Local Mechanical Property Evolution During High Strain-Rate Deformation of Tantalum

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Abstract Damage within ductile metals is often linked to local heterogeneities. In ductile metals, damage typically occurs after plastic deformation, which evolves the microstructure and its properties in ways that are not easily measured in situ. This is particularly true for materials subject to dynamic loading. Here, we use a combination of spherical nanoindentation testing and electron microscopy to quantify changes in local dislocation slip resistance as a function of grain orientation in polycrystalline tantalum subjected to high strain-rate deformation. A nanoindentation data analysis technique is used to convert spherical nanoindentation data into stress–strain curves. This technique works with microstructural characterization at the indentation site and involves two steps: (1) determination of the functional dependence of the indentation yield strength (Y_{ind}) on the crystal orientation in the undeformed condition, and (2) use of nanoindentation and EBSD measurements on the deformed samples to determine changes in the local slip resistance. In this work, undeformed Ta had indentation yield values that varied by as much as 40% depending on the crystal orientation. The dynamically deformed Ta displayed a large variance in the strain hardening rates as a function of grain orientation. Soft grains (those with low Taylor Factor) were found to

harden significantly more as compared to hard grains (those with a high Taylor Factor). These data are discussed in terms of grain interactions where the hard grains impose additional work on neighboring soft grains due to constraint at the boundaries.

Keywords Dynamic deformation · Tantalum · Nanoindentation · Indentation stress–strain · Split Hopkinson Pressure Bar

Introduction

A number of engineering applications subject structural materials to the extreme environment of dynamic loading. Components designed for defense, aerospace, automotive and even industrial applications can experience high stresses and high strain rate loading conditions that are characteristic of impact or shock loading. Furthermore, this type of loading can lead to catastrophic failures. For this reason, the role of dynamic loading on damage evolution and failure has received a great deal of attention.

Tantalum is considered to be an excellent material for several dynamic applications because of its high density, superior strength, and dynamic ductility over a wide range of temperatures and strain rates [1–3]. Tantalum exhibits temperature and strain-rate sensitivity that is typical of many bcc metals [4–13]. This is attributed to a high thermal activation for overcoming Peierls–Nabarro stress barriers within the material [14, 15] and deformation that is governed by planar dislocation slip for a wide range of loading conditions. Under dynamic conditions such as plate impact loading, it has been shown that void nucleation and growth is influenced by plastic processes [16–18]. This plasticity also affects local properties of the material. How

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these evolved properties influence void nucleation and growth, has not been quantified.

Furthermore, shear localization is often a mechanism for damage evolution and failure in ductile materials including tantalum [18–27]. Catastrophic shear is enhanced under high-strain rate conditions because of the lack of time for thermal diffusion leading to suppressed work hardening and localized softening [19]. Microstructure and substructure evolution leading to the formation of shear bands has been the subject of several studies [20–28], mainly utilizing optical microscopy, TEM and EBSD [29] techniques to characterize the deformation. As with void nucleation, another equally important attribute, is the evolution of the local mechanical properties leading up to and occurring during shear localization. This lack of understanding regarding the evolution of the local mechanical behavior during plastic deformation is a critical, missing piece in current crystal plasticity and damage models. In general, existing models fail to accurately capture the local features and statistical distribution of heterogeneous, plastic deformation [30, 31].

Nanoindentation testing [32, 33], with its high spatial resolution, is an ideal tool for interrogating the mechanical response in materials at the nano-scale length scale. While indentation experiments are easy to perform and require minimal sample preparation (compared to other small scale mechanical testing options such as micro-pillar testing [34–36]), the data analysis is complicated, due to the continuously evolving stress state under the indenter tip. Traditionally, indentation experiments have been performed with sharp tips, and the values of the elastic modulus and hardness are extracted from an analysis of the unloading portion of the test [37–39]. However, recent advances in instrumentation (e.g., the availability of the continuous stiffness measurement (CSM) [40]) and new data analysis protocols have made it possible to convert the measured load–displacement data from spherical nanoindentation into more meaningful indentation stress–strain (ISS) curves [41, 42]. This ISS data allows for the local elastic, yield, and post-elastic behavior of materials during the indentation process to be examined and has been shown to produce repeatable measurements [43]. Recent work involving the use of ISS curves for nanoindentation data analysis, has demonstrated promise in providing new insights into material behavior, including: quantification of the role of crystal orientation [44, 45] and grain boundaries [46, 47] during macroscale, quasi-static deformation in metals, buckling behavior of carbon nanotube forests [48], and lamellar level properties in bone [49].

