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Incipient Plasticity during Nanoindentation at Elevated Temperatures

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Abstract

The onset of plastic deformation during nanoindentation is studied, focusing upon the effects of temperature variation. By performing indentations on pure (100)-oriented platinum at room temperature, 100 and 200° C, we demonstrate that higher temperatures promote the discretization of plasticity into sharp bursts of activity. Additionally, the transition from elastic to plastic deformation occurs at progressively lower stress levels as temperature is increased. 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.

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 homogenous 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 [4,11-14], 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 [11-13,15-17], these investigations did not examine the issue of dislocation nucleation, and where pop-in events have been explicitly studied, significant oxide layers were known to be present. Accordingly, to date there has been no study of the effect of temperature on the very earliest stages of plasticity during which dislocations are first nucleated beneath the indenter; it is the purpose of this letter to present the first such study 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 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, which is reasonable given the high melting point of platinum. However, 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 significantly different at the three temperatures investigated, with lower temperatures 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 [18] for a diamond indenter on a platinum substrate. For these calculations we have included the temperature dependence of the elastic modulus [19], 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.

In Fig. 3 we examine the distribution of pop-in events observed at each test temperature. For this analysis we take a simple approach of defining pop-ins of at least a critical size as ‘large’, with all other pop-ins designated as ‘small’. The fraction of the indentation depth that results from large pop-ins is presented as a function of temperature in Fig. 3. Regardless of the arbitrary choice as to what constitutes a large pop-in, we find that temperature decidedly promotes the activation of these discrete events. This is the same trend that is observed qualitatively in Fig. 1, but we now suggest that the prominence of pop-in events at 200° C is approximately twice that at ambient temperature.

All of the data presented above are consistent with the notion that discrete plasticity during nanoindentation corresponds to the operation of stress-assisted, thermally-activated dislocation creation. For the first departure from the elastic P-h curve (Fig. 2), the pop-in events are likely homogeneous dislocation nucleation events, which would occur with a rate that scales as:

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

with ε the intrinsic energy barrier to dislocation nucleation, τ the applied shear stress that reduces the nucleation barrier, V the activation volume, and kT the thermal energy. According to Eq. (1), dislocations would frequently nucleate at lower stresses when the temperature is raised, as we observe in Fig. 2. Furthermore, for a given set of indentation conditions, more nucleation events would occur on average at higher temperatures. Apart from the first dislocation nucleation event, it is difficult to speculate analytically about the dislocation configurations responsible for the observed pop-in behavior. However, it seems reasonable that with more dislocations nucleating around the indenter, 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 (Fig. 1). 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 (i.e., averaged over many indentations) could help to elucidate this complex behavior.

Finally, we note that the behavior observed here on the nanoscale is rather opposite to classical expectations for plastic flow of crystals, in which higher temperatures usually promote homogeneous flow through the operation of diffusive mechanisms. In the present case the temperatures are simply too low for diffusive mass transport to accommodate the applied strain, but we expect that at sufficiently high temperatures the transition to homogeneous diffusive flow will still be observed in nanoindentation experiments.

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References:

1 S. Suresh, T. G. Nieh, and B. W. Choi, Scripta Materialia **41,** 951-957 (1999).

2 D. F. Bahr, D. E. Kramer, and W. W. Gerberich, Acta Materialia **46,** 3605-3617 (1998).

3 A. Gouldstone, H.-J. Koh, K.-Y. Zeng, A. E. Giannakopoulos, and S. Suresh, Acta Materialia **48,** 2277-2295 (2000).

4 Y. L. Chiu and A. H. W. Ngan, Acta Materialia **50,** 1599-1611 (2002).

5 S. G. Corcoran, R. J. Colton, E. T. Lilleodden, and W. W. Gerberich, Physical Review **B55,** R16057-R16060 (1997).

[6] W. W. Gerberich, J. C. Nelson, E. T. Lilleodden, P. Anderson, and J. T. Wyrobek, Acta Materialia **44,** 3585-3598 (1996).

[7] J. Li, K. J. Van-Vliet, T. Zhu, S. Yip, and S. Suresh, Nature **418,** 307-310 (2002).

[8] C. L. Kelchner, S. J. Plimpton, and J. C. Hamilton, Physical Review B **58,** 11085 (1998).

[9] J. Knap and M. Ortiz, Physical Review Letters **90,** 226102 (2003).

[10] E. T. Lilleodden, J. A. Zimmerman, S. M. Foiles, and W. D. Nix, Journal of the Mechanics and Physics of Solids **51,** 901-920 (2003).

[11] D. F. Bahr, D. E. Wilson, and D. A. Crowson, Journal of Materials Research **14,** 2269-2275 (1999).

[12] S. A. Syed-Asif and J. B. Pethica, Philosophical Magazine **A76,** 1105-1118 (1997).

[13] D. E. Kramer, K. B. Yoder, and W. W. Gerberich, Philosophical Magazine **A81,** 2033-2058 (2001).

[14] W. Wang, C. B. Jiang, and K. Lu, Acta Materialia **51,** 6169-6180 (2003).

[15] J. Xia, C. X. Li, and H. Dong, Materials Science and Engineering **A354,** 112-120 (2003).

[16] B. D. Beake and J. F. Smith, Philosophical Magazine **A82,** 2179-2186 (2002).

[17] S. A. S. Asif and J. B. Pethica, Journal of Adhesion **67,** 153-167 (1998).

[18] K. L. Johnson, *Contact Mechanics* (Cambridge University Press, Cambridge, UK, 1985).

[19] R. Farraro and R. B. McLellan, Metallurgical Transactions **8A,** 1563-1565 (1977).

