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A New View of Incipient Plastic Instability during Nanoindentation *

Jian-Qiao Hu(胡剑桥), Zhan-Li Liu(柳占立)**, Yi-Nan Cui(崔一南),
Feng-Xian Liu(刘凤仙), Zhuo Zhuang(庄茁)**

Applied Mechanics Laboratory, School of Aerospace Engineering, Tsinghua University, Beijing 100084

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Whether the dislocation nucleation or the sudden dislocation multiplication dominates the incipient plastic instability during the nanoindentation of initial defect-free single crystal still remains unclear. In this work, the dislocation mechanism corresponding to the incipient plastic instability is numerically investigated by coupling discrete dislocation dynamics with the finite element method. The coupling model naturally introduces the dislocation nucleation and accurately captures the heterogeneous stress field during nanoindentation. The simulation results show that the first dislocation nucleation induces the initial pop-in event when the indenter is small, while for larger indenters, the incipient plastic instability is ascribed to the cooperation between dislocation nucleation and multiplication. Interestingly, the local dislocation densities for both cases are almost the same when the sudden load drop occurs. Thus it is inferred that the adequate dislocations generated by either nucleation or multiplication, or both, are the requirement for the trigger of incipient plastic instability. A unified dislocation-based mechanism is proposed to interpret the precipitate incipient plastic instability.

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Nanoindentation experiments[1,2] serve as an effective technique to study the incipient plasticity of single crystal at small scales. In a displacement-controlled indentation test, the transition from elasticity to plasticity is manifested as a sudden load drop following a smooth Hertzian contact curve. This instability during nanoindentation is of great importance and always used to predict the theoretical shear stress limits.[3] Either dislocation nucleation or sudden activation of dislocation multiplication has been proposed to account for the instability event. The debate on the physical mechanism of plastic instability is whether the dislocation nucleation happens just at the first pop-in[3,4] or prior to the pop-in event.[5,6] Thus it still remains unclear how the dislocation structures evolve when the first pop-in occurs and whether it is reasonable to use the critical load to describe the dislocation nucleation stress. To clarify these issues thoroughly, the incipient dislocation nucleation and subsequent dislocation multiplication during nanoindentation need to be carefully analyzed.

Numerical simulations[7,8] provide an effective way to study the dislocation microstructures during nanoindentation. Among all kinds of methods, discrete dislocation dynamics (DDD) simulation has played an important role in modeling the dislocation behaviors.[9,10] Tsuru *et al.*[11] suggested that the incipient plastic deformation required much higher shear stress than the theoretical shear strength during nanoindentation by DDD modeling combined with the boundary element analysis. The majority of DDD methods are based on the superposition of infinite domain dislocation solutions. That is, the solution on a finite domain is obtained by applying the image tractions from the infinite domain solutions of all dislocations to the model boundary, and the image field is usually computed by the finite element method (FEM)[12] or the boundary element method.[13] However, in most DDD simulations, it is assumed that the dislocation sources exist before loading and it is not possible to consider the dislocation nucleation which is important for the study of incipient plasticity.

For the DDD model employed here, the same as our previous work,[14,15] dislocation curves are discretized into straight segments. Considering that the dislocation motion is in the over-damped regime, the velocity of each dislocation segment is determined by the total force acting on it divided by the viscous drag coefficient. Then, the dislocation positions are updated for the next time step. At the same time, the topology also updates every time step to deal with dislocation surface annihilation and the short range interactions between perfect non-dissociated dislocations, such as collinear interaction, the formation and destruction of dislocation locks. For the topic of interest in this study, it remains a problem how to introduce the heterogeneous stress field under the indenter into the DDD simulations.

DDD-FEM simulation merges discrete and continuum modelling and is used to bridge the connections between dislocation microstructures and macroscopic mechanical behavior. In the study of nanoindentation, FEM provides the heterogeneous stress field under the indenter and DDD calculates the evolution of dislocations. The coupling of DDD and FEM is based on the superposition principle proposed by Erik *et al.*[16] This model makes it possible to address various boundary value problems which may contain complicated dislocation microstructures. It is worth mentioning that the geometrical boundary change of the computational domain is also taken into consideration in the coupling model. The intensive displacements due to

*Supported by the Key Program of the National Natural Science Foundation of China under Grant Nos 11132006 and 11302115.
**Corresponding author. Email: zhuangz@tsinghua.edu.cn; liuzhanli@tsinghua.edu.cn
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finite deformation will bring a mismatch of the FEM cell and the DDD cell. Thus the displacement calculated in the FEM also returns to DDD simulation for geometry update of dislocations.[17] This synchronous coupling procedure will make it more accurate to calculate the total stress and deal with finite deformation problems at micron and submicron scales.