Here, we extend the use of the above-described technique to understanding the evolution of microstructure and local mechanical properties in dynamically-deformed (strain rate $\sim 10^3$ /s), high-purity, tantalum. Note, Ta lends

itself readily to this initial type of study since the number of factors that can influence the evolution of microstructure during deformation, such as second phases and deformation twinning, are limited. One of the significant gaps in the understanding of polycrystalline plasticity comes from the lack of reliable information regarding how individual grains harden in polycrystalline materials during the process of accommodating an imposed macroscale plastic deformation. This hardening is also expected to be heterogeneous at the grain scale, for the same reasons that lead to heterogeneity in the grain scale microstructure. The approach utilized in this research is specifically aimed at measuring the increase in the local indentation yield strength within individual grains and correlating that increase with crystal orientation to quantify this heterogeneous response and its spatial dependency. More specifically, a combination of spherical nanoindentation and electron back-scattered diffraction (EBSD) was utilized to characterize changes in the local slip resistance in deformed, polycrystalline Ta.

This work has shown that the high purity Ta examined in this study has indentation yield values that vary by as much as 40% depending on the crystal orientation. The dynamically deformed Ta displays a large variance in the strain hardening rates among grains of different orientations. Soft grains (low Taylor factor) are found to harden more as compared to the hard grains (high Taylor factor) within the microstructure. Furthermore, soft grains neighboring hard grains harden more than soft grains surrounded by other soft grains. These data are discussed in terms of grain interactions, where the hard grains impose additional work on the neighboring soft grains due to constraint, thus resulting in enhanced hardening.

Indentation Stress–Strain (ISS) Curves

Indentation is a tool for measuring the mechanical properties from small material volumes. The analysis of nanoindentation data is based on Hertz Theory [50, 51], which describes the frictionless, linear-elastic contact between two, isotropic solids. The ISS analysis approach [41] used in this work utilizes the Continuous Stiffness Measurement (CSM) [40] option, where a dynamic, oscillating load signal (of a fixed displacement amplitude) is superimposed on the monotonically increasing load signal. Since the oscillation amplitude is relatively small, every unloading segment of the CSM oscillation is predominantly elastic and the Hertz equations can be applied to this unloading segment, even in the post-elastic deformation regime.

A summary of this approach, to convert nanoindentation data into ISS curves is provided in [43]. Briefly, this

procedure follows a two-step approach consisting of (i) the determination of an effective zero point followed by (ii) the calculation of the contact radius for its use in computing the indentation stress and strain values.

Materials and Processing

High-purity, polycrystalline Ta plate material was used in this investigation. As seen in Fig. 1a, the microstructure of the as-received tantalum is mildly textured with slightly elongated grains. The average grain size in the as-received condition is about 40 μm and the average grain aspect ratio is 0.44. The details of this tantalum plate material (chemistry, texture, grain size) can be found elsewhere [52].

For mechanical tests, right, cylindrical compression samples were cut from the as-received plate of tantalum (5 mm in diameter by 5 mm in height). A split-Hopkinson pressure bar (SHPB) was used to dynamically (strain

rates $\sim 1800/\text{s}$) impart predetermined levels of strain into these tantalum specimens. A typical stress–strain response for the tantalum examined in this study is shown in Fig. 1b. The macroscopic stress–strain response shows an initial elastic loading followed by a yield point, after which there is a drop in flow stress prior to deformation continuing at an almost constant flow stress. This drop in stress at yield is characteristic of many annealed BCC metals and alloys where high stresses are necessary to create/activate dislocation sources and/or unlock dislocations captured at interstitials before “bulk” plastic deformation occurs by more uniform dislocation slip [6]. In addition to the test shown in Fig. 1b, interrupted SHPB tests were performed at the same strain rate but to plastic strains of 3, 10 and 20%. These tests were performed using tool steel, stopper rings around compression specimens in a way that is similar to the methods described in [22–24]. All specimens tested in the SHPB were sectioned along the compression axis and mounted in epoxy. Since a high quality surface

Fig. 1 **a** Starting microstructure and **b** Typical stress–strain response for high purity polycrystalline tantalum deformed in a split Hopkinson bar at a strain rate of $\sim 1800/\text{s}$. Also shown is the IPF and the GOS maps for the microstructures at four different levels of strain

finish is critical for both nanoindentation [53] and EBSD, mechanical grinding was followed by polishing with alumina and subsequently silica suspensions. The samples were etched using a mixture of water (10 ml), nitric acid (10 ml), hydrochloric acid (10 ml) and hydrofluoric acid (5 ml) to remove the surface, worked layer remaining from polishing.

