INITIAL STAGES OF YIELD IN NANOINDENTATION
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ABSTRACT

We have used the Interfacial Force Microscope to perform nanoindentations on Au single-crystal surfaces. We have observed two distinct regimes of plastic deformation which are distinguished by the magnitude of discontinuities in load relaxation. At lower stresses, relaxation occurs in small deviations from elastic behavior, while at the higher stresses they take the form of large load drops often resulting in complete relaxation of the applied load. These major events create a relatively wide plastic zone that subsequently deepens more rapidly than it widens. We discuss these findings in terms of contrasting models of dislocation processes in the two regimes.

INTRODUCTION

One of the challenges of nanoindentation is developing an understanding^{of} the detailed processes responsible for initial plastic yield. At larger scales of indentation, a number of models exist for plastic flow¹⁻⁵, but as experimental scales shrink, properties change⁶⁻⁹, and these models must be reconsidered. It is clear that obtaining a detailed understanding of the atomic-level processes that contribute to the initial nucleation of dislocations, their motion and multiplication will greatly facilitate the design of tailored materials with specific mechanical properties.

Two well documented observations from nanoindentation experiments^{6,8-12} illustrate the potential of this technique for providing fundamental insight into initial yield process: 1) that single crystals with low defect density can support stresses near the theoretical limit without yielding and

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2) initial plasticity is usually observed as a sudden deviation from elastic behavior. Stresses near the theoretical limit can be supported because nanoindentation is an effective method of probing a volume sufficiently small that defects are unlikely. The sudden deviation from elastic loading, termed the 'yield point', has been attributed to the nucleation, multiplication, motion and pile-up of dislocations⁹⁻¹⁴.

Nanoindentation has also been established as a technique for measuring time-dependent deformation. It has been shown that there is some time-dependence to the phenomenon of the yield point¹⁵, suggesting that creep of the material under the indenter may contribute to the onset of plasticity. Diffusion of material from beneath the indenter has been reported^{16,17}, which also suggests that surface diffusion within the contact area occurs and may contribute to yield. Nanoindentation on a sufficiently small scale may allow quantification of creep during very initial stages, when the first several atomic layers of deformation occurs, which may identify the contribution of creep to later plasticity.

In this paper, we present results of constant displacement-rate nanoindentation experiments, combined with constant repulsive-force imaging of surface morphology, applied to large-terrace regions of the surface of single-crystal Au (111). We demonstrate that initial plastic deformation takes place in two distinct stress regimes. In the lower stress region, "minor" events occur between about two and six GPa and take the form of abrupt changes in slope or discontinuous drops in the load having relative magnitudes of less than ~5%. At higher stresses, "major" plastic events involve discontinuous load relaxation with magnitudes always greater than 20% and often show full applied-load relaxation values of 100%. Minor events result in depressions one to five atomic layers deep, while major events produce depths in excess of 15 - 20 atomic layers with no depths occurring at intermediate values. We discuss these loading cycle discontinuities and the resulting plastic deformation in terms of possible mechanisms involving dislocation nucleation, motion and multiplication and will show that at the nanometer scale, models of yield and plastic zone evolution must be reconsidered.

EXPERIMENTAL PROCEDURES

Measurements were performed using the Interfacial Force Microscope (IFM). This instrument, which has been described in detail elsewhere¹⁸, is distinguished by its use of a novel electrostatically-driven, force-feedback sensor to ensure rigid displacement control during a loading experiment. This characteristic of the sensor will be exploited to quantify individual yield events and will also be used to isolate plasticity due to single events. In addition, the IFM has the capability to perform constant-force imaging, similar to other scanning force techniques. Two instruments were used. One involved an IFM-retrofitted commercial Atomic Force Microscope (Digital Instruments, Nanoscope II), while the second had imaging and loading-cycle software developed specifically for nanoindentation measurements with the IFM.