Through the previous review, it can be seen that the simulation of plastic instability during nanoindentation has many challenges which involve dislocation nucleation, heterogeneity of the stress field, topological algorithm of dislocations and surface morphology, etc. The debate whether the pop-in event is exactly equivalent to the dislocation nucleation still continues. The incipient plastic instability during nanoindentation is investigated by the coupling model. The computational models are set as follows: all specimens are initial defect-free, and have the same dimension $200 \times 200 \times 50\,\text{nm}^3$ as shown in Fig. 1(a). The FEM specimen is meshed into 10880 eight-node solid elements and the region just below the indenter uses a much finer grid to capture a more accurate mechanical response. The spherical indenters with radius R ranging from 20 nm to 100 nm are simulated under a displacement-controlled mode.

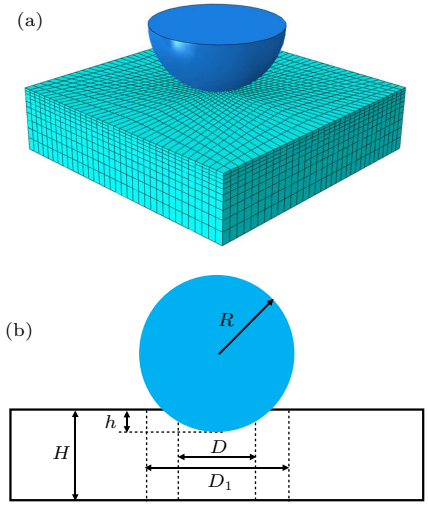

Fig. 1. Finite element model for (a) nanoindentation and (b) schematic diagram.

In the indentation simulation, the topology of dislocation nucleation is necessary since all the specimens are initially defect-free. We use a nucleation criterion of critical resolved shear stress[18] and this nucleation rule is more physical and natural than the global criterion adopted by Fivel *et al.*[19] The local resolved shear stress is calculated at each step and the prismatic dislocation loops will nucleate at the source points where the stress reaches a critical value τ_{nuc}. As suggested by William *et al.*,[20] geometrically necessary dislocations would generate to accommodate the deformation just below the indenter. Thus the nucleated dislocations in the simulation have four activated slip systems involving distinct Burgers vectors $\boldsymbol{b}_1 = \frac{1}{2}[0\bar{1}1]$, $\boldsymbol{b}_2 = \frac{1}{2}[101]$, $\boldsymbol{b}_3 = \frac{1}{2}[\bar{1}01]$ and $\boldsymbol{b}_4 = \frac{1}{2}[011]$ on the planes $\{111\}$. This setup makes the sum of four Burgers vectors in coordination with the deformation in the loading direction. The initial radius of prismatic loops is set to several Burgers vectors. The single-crystal material is taken to be isotropic elastic medium with $G = 27\,\text{GPa}$, $\nu = 0.3$ and $b = 0.256\,\text{nm}$. The critical resolved shear stress for dislocation nucleation is initially set to 2 GPa and will be further discussed.

To quantitatively characterize the plastic instability process, the local dislocation density ρ_{ls} at an effective region just below the indenter is introduced here. For the case of bulk material, this effective region is nearly a hemisphere,[20] but it is considered as a cylinder with diameter D_1 in the case of a thin film as shown in Fig. 1(b). Thus in this study, the local dislocation density ρ_{ls} under the indenter is expressed as

$$\rho_{\text{ls}} = \frac{4L_{\text{d}}}{\pi D_1^2 \cdot H}, \tag{1}$$

where $D_1 = \alpha D$ is the diameter of the effective region with defects, D is the diameter of contact area given by the Hertz contact theory $D = 2\sqrt{Rh}$, h is the penetration depth of the indenter, α is a proportional constant ranging from 1 to 3,[21] and L_{d} and H are the total length of dislocation lines and the thickness of specimen, respectively.

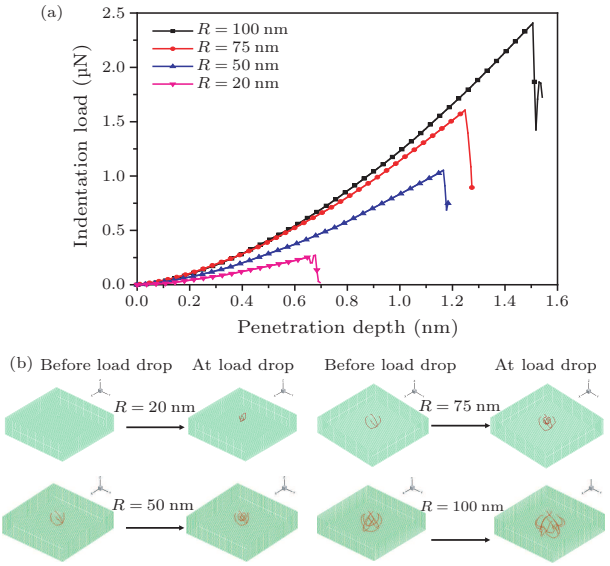

Fig. 2. (a) Computational results of nanoindentation under different indenters. (b) Dislocation structures before and at the point of load drop.