Grain orientation maps were obtained via EBSD. EBSD scans, with a 0.1 μm step size, were collected using an FEI XL-30 scanning electron microscope equipped with the TSL/OIM EBSD system using an accelerating voltage of 20 kV. Data processing utilized the built-in tools in the TSL/OIM Data Analysis software package. Each scan was cleaned by removing all points with a confidence index of less than 0.05. For each of the deformed tantalum samples, inverse pole figure (IPF) maps as well as local misorientation maps to assess meso-scale deformation and stored work in the form of misorientation, respectively, are shown in Fig. 1b. Local misorientation information is represented by measures such as grain orientation spread (GOS) and kernel average misorientation (KAM). GOS is defined as the average deviation between the orientation of each point in the grain and the grain average and KAM is the average misorientation between a point and its nearest neighbors [54].

It is seen from the GOS maps that the in-grain misorientation and consequently the stored work increases with strain. Also, within each sample, some grains are seen to have a higher spread in orientation than others, indicating that the deformation is not uniform throughout the samples. Further characterization of the deformed material using spherical nanoindentation focused on the as-received sample and the samples deformed to 3 and 20% strain. The other sample (10% strain) will be the focus of future work and is included here only to illustrate the evolution in the microstructure.

Nanoindentation tests were performed on these (0, 3 and 20% strained) samples using an MTS nanoindenter XP[®] equipped with the CSM attachment. A 100 μm radius, spherical indenter tip was used and indents were made on the sample surfaces in a rectangular grid. Due to the use of a relatively large indenter tip, only one indent (two in rare cases) could be accommodated within each grain. The size of the final residual impression was approximately 7 μm in diameter (estimated from EBSD images) and the spacing between the indents was kept to 70 μm . After the indentation tests were performed, the regions of interest were imaged again in the SEM, to confirm the location of each indent and determine the local crystal orientation at the indentation site. Any indent whose residual impression intersected a grain boundary was excluded from the analysis. This is reasonable because the analysis uses information about the elastic–plastic transition, which occurs at an indentation depth of approximately 15–20 nm and the

residual impressions observed in this study were after indentations to a depth of 400 nm. Thus, we assume that if the residual indent did not intersect a grain boundary, at the elastic–plastic transition, the indentation zone was within a single grain. Over 200 indentations in the undeformed samples and about 120 indentations in each deformed sample (3 and 20% strain) were performed. Of these, approximately 160 indents in the undeformed sample and about 80 indents in each of the two deformed samples were used for analysis. The rest were excluded from the analysis mostly because of their proximity to visible grain boundaries on the sample surface. Specifically, an indent equal to or less than 4 μm from the grain boundary was assumed to have an impression that would intersect the boundary.

While using smaller indenter tips would provide better spatial resolution, a major challenge with using small indenter tips is the occurrence of displacement bursts or “pop-ins”. As reported in prior studies, indentation measurements performed on annealed metals using a small indenter tip frequently result in pop-ins [53, 55]. These pop-ins are attributed to difficulty in activating a potential dislocation source in relatively small volumes ($<1 \mu\text{m}$) associated with indentations with small tips. This manifests as a strain burst at increased stresses followed by an unloading to stresses that represent indentation flow stress values in the absence of a pop-in. Accordingly, the pop-ins mask the yield behavior, make the extraction of the indentation yield stress (Y_{ind}) unreliable, and are therefore, undesirable. Using a large indenter, the indentation zone is larger and therefore the probability of establishing the necessary dislocation sources to accommodate the imposed deformation is much higher. Hence the probability of the occurrence of a pop-in is greatly reduced. The effect of indenter-tip-size on the occurrence and size of the pop-in is shown in [45]. To avoid the occurrence of pop-ins in the indentation measurements performed in this work, the 100 μm radius indenter tip was used.