The IFM force sensors and piezo actuators were calibrated using a standard laboratory electronic balance and a displacement indicator, respectively. Both calibrations are necessary for quantitative measurements. Two types of probes were used in this study. Most were electrochemically etched 100 μm tungsten wires while several were diamond tips shaped to a parabolic end form by a focused ion beam (FIB) technique¹⁹. Tip radii and overall tip shapes were determined for all tips by field-emission Scanning Electron Microscopy. Au samples with (111) orientation were prepared by flame annealing Au wire to give {111} facets^{20,21}. Immediately after cleaning, samples were immersed in a 0.5 mM ethanolic alkanethiol ($\text{CH}_3(\text{CH}_2)_{15}\text{SH}$) solution for 24 hr to develop a self-assembled monolayer (SAM) of hexadecanethiol to passivate the probe-sample interaction²². Without the SAM, a strong adhesive interaction occurs between the Au and W tip and the material is plastic on contact²³. Passivation eliminates this adhesion and allows us to analyze elastic force profiles using Hertzian theory^{5,24}.

The probe was displaced at a constant rate of 5 $\text{\AA}/\text{s}$ and the force necessary to maintain sensor equilibrium monitored. In the elastic regime, forces were compared to the force-displacement relationship predicted by Hertzian mechanics⁵,

$$F = \frac{4}{3} E^* \sqrt{R_{tip}} \delta^{3/2}, \quad (1)$$

where E^* is the composite modulus, R_{tip} is the tip radius and δ is the elastic deformation. Stresses at a particular point of yield were quantified by the mean normal stress,

$$\sigma_y = \frac{F_y}{\pi a_y^2}, \quad (2)$$

where the contact radius at yield (a_y) was estimated from the relation ⁵:

$$a_y = \sqrt{R_{tip} \delta_y}. \quad (3)$$

RESULTS

We present the results of investigations in broad-terrace regions of $\{111\}$ surfaces. Loading cycles exhibited two types of deviations from elastic behavior, which we have characterized in terms of the relative magnitudes of the associated load relaxation. Smaller deviations appeared as load drops of less than 5% or as abrupt changes in slope. There were often a series of these deviations, which will be referred to as minor events. If the loading cycle was continued to higher stresses, a large discontinuity appeared that was easily distinguished from minor events because it was an order of magnitude larger in scale. These major events involved load drops that were always greater than 20% and often reached full applied-load relaxation values of 100%. In the following, we discuss our results for these two loading regions separately.

Minor Events

After initial surface contact, the loading cycles followed the force-deformation relationship predicted by Hertzian mechanics. A deviation from the Hertzian relationship signals the occurrence of plastic flow. We often, but not always, observed slight deviations from Hertzian behavior while the stress was in the range of 2-6 GPa, as illustrated in Fig 1a. In this loading cycle, which was performed with a tip radius of approximately 500 Å, the force rises according to the Hertzian relationship (shown as the solid curve) up to approximately 5 µN, where a slight deviation from elastic behavior was observed. In this case, the deviation appears as a change in slope that persists over ~20 Å of deformation. As deformation proceeds, the force continues to rise according to the Hertzian prediction. Upon retraction, no discontinuities were observed, but some hysteresis developed. Below 10 µN, the two curves appear to be approximately parallel but offset by ~5 Å. Thus, we expect a permanent deformation of ~5 Å.

An independent measure of the resulting deformation was obtained from a line-scan view of the topography after indentation, as is shown in Fig 1b. While this line-scan data is strongly affected by the fact that the topography is viewed by the same tip used to create the indentation (as evidenced by the cusp-like shape in Fig. 1b), the depth is reasonably accurately measured by this technique. It is apparent that the hysteresis seen in the loading cycle is due to the creation of a depression, the depth of which was measured from Fig. 1b to be 5.1 Å. This correlates very well with that expected from the loading-cycle hysteresis and, since the interplanar spacing of {111} planes in the Au lattice is 2.49 Å, two independent measures indicate that the depression is two atomic layers deep. While minor events produced indentations with integral values of the Au interlayer spacing, no indentations were observed with depths exceeding about five atomic layers.

Minor events were not observed in all loading cycles, and their occurrence was found to be dependent upon the tip radius. Experiments in which we observed some type of plastic event were surveyed, and for a given tip size, the fraction of loading cycles exhibiting minor events was determined. The results are shown in Fig. 2 and clearly identify the influence of tip radius. The

fraction of loading cycles in which minor events were observed decreased as the tip radius increased. While the statistical certainty of some of these data is poor, due to the small number of experiments performed with some of the probes, the trend is clear.

