Incipient plasticity during nanoindentation at elevated temperatures

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(Received 3 February 2004; accepted 21 June 2004)

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]
able given the high melting point of platinum. Additionally, in situ imaging of the indentations at temperature 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 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 for a diamond indenter on a platinum substrate. For these calculations we have included the temperature dependence of the elastic modulus, 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. 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

$$N \propto \exp \left( - \frac{\varepsilon - \sigma V}{kT} \right).$$

(1)

Here $\varepsilon$ is the intrinsic energy barrier to dislocation nucleation, $\sigma$ 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 $\varepsilon$ 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 \( \sim 2.5 \) 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.

Parts of this work were performed under the auspices of the U.S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48. This work was partially supported by the Defense University Research Initiative on Nanotechnology (DURINT), which is funded at MIT by the Office of Naval Research, Grant No. N00014-01-1-0808. Collaborative support of Hysitron, Inc., is also gratefully acknowledged.