Even with the 100 μm radius indenter tip, pop-ins were occasionally observed. This was particularly the case in the undeformed sample where potential dislocation multiplication sources as well as the density of glissile dislocations present in the microstructure was low by design. To minimize the effect of pop-ins on the indentation measurements, Y_{ind} was extracted by back-extrapolating from the post-elastic segment shown between the two vertical dashed lines in Fig. 2. From the ISS curves, Y_{ind} was determined as the point of intersection between the modulus line (i.e., the elastic portion of the initial loading segment) and the best-fit line for the stress–strain curve within the strain range of 0.01 and 0.018. This protocol was standardized for all the tests reported in this paper. Macroscale deformation increases the dislocation density, and hence, in the deformed samples, pop-ins were almost

Fig. 2 Representative **a** load–displacement and **b** corresponding indentation stress–strain response for two grains in the as received tantalum sample

completely absent. However, to maintain consistency, the same back-extrapolation method was used to extract the indentation yield point in the deformed samples, as well.

Results and Discussion

Representative load displacement curves and corresponding ISS curves for two orientations tested in the undeformed condition are presented in Fig. 2. The inset in Fig. 2a shows the orientations of grains A and B plotted on the standard [001] inverse pole figure (IPF) map. Specifically, the position of grains A and B, on the IPF map, represent the crystal direction of the grain that is parallel to the indentation direction. Note, that since we use an axisymmetric indenter tip, in-plane rotations of the crystal orientation do not alter our indentation response. Consequently, only two, (Φ, φ_2) of the three Bunge-Euler angles used to represent the crystal orientation have an effect on the indentation response. In Fig. 2, grain A is close to the [001] corner of the IPF map, meaning that the [001] crystal direction is parallel to the indentation axis. Grain B is close to the [111] corner of the IPF map and has the [111] crystal direction almost parallel to the indentation axis. The difference in the number of active slip systems expected

between these two high symmetry orientations is close to the maximum possible value for cubic materials and as expected, the two orientations show a large difference in their nanoindentation response, most clearly observed in Fig. 2b. Local crystal anisotropy affects both the elastic and plastic response. Based on the modified theory of Vlassak and Nix [56], the sample indentation modulus for tantalum grains, which has an elastic anisotropy factor of 1.56, has been predicted to be within the range of 139.3–149.5 GPa. It is once again emphasized that although the tantalum sample being studied is polycrystalline, as the indents are much smaller than the grain size ($\sim 7\mu\text{m}$ in diameter), the local hardness values measured here, effectively characterize the response of individual grains. The 160 indentations utilized to characterize the undeformed Ta, reveal that as expected, the measured values of both the indentation elastic modulus (E_{ind}) and Y_{ind} exhibit a strong dependence on the crystal lattice orientation. The values of E_{ind} were found to range from 138 to 148 GPa with the lowest values measured for orientations close to [001] and highest values for orientations close to the [111] crystal directions. These observations validate the predictions of the Vlassak and Nix work.

The Y_{ind} values extracted for the different orientations ranged from 260 to 380 MPa. Once again, the Y_{ind} values were found to be the lowest for orientations close to [001] and highest close to the [111] crystal directions. In a highly simplified case, if one were to assume the stress fields during indentation to be predominantly compressive along the z-direction, then it follows from Taylor analysis that the [001], which has the lowest Taylor factor for compression would be the softest and the [111] crystal direction, which has the highest Taylor factor, would be the hardest. Therefore these trends in these Y_{ind} values are expected and are quantified here for this high purity Ta material. Although a major component of the stresses during spherical nanoindentation is along the z-axis, in reality, the stress state during spherical nanoindentation is tri-axial in nature and heterogeneous. Note, to the best of our knowledge, this is the first time that the plastic anisotropy in annealed tantalum has been quantified; this is merely a first estimate of the expected relationship.

Figure 3a, b give a summary of the quantification of the effect of crystal orientation on E_{ind} and Y_{ind} , respectively, in the form of contour plots. The contour plots were created by interpolating between the measured E_{ind} and Y_{ind} using generalized spherical harmonics (GSH) [20]. The contour plot in Fig. 3b, henceforth referred to as the indentation yield surface, is a critical baseline for comparison against deformed specimens. Using this map, Y_{ind} , for any given orientation in this high-purity tantalum material, can now be estimated. Since the dislocation density in the undeformed Ta is expected to be low and independent of the

Fig. 3 Contour plot generated by interpolating between the **a** E_{eff} and **b** Y_{ind} values extracted for about 160 orientations in the as received condition. The *black circles* represent the positions of the tested orientations on the standard IPF plot

crystal lattice orientation, all the measured variation in Y_{ind} is attributed to the differences in the orientation of the slip systems with respect to the indentation direction. Thus, this indentation yield surface can be utilized to decouple the effects of orientation and local dislocation density on the measured Y_{ind} in the deformed material. Having established a quantitative relationship between local crystal orientation and Y_{ind} , we focus our attention now on the deformed tantalum sample, with the goal of obtaining insights into the hardening rates in the grains of different orientations as a function of applied strain.