Major Events

Regardless of whether minor events occurred, if loading cycles were continued to higher stresses, they *always* exhibited major plastic events. The scale of these events (in terms of the percentage of the load drop) was much greater than in minor events and, in many cases, reached a value of 100%, indicating a full relaxation of the applied load. An example of a loading cycle in which such a major event occurred is shown in Fig. 3a. In this case, the tip radius was over 3000 Å and no minor events were observed. The loading cycle was reversed immediately after the event was detected so that the resulting deformation could be directly quantified.

A line-scan topograph of the deformation due to the loading cycle of Fig 3a is shown in Fig. 3b. This data allows us to evaluate the extent of the plastic zone by comparing the surface after indentation with the undisturbed surface, shown as the dotted line in Fig. 3b. Also, it is apparent that the indentation itself is asymmetric showing “pile-up” only on the right-hand side and “sink in” on the left-hand side. This is due to the three-fold symmetry inherent in indentations on {111} surfaces¹² and is evidence that the line scan was taken approximately perpendicular to one of the major crystallographic axes. The size of the plastic zone and the depth of the indentation are identified on the figure and were roughly 3000 and 65 Å, respectively. Note that the depth of the indentation is greater than the depth of the elastic deformation just prior to the event, which was about 45 Å (see Fig. 3a) indicating that the tip has lost contact with the surface after the event.

Yield after the first major event occurred in a series of smaller events. An example of a loading cycle that was continued well beyond the first major event is shown in Fig 3c. Several minor relaxation events occurred before the major event, and the first major event again resulted in the applied load dropping to zero. This cycle was only reversed once the force reached 35 μN,

which gave a total deformation of approximately 680 Å. Clearly, yield after the first major event occurs in smaller, discrete steps. Deformation is not uniform; it is a combination of elastic and plastic deformation, meaning the lattice behaves elastically until some other critical stress is reached, at which point, another event occurs. This behavior repeats itself, such that the deformation after the first event can be described as a succession of events, which typically give rise to increments of plastic deformation of approximately 10 Å, but which decrease in magnitude as deformation proceeds.

The line-scan topography of the permanent deformation resulting from this indentation cycle is shown in Fig 3d. As with indentations to smaller depths, the shape is not symmetric and, again, the pile-up is greater on the right side. The measured depth of the indentation (about 550 Å) correlates well with that expected from the width of the loop in the loading cycle and the general topographic shape is very similar to that resulting from the first major event. Specifically, because the horizontal axes Fig. 3b and 3d are identical but the vertical axes differ by a factor of seven, comparison of these two demonstrate that the relative shape of indentations changes during the course of an indentation.

Indentation Morphology

The topographs of Fig.'s 3b and 3d indicate that it is possible to characterize the evolution of plasticity during an indentation. We performed a survey of the morphology of indentations formed by various stages of yield. Results were obtained from three different stages of yield: after minor events, after the first major event and after some amount of deformation beyond the first major event. Morphology was parameterized using an average plastic zone radius (R_{pl}) and the depth of the permanent indentation (δ_{pl}). The plastic zone radius was taken as half the diameter of the surface length perturbed by the indentation, meaning that it included both the pile-up and sink-in (see Fig. 3b and 3d for examples). The depth of the indentation was measured at the deepest point. As a means of comparison, the widths of the indentations were compared to the

elastic contact radius immediately prior to the first deviation from Hertzian (a_y), which was estimated using Eq. 3.

Indentations due to minor events had very large aspect ratios (i.e., R_{pl}/δ_{pl} was large). For the indentation in Fig. 1b, the depth was 5 Å and the radius was 350 Å, giving an aspect ratio of 70. In this case, no pile-up was observed, so the width of the depression was taken as the plastic zone size.

