanoindentation

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ABSTRACT

In this work, we use novel protocols based on spherical nanoindentation and orientation imaging microscopy (OIM) to quantify the local changes in slip resistances in the grain boundary regions of deformed, polycrystalline aluminum. The new protocols involve the use of the recently developed methods for extracting indentation stress–strain (ISS) curves from raw nanoindentation data in conjunction with the measurement of local microstructure at the indentation site using OIM to study the changes in the local slip resistances as a function of distance from the grain boundaries. Eight grain boundaries were selected for this work such that they included a broad range of boundaries, including low and high (grain-to-grain misorientation) angle boundaries as well as low, moderate, and high deviations in the Taylor factors of the grains on either side of the boundary. It was concluded that there was additional hardening in the grain boundary region when a Taylor 'soft' grain was present next to a Taylor 'hard' grain. This hardening was consistently observed on the soft grain side with one exception where hardening was observed on both sides of the boundary. A positive correlation was observed between the difference in Taylor factor across the boundary and the amount of hardening on the 'soft' grain side. However, no correlation was observed between the grain-to-grain misorientation angle and the extent of hardening at the grain boundary.

1. Introduction

Mesoscale interfaces in materials, including grain boundaries, play a significant role in determining the mechanical properties of materials. The Hall-Petch effect (Hall, 1951; Petch, 1953), discovered over sixty years ago, provides a robust but empirical relationship between the macroscopic yield strength and average grain size in a material. However, there is still very little understanding about the precise role of grain boundaries during plastic deformation and there is no consensus on the correct physics-based model for predicting this effect (Cottrell, 1958; Meyers and Ashworth, 1982). Moreover, the Hall-Petch effect, captures only the homogenized effect of grain boundaries on the macroscale mechanical properties of metallic materials. In other words, this simple model does not pay attention to the difference in the behavior of the different type of boundaries. Various criteria have been put forth to describe the behavior of grain boundaries based on grain boundary

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character (Adams et al., 1994; Tsurekawa et al., 1994; Watanabe and Tsurekawa, 1999), interfacial structure (CSL boundaries) (Watanabe and Tsurekawa, 1999; Palumbo et al., 1991; Dahlberg et al., 2013), grain boundary energy (special boundaries) (Rohrer et al., 2004; Tschopp and McDowell, 2007), and slip transfer rules (Clark et al., 1992; de Koning et al., 2003; Bieler et al., 2009). However, these dependencies are not yet established quantitatively. An accurate understanding of the role of grain boundaries is critical for the development of robust physics-based crystal plasticity models (Asaro, 1983; Kalidindi et al., 1992; Kalidindi, 1998; Van Houtte et al., 2002) to accurately predict material behavior during and following plastic deformation of polycrystalline materials.

Lack of rigorous protocols for reliably capturing the mechanical response at the sub-micron length scales of interest constitutes the foremost technical gap in efforts aimed at advancing the fundamental understanding of the role of grain boundaries during plastic deformation. Approaches such as in-situ testing of micro-pillars (Ng and Ngan, 2009; Kunz et al., 2011; Dietiker et al., 2011; Uchic et al., 2004) have an inherent drawback in that these methods require sophisticated equipment and are effort intensive, and therefore cannot be used to test a large number of interfaces in a cost and time efficient manner. Alternatively, instrumented indentation testing (both nano and micro) (Fischer-Cripps, 2002; Fischer-Cripps, 2001, 2006; Hay and Pharr), when combined with improved data analyses protocols provides a high throughput approach suitable for addressing this challenge. The use of nanoindentation testing for characterizing grain boundary regions has been reviewed in Ref. Kalidindi and Vachhani (2014).