As noted in the previous section, the high purity tantalum samples were subjected to high strain-rate deformation. Through these tests, two samples, one deformed to slightly past yield (3% strain) and another subjected to moderate deformation (20% strain) were obtained. Through-thickness EBSD micrographs, for sections parallel to the compression axis, for these two samples, are shown in Fig. 1b. From the IPF map as well as the KAM

maps, it is clear that large in-grain misorientations exist in all of the deformed samples. Furthermore, the spread in orientation within grains increases with increasing deformation. Qualitatively, the heterogeneity in the distribution of highly misoriented grains within the microstructure can be explained by the necessity to accommodate the imposed deformation by individual grains in ways that satisfy governing field equations (i.e., equilibrium equations) while utilizing only the limited number of slip systems that are favorable in each grain.

Spherical indentation tests were performed within the individual grains in the deformed samples and EBSD was used to determine the local crystal orientation at the indentation site, as was done in the undeformed case. In this case, pop-ins were almost absent due to the higher dislocation density as a result of the imposed macroscale deformation. However, the back-extrapolation method was used to extract the Y_{ind} values, to maintain consistency. As a specific example of the effect of macroscale deformation on the indentation stress–strain curves, we show in Fig. 4, the nanoindentation response for four grains: two tested in the as-received condition (same data as Fig. 2: Grains A and B) and two in the 20% deformed sample (Grains A2 and B2). Grains A2 and B2, tested in the deformed condition are of almost the same orientation as grain A and B, respectively, which were tested in the undeformed condition. As seen in

Fig. 4 **a** The load–displacement data, and **b** corresponding indentation stress–strain curves, for two pairs of almost identically oriented grains, showing the effect of deformation on the ISS response

the data, the increment in the Y_{ind} due to the imposed deformation is significant and can be as high as a 60% increase in yield depending on the location of the indent.

For each of the grain orientations tested in the deformed condition, the Y_{ind} prior to deformation is taken from the yield surface in Fig. 3(b) for that orientation. A reliable estimate of the increment in the indentation yield strength (ΔY_{ind}) for any given orientation can then be determined as the difference between the measured indentation yield point in the deformed condition [$Y_{ind}(g, d)$] and the estimated indentation yield point in the fully annealed condition $Y_{ind}(g, 0)$ [44, 46]. This is given as:

$$\Delta Y_{ind}(g, d) = Y_{ind}(g, d) - Y_{ind}(g, 0) \quad (1)$$

where d refers to the amount of deformation (3 or 20% strain under dynamic loading conditions, for this study) and g refers to the local crystal lattice orientation at the indentation site.

The simplest relationship that can be established between the increment in the indentation yield point (ΔY_{ind}) and the local dislocation content (ρ) is through the increment in the critical resolved shear strength ($\Delta \tau_{crss}$) of the slip system. In a highly simplified manner, this relationship can be expressed as

$$Y_{ind} = M(\phi, \varphi_2) \tau_{crss}(d) \quad (2)$$

$$\Delta \tau_{crss} = \tau_{crss}(d) - \tau_{crss}(0) \propto \sqrt{\rho} \quad (3)$$

where M is the Taylor factor that depends only on the grain orientation with respect to the indentation direction (in this case, only two of the three Bunge-Euler angles describing local crystal orientation because the indenter is axisymmetric), τ_{crss} is the average critical resolved shear stress in the crystal, $\Delta \tau_{crss}$ is the increment in the local averaged critical resolved shear strength between the annealed and deformed conditions. The Taylor factor M is defined as:

$$M = \frac{\sum \Delta \gamma}{\Delta \varepsilon} = \frac{\sigma_y}{\tau_{rss}} \quad (4)$$

where $\sum \Delta \gamma$ is the sum of the slip shears on all the slip systems, $\Delta \varepsilon$ is the imposed macroscopic strain, σ_y is the yield stress and τ_{crss} is the critical resolved shear stress for the activated slip system [57]. A high value for the Taylor factor (for a given mode of deformation) implies a high degree of slip activity is required to accommodate the imposed macroscale plastic deformation. Grains with a high Taylor factor are generally referred to as hard grains and those with a lower Taylor factor are referred to as soft grains. It follows that the grains with relatively higher Taylor factors are more difficult to deform and require higher local stresses to produce the required slip to accommodate deformation as compared to the softer grains.