Indentations created by plasticity at the first or subsequent major events had smaller aspect ratios. The topography of these indentations is summarized in Fig. 4a. Experiments in which loading was reversed after the first major event are identified by circles and those in which plasticity was continued, by diamonds. As indentations become deeper, the aspect ratio decreases. In experiments reversed immediately after the first major event, we found that the R_{pl} was 10 to 25 times δ_{pl} , with shallower indentations having larger aspect ratios. For indentations in which deformation continued, the ratio of R_{pl} to δ_{pl} decreased, such that when plastic deformation was over 250 Å deep, R_{pl} leveled off at approximately 7-8 δ_{pl} . This trend continued for even deeper indentations, and, as an example, R_{pl}/δ_{pl} was approximately 3.5 for the experiment shown in Fig 3c-d (which resulted in a 550 Å deep indentation).

For comparison, we have plotted the continuum mechanics prediction of a_y/δ_{pl} as a function of δ_{pl} shown as the lower dotted line in Fig 4a; it and the data follow the same functional form. We have multiplied this function by 3, shown by the solid line, which gives a very good fit to the data. This demonstrates that the width of the pile-up, when normalized by the elastic contact radius, was relatively invariant with δ_{pl} ; throughout the initial stages of yield R_{pl} was $\approx 3 a_y$.

This gives considerable insight into the evolution of the plastic zone. Immediately after the first major yield event, with $\delta_{pl} \approx 40 - 50$ Å, R_{pl} was $\approx 3 a_y$. This means that plasticity at the first

major event occurred over a lateral extent three times the size of the contact radius just prior to the event. This distance is much larger than expected²⁵. As deformation continues, indentations get deeper, but they widen only slightly, with the result that aspect ratios decrease. An example of this is illustrated in the comparison of topographs in Fig. 3b and 3d. The plastic zone due to first major event (Fig 3b) is approximately 1800 Å. After the deformation of Fig. 3d, which has a deformation 8 - 9 times deeper (Fig. 3d), R_{pi} is only about ~20% larger.

DISCUSSION

We have observed two distinct types of inelastic behavior that results in permanent deformation on two distinct scales. At the smaller scale, depressions of one to five atomic layers were observed, while at the larger scale, indentations greater than about 15 to 20 atomic layers deep were observed. As mentioned earlier, we observed no indentations of intermediate depth. These two distinct scales suggests that either two different processes give rise to the two different scales of deformation or that one process is initiated to give several Å's of deformation, but encounters some barrier that must be overcome for further plasticity. We discuss the likelihood of whether dislocation nucleation or diffusion is responsible for minor events, and then discuss the characteristics of plasticity at major events.

Processes occurring at minor events

Atomic simulations suggest that dislocations are responsible for initial yield events similar to those we identify as minor events. Embedded Atom Method (EAM) simulations of a rigid, noninteracting spherical tip indenting the Au (111) surface show that initial yield occurs when partial dislocation loops are nucleated beneath the indenter on multiple {111} slip planes²⁶. These dislocation involve slip planes which intersect the surface near the contact periphery and slope toward the axis of symmetry. They rapidly and unstably grow to form loops in which two partial

dislocations are separated by a stacking fault. The loops expand until their effective radius is on the order of the contact radius, at which point they intersect below the tip and become locked. The production and motion of these loops result in an indentation that is one atomic layer deep and the dislocation locking acts as a barrier to further glide. The probe tip used in the calculations was about an order of magnitude smaller than that used in the present experiments, but these simulations suggest that initial plastic deformation occurs through dislocation processes that result in a barrier to further plasticity.

If minor events are created by the nucleation of dislocations, the strong tip-radius dependence in their rate of observation may be due to decreasing sensitivity as tip radii increase. A clue may come from the EAM calculations where individual plastic events seen in the model loading curves involve load discontinuities that fit our definition of major events with relative load drops of 10-50%. However, larger tips imply higher loading to produce the same stress levels beneath the tip. This means that individual dislocation-loop nucleation produces a smaller percentage drop in the load and makes event identification more difficult. In addition, the larger volume of material under stress makes it possible to nucleate a broader spectrum of loop sizes. The overall result is to wash out the plastic-deformation threshold and make it look more continuous in the experimental results.