Recent advances in the analysis of spherical nanoindentation data now provide a means of extracting very reliable and repeatable measurements of local mechanical properties at a sub-micron length scale (Kalidindi and Pathak, 2008; Pathak et al., 2009c, 2012a, 2009a). This local mechanical behavior data can be combined with local structure information obtained using complementary techniques such as orientation imaging microscopy (OIM) to further our understanding of local structure–property relationships in metallic materials and their evolution during macroscopically imposed plastic deformation. In previous work (Vachhani and Kalidindi, 2015), we studied the evolution of slip-resistances within individual grains (i.e., in the grain interiors) during deformation in polycrystalline aluminum (Al). In this work, we have conducted a detailed study into the evolution of local mechanical properties in the grain boundary regions in plastically deformed samples of polycrystalline Al. As mentioned earlier, this information is critical to the development of physics-based multiscale models for plastic deformation in polycrystalline metals. Pathak et al. (2012b) have already demonstrated the viability of the combined spherical nanoindentation-OIM testing and analysis protocols in quantifying changes in mechanical properties in the grain boundary regions of a deformed Fe- 3% Si steel sample. In that work, three specifically selected grain boundaries were studied in 30% deformed samples. In this work, we have systematically investigated regions around eight grain boundaries in a high purity aluminum sample deformed by plane strain compression to obtain a 20% reduction in height. The smaller strain level and the larger number of grain boundaries were selected for the present study with the specific goal of quantitatively relating salient grain boundary parameters to the measured changes in the local mechanical properties in the grain boundary regions. The salient grain boundary parameters explored in this work are the grain-to-grain misorientation across the boundary and the difference in the Taylor factors. Although both parameters are related to the orientations of the grains adjoining the grain boundary, the Taylor factors provide a more direct measure of the influence of the crystal lattice orientation on the plastic flow stress, while accounting for the details of the imposed deformation mode (typically expressed as a stretching tensor). Historically, there has been a general hypothesis in prior literature that the strain hardening in the grain boundary region would be strongly influenced by the grain-to-grain misorientation across the boundary (Zaefferer et al., 2003; Ma et al., 2006; Dalla Torre et al., 2007). However, some prior studies (Vachhani and Kalidindi, 2015; Kalidindi et al., 2004; Bhattacharyya et al., 2001; Pathak et al., 2012b) have highlighted the possibility that the difference in the Taylor factors across the grain boundary might play a more influential role in grain boundary plasticity as it explicitly captures the relative capability of the grains involved in accommodating the imposed plastic deformation. In order to evaluate critically the relative roles of both these grain boundary parameters, the grain boundaries for the present study were specifically selected to include different combinations of grain-to-grain misorientation across the boundary and the difference in Taylor factors.

2. Spherical nanoindentation and indentation stress–strain (ISS) curves

Nanoindentation is a versatile tool for measuring the mechanical properties from small material volumes (Tabor, 1951). Traditionally, indentation experiments have been carried out with sharp tips (Bucaille et al., 2003; Rho et al., 1997; Poole et al., 1996), and the values of local elastic modulus and hardness were extracted mainly from an analysis of the unloading portion of the test segment (Fischer-Cripps, 2001; Oliver and Pharr, 1992, 2004). However, recent advances in instrumentation (e.g., the availability of the continuous stiffness measurement (CSM) (Li and Bhushan, 2002)) 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 (Kalidindi and Pathak, 2008). It has been demonstrated that these new protocols produce reasonable estimates of the indentation modulus and the indentation yield strength (Y_{ind}). The procedures used to convert raw indentation data into ISS curves and extract Y_{ind} from the ISS data have been detailed in prior publications (Kalidindi and Pathak, 2008; Vachhani and Kalidindi, 2015; Pathak et al., 2009d). A brief overview of this strategy is provided here.

The methodology for converting raw spherical nanoindentation data into ISS curves is largely based on Hertz Theory (Hertz, 1896; Johnson, 1985; Sneddon, 1965). The first step involves identification of the effective zero point, such that all the measured signals provide the best agreement with the Hertz theory beyond this point. If $\tilde{P}$, $\tilde{h}$ and S are the measured load,