The relative hardening rates for the differently oriented grains within the deformed sample can be compared by expressing the increase in the slip resistance as a percentage change in the critical resolved shear stress as compared to the undeformed condition.

Combining Eqs. (1) and (2), the percentage increase in the critical resolved shear stress is given as:

$$\% \Delta \tau_{crss} = \frac{\Delta \tau_{crss}}{\tau_{crss}(g, 0)} \times 100 = \frac{\Delta Y_{ind}}{Y_{ind}(g, 0)} \times 100 \quad (5)$$

Since the change in the average critical resolved shear stress in the crystal can be related to the local dislocation density (Eq. 2), the $\% \Delta Y_{ind}$ provides an indirect measure or a trend in the local dislocation content as a function of grain orientation and dynamically induced macroscopic strain.

Using the $Y_{ind}(g, d)$ measured on the deformed samples and $Y_{ind}(g, 0)$ predicted using the indentation yield surface (Fig. 3b), the $\% \Delta Y_{ind}$ (or $\% \Delta \tau_{crss}$) was computed for each indent performed on the deformed sample. It is seen that the computed $\% \Delta \tau_{crss}$ is highly non-uniform across all the tests and varies significantly depending on the grain orientation for both the 3 and 20% deformed samples. The variation of the increment in the local critical resolved shear strength as a function of crystal orientation for the 3% deformed sample is plotted in Fig. 5a. It is seen from this figure that the highest values for $\% \Delta \tau_{crss}$ occur for orientations close to the [001] orientation and the lowest values occur close to the [111] orientations.

In the present study, the highest values for $\% \Delta \tau_{crss}$ are seen for orientations close to the [100], which is the softest orientation and the lowest values are seen close to the [111] orientation, which is a hardest orientation. The trends observed are opposite to those seen for grain interiors in the case of aluminum [44, 46]. Instead, for this study, the observed trends were similar to those seen close to grain boundaries in aluminum. In the case of the tantalum studied here, the average grain diameter is $\sim 40 \mu\text{m}$. This is close in value to the hardened boundary layer observed in large grained aluminum. If the thickness of the region affected by neighboring grains is similar for both aluminum and tantalum, it would mean the effect of a neighboring grain could impact the behavior of an entire grain in this Ta material, not just a narrow region close to the grain boundary. It appears that the smaller grain size in the tantalum studied here, causes the grain interactions to dominate the local mechanical response within an entire grain. This is unlike the case of coarse-grained aluminum, where the effect of grain interactions was limited to a relatively thin (as compared to the grain size) layer and the grain interiors deformed as per the predictions of the Taylor model.

Fig. 5 Contour plot generated by interpolating between the change in slip resistance values extracted for about 100 orientations in the 3% deformed sample. The *black circles* represent the positions of the tested orientations on the standard IPF plot. **b** Rotated view of **a** and locations for a few selected grains showing the diverse neighborhood and its effect on the change in critical resolved shear stress. The white circles indicate the location of the indents

This conclusion is further supported by the fact that the scatter in the values of $\% \Delta \tau_{crss}$ is much higher for the softer (close to the [100]) grains as compared to harder (close to the [111]) grains. This is shown in (Fig. 5b) the 3-D plot showing orientation effects of crystal orientation on $\% \Delta \tau_{crss}$ (seen in Fig. 5a). IPF maps in the vicinity of a few select indents, where local microstructure around the indents is very different, are also shown. For the hard grains (blue grains) shown in Fig. 5b, the $\% \Delta \tau_{crss}$ is about the same, irrespective of the neighboring grains. Whereas, in the case of the soft grains (red grains), the values of $\% \Delta \tau_{crss}$ differ, significantly, as a function of local neighborhood. The $\% \Delta \tau_{crss}$ is much lower for a soft grain surrounded by other soft grains, whereas it is higher for a soft grain that is surrounded by hard grains. This suggests that the grains with hard orientations, all deform in a similar

manner, irrespective of the neighbors surrounding individual grains. This leads to the lower amount of observed scatter in the $\% \Delta \tau_{crss}$ measurements for grains close to the [111] orientation. In contrast to this, the behavior of the soft grains appears to be related to their neighboring grains. Specifically, soft grains when surrounded by hard grains will harden much more than soft grains surrounded by predominantly soft grains. It is important to keep in mind that only the neighbors in 2-D can be determined through EBSD and the information about the out of plane neighbors is still missing. Therefore, just from the 2-D EBSD images of the microstructure around the indents, it is impossible to verify this conclusion for every case.