On the other hand, another reason why we see a strong tip-radius dependence may be that these events involve diffusion. Each load drop or change in slope may correspond to the diffusion of Au atoms from beneath the indenter at the Au-indenter interface. If we take the view that for an event to be detected, a monolayer must diffuse from beneath the indenter over the entire contact area, then we expect that given the same time, temperature, stress, etc., events would be more likely for smaller contact areas (i.e., smaller tips).

To determine whether this is a likely mechanism, we have adapted the calculation of the diffusion-limited displacement rate of a flat punch used by Pharr and Oliver⁸ for our experimental situation. Surface diffusion is relatively rapid for Au at room temperature, with an activation

energy with an upper bound of about 50 kJ/mol ²⁷, giving a surface diffusivity of 10^{-15} cm²/s ²⁷. However, adsorption of the SAM increases the activation energy to about 60 kJ/mol ²⁸, which results in a surface mobility reduction of about 1000 ²⁸. Using $D_s = 1 \times 10^{-22}$ m²/s, $\Omega = 16.9$ Å³, $\sigma = 1$ GPa, and $a = 200$ Å, we find that the indenter creep velocity would be $\approx 7 \times 10^{-5}$ Å/s, meaning that the creep rate should be five orders of magnitude smaller than our displacement rate. From this we conclude that diffusion is not the likely mechanism.

Images of indentations created by minor events showed no pile-up and gave the impression that several atomic layers had been removed. Fig. 5 presents one such image, which resulted from an indentation adjacent to a monatomic surface step. We must emphasize that all other results were obtained on wide surface terraces and this is the sole indentation performed close to a step. Not only is a depression of two atomic layers formed, but the step edge has retracted from the indenter. It is easy to understand how surface diffusion, aided by the proximity of the step edge, can account for deformation of the upper layer. The loading cycle associated with this deformation showed several minor events, which likely correspond to deformation in first the terrace and then the subsequent atomic layers. Note that the shape of the depression does not have any obvious crystallographic features.

Processes occurring at major events

The most remarkable attribute of the major events was that the load often dropped to zero, indicating a complete relaxation of the applied load. When this happened, the indentation depth was greater than the prior elastic depth (see Fig 3), which indicates that the tip loses contact with the surface after the event. These observations were unexpected, but were found to be very repeatable *when the surrounding surfaces was free of steps*. If steps were present, load drops were never 100% and were typically much smaller ^{11,29}. Also, once plasticity was initiated, which results in surface and bulk defects, we did not see such large load drops. This seems to be

a phenomenon related to atomically-smooth surfaces. We have used different probes, samples, sensors and instruments and have repeatedly observed this phenomenon. We did not always see 100% load drops, though, and the magnitude of the drop correlated with the stress at which the event occurred. When it occurred at lower stresses (~ 6.5 GPa), the magnitude of the load drop was lower than when it occurred at higher stresses (~ 8 GPa).

These events begin with high stress concentrations and conclude with strain energy being transformed to new surface area and plastic deformation, so the parallels to fracture are obvious. In fracture, a common occurrence is unstable fracture, which occurs when the rate of change (with crack length) of the strain energy release rate is greater than the rate of change of the fracture resistance³⁰. The result is dynamic fracture. In constant displacement loading, unstable fracture results in a crack accelerating from its initial equilibrium length, but then overshooting the next equilibrium length³⁰. The implication for nanoindentation is that overshooting of the equilibrium indentation depth may occur when dynamic effects are considered.

There is always a question as to whether the instrument somehow contributes to the large levels of relaxation. In load-controlled instruments, it is possible that the sensor aids the relaxation by continuing to apply the threshold-load value during the relaxation process. This results in the load discontinuity appearing as a horizontal line in the load vs. displacement curve—a so-called “pop in” event¹¹. However, in our displacement-controlled loading and IFM sensor, the force generated by the sensor is totally in response to the “back force” resulting from the sample’s elastic deformation. There is no force due to the sensor deformation and no energy is stored in the sensor as a result of generating the force. There is energy stored in the probe, but it is doubtful that it is sufficient to cause these events. The sensor does, of course, have a transient response to the sudden removal of an applied force, which causes it to momentarily move toward the sample before correcting itself and returning to its quiescent position. We have monitored the motion of the sensor during one of the major events and it was observed that the probe moved from its equilibrium position only a few Å’s and for a period of approximately 2 ms. The resulting transient is indistinguishable from that obtained in a non-contact test where the load is suddenly