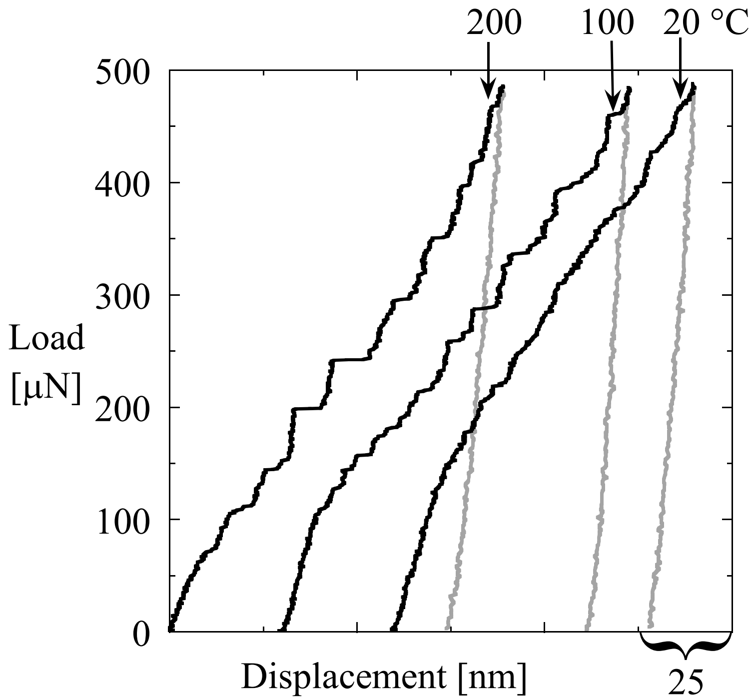

Figure 1: Typical load-displacement curves measured at 20, 100, and 200° C, with their origins offset for clarity of presentation. The loading portion of each curve is shown in black, and the unloading portion in grey. As temperature is raised, there is a pronounced increase in the prominence of the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

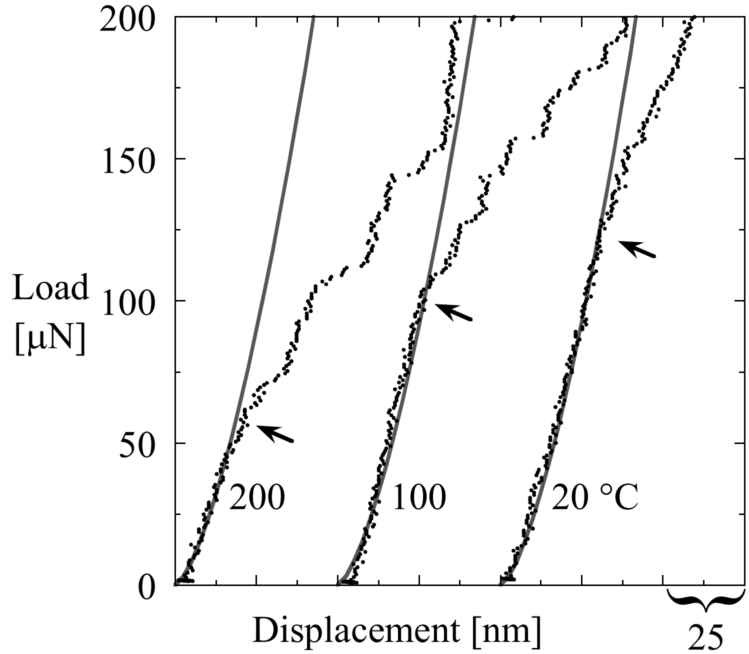

Figure 2: Initial load-displacement response of (100) Pt at three different temperatures. In each case, the first portion of the experimental data (black points) can be well described by the Hertzian elastic contact law (grey lines), but the departure from ideal elasticity occurs earlier for specimens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

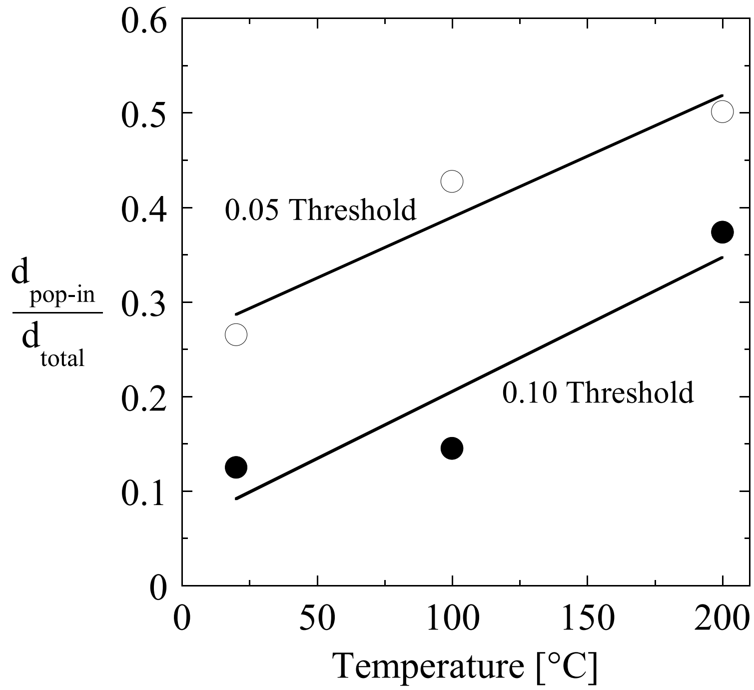

Figure 3: Fraction of the indentation depth due to large pop-in events, defined as those events constituting at least 5% or 10% of the total depth. Regardless of the threshold chosen to define a large pop-in, the tendency for such events increases monotonically with temperature.