The load-displacement relationship for different indenter radii is shown in Fig. 2(a). The same as the conventional mechanical behavior, the load firstly rises in an elastic way before the macroscopic load drops, which indicates that the incipient plastic instability happens during nanoindentation. This elastic stage has been studied in depth by the Hertz contact theory. In the following we analyze the dislocation structures and explore what kinds of dislocation activities induce the global plastic instability.

The dislocation microstructures just before and at the point of load drop are given, respectively, as shown in Fig. 2(b). It can be seen clearly that for the small indenter with radius 20 nm, the global plastic instability originates from the very initial dislocation nucleation event since no dislocation is detected before load drop, while for larger indenters, the load can continue to increase after the initial nucleation occurs, thus the instability results from the cooperation of dislocation nucleation and multiplication. The results are not in conflict with the previous conclusions achieved by various molecular dynamics (MD) simulations[22,23] in which it is the dislocation nucleation leading to the load drop. Because of the limit by the scale of MD simulation, the first dislocation nucleation event can always make enough contributions for the burst of plastic instability. However, it was also reported that the first dislocation nucleation is not accompanied by the macroscopic load drop in the case of the indenter with radius as large as 70 nm.[24]

Through the above analysis, it can be inferred that the incipient plastic instability is associated with the local dislocation behaviors, accompanying either dislocation nucleation or multiplication, or both. The load drop cannot be simply attributed to dislocation nucleation, which is also consistent with the experimental observation.[6] After the analysis of dislocation microstructures, the critical local dislocation density at the point of load drop is further investigated and proposed to quantitatively characterize the incipient plastic instability.

The local dislocation density under the indenter from Eq. (1) is presented exactly at the time of the first load drop, as given in Table 1. Considering that the dislocation can slip away rapidly under the high indentation pressure, the diameter of effective plastic region is as large as $D_1 = 3D$, which is also supported by careful measurement of the dislocation structures under the in-situ nanoindentation tests.[25] It is interesting to find that the local dislocation densities are generally around $4 \times 10^{15}\,\mathrm{m}^{-2}$ for all the indenter sizes, though the evolution of dislocation structures is not the same at the point of load collapse as shown in Fig. 2(b). This discovery shows to some extent that there may be an intrinsic size independent factor controlling the macroscopic plastic instability. Both dislocation nucleation and multiplication dominant mechanisms may be unified within this intrinsic framework. However, the incipient dislocation nucleation event is obviously related to the choice of nucleation stress, which is artificially determined to be 2 GPa and needs to be further studied to confirm the conclusion.

Table 1. Simulation results under different indenters at the critical load.

Radius of indenter (nm)	20	50	75	100
Local dislocation density (m^{-2})	3.81×10^{15}	4.33×10^{15}	3.71×10^{15}	3.96×10^{15}

Table 2. Values under the 50 nm indenter with different nucleation stresses at the critical load.

Nucleation stress (GPa)	1.5	2.0	2.5	3.0
Local dislocation density (m^{-2})	3.71×10^{15}	4.33×10^{15}	6.02×10^{15}	7.19×10^{15}

The resolved shear stress for dislocation nucleation is artificially defined as 2 GPa since various literature pointed out the stress of dislocation nucleation ranged from $\frac{1}{30}$ to $\frac{1}{8}$ of the shear modulus.[26,27] The influence of dislocation nucleation stress in the simulation is carefully studied in the case of an indenter with radius 50 nm. The simulations are carried out under four typical nucleation stresses ranging from 1.5 GPa to 3 GPa with an increase of 0.5 GPa. The load-displacement relationship is shown in Fig. 3(a). It can be seen that the sudden load drop would occur at different critical displacements due to the change of nucleation stresses. The dislocation microstructures before and at the load drop with nucleation stresses 1.5 GPa and 2.5 GPa are also presented in Fig. 3(b). It can be seen clearly that it is also the cooperation of dislocation nucleation and multiplication inducing the load drop in the case of 2.5 GPa. However, when the nucleation stress value decreases to 1.5 GPa, it is interesting to find that the initial dislocation nucleation can lead to the incipient macroscopic instability.