applied and removed electronically. Initial yield events have been reported to occur over periods on the order of $10\ \mu\text{s}$ ³¹, so it is not unrealistic to assume that the material response is much faster than the response of the sensor. Thus, while the sensor may contribute marginally, it seems clear that the sensor is not responsible for large levels of relaxation corresponding to our major events.

One characteristic of these 100% load-drop events is that very large stresses exist prior to yield and large levels of energy are stored in the lattice. Several models exist of the dislocation processes occurring during yield point events^{10,11,13} and a common feature is the creation of a multiplication source which subsequently produces large numbers of dislocations. Depending on the size of the multiplication source, it may operate at stresses much lower than the GPa stresses supported by the lattice³². Thus, once created, it is likely to be highly overdriven, and deformation cannot be accurately described using static equilibrium conditions.

It is also believed that such sources cease to operate when dislocations meet obstacles or the edge of the indenter stress field and form dislocation pile-ups, which in turn give rise to back stresses^{10,11,32}. If dislocations in some way could be removed such that no back stress develops, these sources would continue to operate until the applied stress was very low. The free surface may, in fact, provide the necessary sink for dislocations to allow the complete relaxation of the applied load.

Thus, our view of yield processes begins with dislocation loops being nucleated at minor events, gliding inward and downward (as shown in the EAM calculations²⁶), and locking beneath the indenter. This is in part driven by repulsive force the indenter-sample interface exerts on dislocations³³ and the small volume within which they glide. This immobilization will allow the stress to further increase without plasticity, and will store more elastic energy in the lattice. At some point, however, the dislocation mechanism changes, perhaps with the nucleation of dislocations along the $\{111\}$ slip planes parallel to the surface (the resolved shear stresses along these planes are somewhat smaller than along sloping planes but are still large). Once dislocations begin to glide radially outward, they will be encouraged to cross slip and glide toward the surface

because of the attractive image force³⁴. The free surface 'anneals' dislocations out of the sample, thus reducing their effect in creating a back stress. Effectively, this process appears as an internal friction, and if the rate of energy loss due to this friction is relatively low, the process could continue until the surrounding lattice overshoots its quiescent position, much as in dynamic fracture. The large plastic zone observed after the major events indicates the importance of the surface and generally supports the notion that dislocations flow radially outward from beneath the contact area and cross slip to the surface because of its attraction. Major plastic events that occur subsequent to the first proceed at lower stress levels and must involve the motion and further multiplication of dislocations that were established by the first major event.

The remaining loading cycle also gives information about nature of the yield process. Immediately after the first yield point, small amounts of plastic deformation were often observed, but there was virtually always considerable elastic deformation. The existence of elastically behaving regions followed by yield points suggests that, as with the first major event, the density of mobile dislocations rapidly increases from zero. That is, after some plasticity, dislocations have either become locked or have escaped via the surface. This, coupled with the observation of long-range plasticity at the first yield point, illustrates the importance of the surface in annealing dislocations and in controlling processes during plasticity.

In addition providing insight into mechanisms, measurements of indentation topography indicate that plasticity models fail to capture the evolution of the plastic zone at this scale. Initial plasticity has historically been believed to be limited to a region close to the indenter, with the first stage being the development of a plastic zone beneath the indenter on the axis of symmetry at a depth of approximately $0.5a_y$ ^{1,5,24,35}. It has also been believed to develop in a spherically symmetric manner, and has been often described using an expanding radial cavity model⁴. We have shown that deformation at the first major event results in a broad, shallow indentation, with plastic zones that are ~20 times the indentation depth. This suggests that surface deformation is a substantial contribution to initial plasticity mechanisms. This has been verified in indentations

performed near steps in which the stress at the first major event was reduced ²⁹ and the size of the load drop ²⁹ or the displacement excursion ¹¹ reduced. After this initial plasticity, deformation is still not spherically symmetric. Comparison of Fig. 3b and 3d show that further plasticity acts primarily to deepen the indentation. The size of the plastic zone for the 550 Å deep indentation was only 20% larger than that of one 65 Å deep.

