Incipient plasticity during nanoindentation at elevated temperatures

Alan C. Lund
Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

Andrea M. Hodge
Materials Science and Technology Division, Lawrence Livermore National Laboratory, Livermore, California 94550

Christopher A. Schuh[a)]
Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

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The onset of plastic deformation during nanoindentation is studied, focusing upon the effects of temperature variation. Indentations on pure (100)-oriented platinum at 20, 100, and 200 °C reveal that the transition from elastic to plastic deformation occurs at progressively lower stress levels as temperature is increased. Additionally, it is shown that during plastic deformation, higher temperatures promote the discretization of plasticity into sharp bursts of activity. These results are in line with expectations for stress-biased, thermally activated deformation processes such as the nucleation of dislocations or the abrupt release of dislocation entanglements. © *2004 American Institute of Physics*. [DOI: 10.1063/1.1784891]

The advent of instrumented nanoindentation has allowed the study of deformation physics in confined volumes of crystals, where the nucleation and motion of individual dislocations can be resolved. The general observation in these types of experiments is that indentation first proceeds along a load-displacement $(P-h)$ curve that matches expectations of elasticity theory, followed by a short burst of displacement (a "pop-in" event) marking the transition from elastic to plastic deformation.[1–6] For nanoindentation of clean metal surfaces, experiments and supporting computational simulations[7–10] have led to a growing consensus that this initial pop-in is associated with homogeneous dislocation nucleation, while subsequent similar events often involve avalanches of dislocation activity. Given the thermally activated, stress-biased nature of such processes, one would expect significant variations in the measured $P-h$ response with changes in indentation rate or temperature. Where some studies have observed a time or rate dependence of the pop-in phenomenon,[4,11–15] temperature variations should induce even more obvious changes in experimental $P-h$ curves; elevated temperature nanoindentation testing could provide important experimental support for the interpretation of incipient plasticity as a dislocation nucleation event. Although there have been a limited number of studies that considered the effects of elevated temperature during nanoindentation,[11–13,16–18] these investigations did not examine the issue of dislocation nucleation, and where pop-in events have been seen at high temperature, significant oxide layers were known to be present on the specimen surface. It is the purpose of this paper to present a preliminary study on the effect of temperature on the very earliest stages of plasticity, on a clean, oxide-free metal surface.

The experimental material was a (100)-oriented Pt single crystal of 99.999% purity from Goodfellow (Berwin, PA), which was chosen for its lack of a native oxide layer at ambient and higher temperatures. The specimen was polished to rms roughness of <1 nm through a regimen of mechanical polishing followed by electropolishing in an aqueous solution of HCl and NaCl. Instrumented nanoindentation experiments were performed using a Triboindenter (from Hysitron, Inc., Minneapolis, MN) equipped with a heating stage significantly modified for the present purposes. This system allowed for conductive heating of the specimen to test temperatures of 100 and 200 °C (in addition to room temperature experiments), while shielding the displacement transducer from the heat source with a cooled copper fixture. The indenter tip was a Berkovich geometry diamond, mounted to a low thermal conductivity shaft. Temperature was monitored and controlled using a J-type thermocouple in direct contact with the Pt specimen, and indentations were selectively placed within 2 mm of the thermocouple probe tip. Prior to indentation experiments, the tip was brought into contact with the specimen surface at a very light load of ~ 2 μN, and the entire system was allowed to thermally equilibrate for more than an hour. For all subsequent indentations performed at the same temperature, the tip was maintained in contact with the specimen surface to promote thermal stability, and moved from one location to the next while maintaining the set-point load of 2 μN. Indentations were performed with a loading rate of 10^3 μN/s to various maximum loads and depths; the results obtained were identical for all of the investigated maximum loads, so for simplicity we discuss only indentations with a maximum load of 500 μN.