Another possible explanation for the observed trends in the $\% \Delta \tau_{crss}$ might be related to the uniformity of stress accommodation within the polycrystalline structure. This suggests that if all locations (therefore all grains) experience the same amount of stress, then the soft grains will start deforming before the hard grains and eventually accommodate higher amounts of local strain leading to the higher $\% \Delta \tau_{crss}$. The observed pattern in the variation in the scatter in the measured $\% \Delta \tau_{crss}$ as a function of grain orientation discussed in the previous paragraph cannot be explained by this argument alone. Also, yield drops during quasi-static testing of tantalum, attributed to texture and microstructure effects were observed by [5] and it was concluded through simulation [58, 59] that polycrystalline microstructures deform under the criteria of uniform work (rather than under uniform strain or under uniform stress). This further supports conclusions of this work that grain interactions dominate local behavior.

The 20% deformed sample displays a similar trend as the 3% deformed sample and data of that sample are given in Fig. 6. As expected, while the trend is similar, the magnitude of the increase in the critical resolved shear stress for the 20% deformed sample is much higher than that for the 3% deformed sample. This is due to the higher amount of macroscopic deformation resulting in an overall increase in the dislocation density and therefore a corresponding increase in the slip resistance. This is also indicated by the much higher grain orientation spread seen in the 20% deformed sample as compared to the 3% deformed, shown in Fig. 1b. However, in spite of this, the macroscopic stress–strain curve, obtained from the split-Hopkinson pressure bar test, Fig. 1b, shows limited work hardening and indicates that the flow stresses at 3% strain are the same as those at 20% strain. This is in contrast with the increase in the in-grain misorientation as well as Y_{ind} from the 3% deformed sample to the 20% deformed sample. A combination of dislocation structure and change in stress state may explain this observation and therefore, TEM studies are being performed and will be the subject of a follow-up publication

Fig. 6 Contour plot generated by interpolating between the change in slip resistance values extracted for about 100 orientations in the 20% deformed sample. The *black circles* represent the positions of the tested orientations on the standard IPF plot

While only samples strained to 3 and 20% were examined in this work, characterization of samples strained to intermediate levels (5 and 10%) may shed more light on the evolution of slip resistance and its dependence on crystal orientation. Also, in this case, the indentation measurements were performed perpendicular to the macroscopic loading axis for compression in the SHPB. Change in the relative axis for indentation with respect to the loading axis during SHPB will not change the dislocation density at the indentation site. However, this will lead to a change in the dislocation structure encountered during the indentation test. While the effect of dislocation structure on the nanoindentation response is expected to be less significant as compared to the dislocation density at the indentation site, further testing is required to confirm this observation. Finally, the impact of this work is to utilize the quantification of the local properties as a function of grain orientation and macroscopic strain due to dynamic mechanical loading to locations for the onset of shear deformation and void nucleation in Ta.

Concluding Remarks

The systematic quantification of the percentage increase in the slip resistance as a function of deformation during dynamic loading of high purity tantalum is presented. A two-step characterization process combines local, mechanical property information obtained using spherical nanoindentation with corresponding structure information gathered using EBSD at increasing levels of strain. Even in the as-received

condition, the Y_{ind} of tantalum varies by as much as 40% depending on the crystal orientation. The increase in the local slip resistance (due to the dynamic deformation) can then be linked to the change in Y_{ind} . It was seen that for the tantalum studied here, with grain sizes of $\sim 40 \mu\text{m}$, grain interactions dominated the change of local slip resistance and the hard grains, impose additional work on the soft grains, thus causing them to undergo enhanced hardening. Two samples, dynamically deformed to 3 and 20% strain were characterized in this work and the trends observed were similar in both these cases, although the magnitude for the increase in τ_{crss} is higher overall, in the latter case. This work serves to establish an initial, comparative basis for future studies focused on quantifying the strength of and the deterministic linkages between microstructure and damage initiation in dynamically loaded tantalum.

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