SUMMARY AND CONCLUSIONS

We have performed nanoindentations on broad terraces of a Au (111) surface and have identified two distinct stages of plasticity. The first minor events occur at mean-stress levels between two and six GPa and results in deformations that are one to five atomic layers deep. The second involved major relaxation events and occur at mean stress of about 7 GPa resulting in deformations in excess of 15-20 layers. No intermediate-depth indentations were found. Minor events are not always observed and their occurrence was found to correlate with the radius of the tip used to probe the surface, with the rate of observation increasing for the smaller radii. We found that major events were accompanied by plastic deformation that occurred over an area with a radius three times that of the elastic contact radius just prior to the event. In addition, it was often found that a major plastic event resulted in a complete relaxation of the applied load and an indentation that was deeper than prior elastic deformation, i.e., the tip actually loses contact with the surface after the event. Plastic flow subsequent to the first major relaxation occurred in individual events but with smaller load drops. These events increased the indentation depth but had little effect on the extent of the plastic zone.

We conclude from these results that the two regions of plastic relaxation result from two distinctly different processes. In the first region, deformation likely occurs through the nucleation, glide and locking of several dislocations, resulting in deformation several atomic layers deep. At higher stress levels, dislocation multiplication occurs and produces mobile dislocations. These dislocations are encouraged to cross slip, glide, and terminate at the surface by the surface image

force, and when they do, they reduce the effect of entanglement and create a relatively large plastic zone. This allows the elastically stored energy to dissipate dynamically in a single relaxation event. Past the first major event, smaller events indicate a lesser level of threshold stress to mobilize or further multiply existing dislocations.

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FIGURE CAPTIONS

Figure 1. Characteristics of minor events. (a) Nanoindentation loading cycle showing a minor event when the applied force reached $\sim 6 \mu\text{N}$. The solid line represents the Hertzian fit to the elastic regime. The entire cycle has approximately $5 \text{ \AA}$ of hysteresis. (b) Topograph of the permanent deformation resulting from the loading cycle in (a). The vertical distance between the markers is $5.1 \text{ \AA}$.

Figure 2. Fraction of loading cycles in which minor events were observed as a function of tip radius. When tip radii were several hundred $\text{\AA}$, nearly all loading cycles showed minor events while fewer did as tip radii were increased.

Figure 3. Characteristics of the major events and subsequent yield. (a) Nanoindentation loading cycle that was reversed immediately after an event was detected. No minor events were observed and the load drop was 100%. (b) Topograph of deformation resulting from loading cycle in (a). The depth of the indentation is larger than the elastic deformation in (a), indicating that the tip loses contact with the surface after the event, and the width of the plastic zone is roughly $3000 \text{ \AA}$. (c) Loading cycle in which deformation was continued beyond the first major event. Plastic deformation continued in events, but the magnitude of the load drops were smaller and continue to diminish as deformation progresses. (d) Topograph of deformation resulting from loading cycle in (c). Compared to (b), the depth is 8 – 9 times larger, but the width of the plastic zone is only 20% larger.

Figure 4. Measurements of indentation morphology, showing that the size of the plastic zone (R_{pl}) is many times the depth of the permanent indentation (δ_{pl}), and the ratio decreases as the indentation deepens. The broken line represents the Hertzian prediction for the elastic contact radius at the yield point (a_y) normalized by δ_{pl} and the solid line is $3 a_y/\delta_{pl}$. The good agreement

between $3 a_y/\delta_{pl}$ and our measurements of R_{pl}/δ_{pl} indicate that $R_{pl} \approx 3a_y$ throughout the initial stages of plasticity.

Figure 5. Constant repulsive-force image of deformation resulting from a minor plastic event with the tip positioned over a step edge. The dark area represents a $\sim 5 \text{ \AA}$ depression and the edge of the step that was in contact with the indenter has retracted. Note the absence of observable pile-up.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5