Typical load-displacement $(P-h)$ curves obtained at each of the three temperatures investigated are shown in Fig. 1, with each curve displaced along the x axis for clarity. In general we have observed that the total indentation depth is not a strong function of temperature; within ±10 nm, indentations at all three test temperatures reached the same depth. This suggests that the material strength is roughly unchanged over the range of temperatures investigated, which is reason-

[a)]Author to whom correspondence should be addressed; electronic mail: schuh@mit.edu

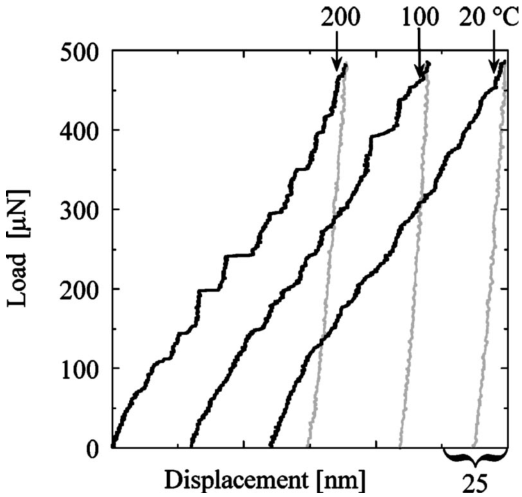

FIG. 1. Typical load-displacement curves measured at 20, 100, and 200 °C, with their origins offset for clarity of presentation. The loading portion of each curve is shown in black, and the unloading portion in grey. As temperature is raised, there is a pronounced increase in the prominence of the horizontal "pop-ins" in the loading part of the experiments.

able given the high melting point of platinum. Additionally, *in situ* imaging of the indentations at temperature (using the contact imaging mode of the Triboindenter apparatus) as well as *ex situ* imaging at room temperature (using an atomic force microscope) revealed indentations of essentially identical size and shape at all three test temperatures. Despite the similar indentation depths of these experiments, two major trends observed throughout the experiments are evident in the curves presented in Fig. 1. First, the shape of the curves at the lowest loads is notably different at the three temperatures investigated, with lower temperatures generally promoting larger "noses." Second, where some small horizontal pop-ins are present at low temperatures, both the number and size of these events noticeably increase with temperature. In the remainder of this letter, we provide more detailed discussions of each of these two effects.

Figure 2 shows magnified views of three typical $P-h$ curves at low loads below 200 μN; here again we see that the initial steep nose of the curve becomes less pronounced

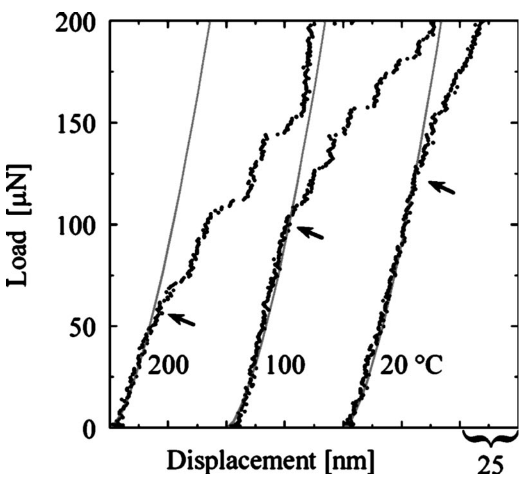

FIG. 2. Initial nanoindentation response of (100) Pt at three different temperatures. In each case, the first portion of the experimental data (black points) can be well described by the Hertzian elastic contact law (grey lines), but the departure from ideal elasticity occurs earlier for specimens at higher temperatures.

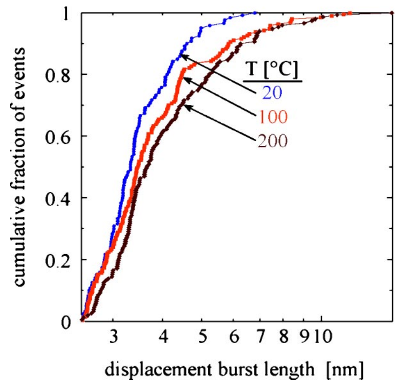

FIG. 3. (Color online). Cumulative fraction of pop-in events of a given size, incorporating data from many indentations under the same conditions; the x axis is logarithmic to better observe trends. The tendency for displacement bursts of every size increases monotonically with temperature.

with temperature. Furthermore, if the indenter is assumed to be blunted into a roughly spherical shape with radius of curvature ~300 nm, we find that all three curves in Fig. 2 can be fitted quite well using the typical Hertzian contact law[19] for a diamond indenter on a platinum substrate. For these calculations we have included the temperature dependence of the elastic modulus,[20] although this has little effect in the narrow range of temperatures studied. The predictions of elastic contact theory are presented in Fig. 2 as solid grey lines, and the agreement with the experimental data is very good at low loads. The departure of the experiment from the elastic theory is usually associated with a small pop-in event, and represents the onset of plastic deformation. This result is generally consistent with prior experiments, but here we also see a significant influence of temperature on incipient plasticity that has not been seen previously. Specifically, we observe that temperature promotes a monotonic decrease in the critical load for plastic deformation. Although the specific load of the elastic–plastic transition varies from one indent to the next even under constant test conditions, the trend with temperature seen in Fig. 2 is quite general for the many hundreds of indentations performed in this work.

Although the trend in Fig. 2 has not been observed before in nanoindentation tests, it supports the notion that the first departure from the elastic $P-h$ curve is associated with homogeneous dislocation nucleation. These nucleation events are stress-biased and thermally activated, and would be expected to occur with a rate that scales as

$$\dot{N} \propto \exp\left(-\frac{\varepsilon - \tau V}{kT}\right). \quad (1)$$

Here ε is the intrinsic energy barrier to dislocation nucleation, τ is the applied shear stress that reduces the nucleation barrier, V is the activation volume, and kT is the thermal energy. According to Eq. (1), dislocations would frequently nucleate at lower stresses when the temperature is raised; the results exemplified in Fig. 2 are therefore in line with expectations. In Ref. 15 we have proposed a general statistical approach for extracting values for the activation volume V in Eq. (1). We now observe that by considering data from multiple temperatures such as we have obtained here, the average defect enthalpy ε could be directly assessed as well.

In Fig. 3 we examine the cumulative distribution of pop-in events of a given size observed at each test temperature, incorporating many dozens of indentations and several hundred individual displacement bursts. For this analysis we include any displacement burst larger than ~2.5 nm, which is judged to be the finest discrete event that can be consistently resolved with the experimental technique used here. The curves in Fig. 3 indicate an unambiguous trend; we find that temperature decidedly promotes the activation of larger pop-in events. Although not evident from Fig. 3, we also see that the average number of displacement bursts increases with temperature (by about a factor of 2 from 20 to 200 °C).

It is interesting to note that the behavior observed here on the nanoscale is rather opposite to classical expectations for plastic flow of crystals; higher temperatures usually promote homogeneous flow through the operation of diffusive mechanisms, while the present data show increasingly serrated flow as the temperature is raised from 20 to 200 °C. This trend is certainly a consequence of the very fine scale of the testing. Because nanoindentation probes the behavior of a small population of dislocations, it is sensitive to individual dislocation interactions and the statistics of thermal activation of these events. Although it is difficult to speculate analytically about the specific dislocation configurations responsible for the serrated flow behavior we observe here, Eq. (1) indicates that dislocation nucleation will increase with temperature. With more dislocations nucleating around the indenter, it seems reasonable that more complex entanglements would be possible at higher temperatures. The release of such entanglements would likely also be promoted by elevated temperatures, and could accommodate larger bursts of strain. These kinds of qualitative arguments may explain the enhanced prominence of large pop-in events we observe as the indentation temperature is increased (Figs. 1 and 3). While this problem seems rather intractable from an analytical point of view, we propose that atomistic simulations performed at multiple temperatures and analyzed in a statistical framework could help to elucidate this complex behavior.

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