It seems that there is a phenomenological transition when the nucleation stresses change. However, we still hold the view that the change of nucleation stress values would not convert the basic physical mechanism of global plastic instability. The difference is that a smaller nucleation stress value makes it easier to nucleate dislocations and the current stress level requires less local defects to dissipate. As a result, for the case of the indenter with 50 nm, the initial dislocation nucleation event is enough to release the elastic energy when the nucleation stress is 1.5 GPa. To further investigate the influence of artificially introduced nucleation stress, the local dislocation densities are also calculated at the critical point as listed in Table 2. It can be seen that the local dislocation density is quite related to the nucleation stress value. It is reasonable that in the case of a larger nucleation stress, the load can keep increasing in an elastic way until the dislocation nucleation happens at a higher stress level. This high stress in return requires more local defects to release the elastic energy and to induce the sudden macroscopic load drop.

The simulation is also carried out for the indenter with radius 100 nm and the nucleation stress is set to 1.5 GPa. It is found that the initial dislocation nucleation does not induce the pop-in event, and the absence of load drop may be attributed to the large contact area, which averages out the local traction fluctuations induced by the incipient dislocation

nucleation. However, for a given single crystal material, the nucleation stress is generally a certain value. Thus these phenomenological transitions of dislocation behaviors will not change the basic idea that the macroscopic plastic instability is induced by sufficient contributions of nano-scale instabilities due to dislocations.

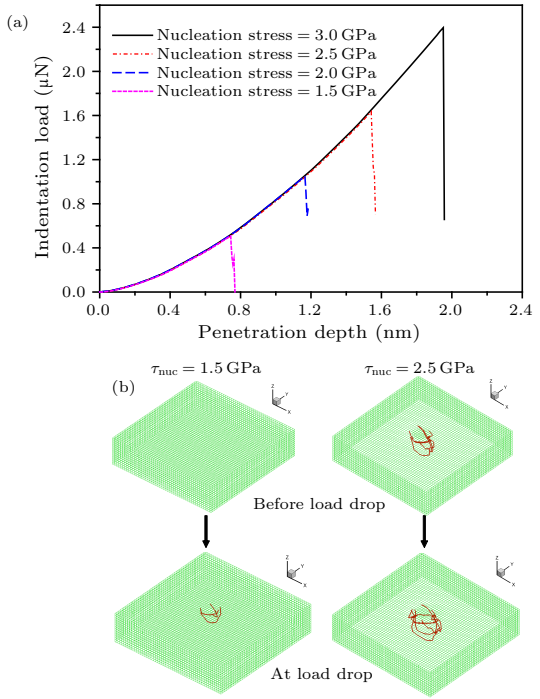

Fig. 3. (a) Computational results of nanoindentation with different nucleation stresses. (b) Dislocation evolutions under two typical nucleation stresses.

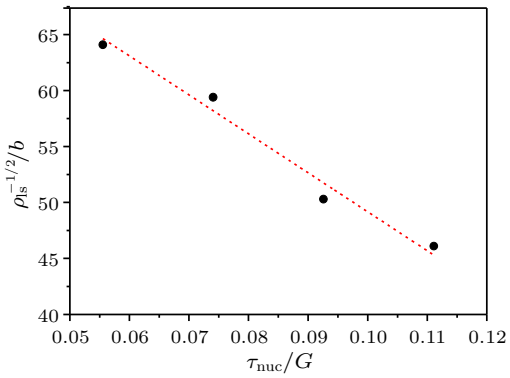

Fig. 4. Average distance between dislocations with different nucleation stresses.

Furthermore, for each nucleation stress, the average distance between dislocations $\rho_{ls}^{-1/2}$ at load drop normalized by the length of Burgers vector b as well as a linear fitting are plotted in Fig. 4. This good linear relationship clearly shows that how many local dislocations are required under different nucleation stresses. It is obvious that a shorter average distance between dislocation defects is obtained with a larger nucleation stress when the incipient plastic instability happens. The results of this analysis extend our understanding of the sudden load drop in nanoindentation. The macroscopic plastic instability cannot be simply interpreted by the incipient dislocation nucleation process or the dislocation multiplication mechanism, while the critical local dislocation density plays a central role. Thus it is proposed that the sufficient local dislocation density is the requirement to trigger the incipient plastic instability.

In summary, how the dislocation evolution influences the incipient plastic instability is investigated by a DDD-FEM model based on the superposition principle. For the case of small indenters, the very first dislocation nucleation event is able to provide enough traction fluctuations to induce the macroscopic load drop, which corresponds to the so-called dislocation nucleation dominant mechanism. In contrast, for larger indenters, the plastic instability is due to the cooperation of dislocation nucleation and multiplication. The results suggest that the experimentally observed first pop-in event during nanoindentation cannot be simply attributed to the dislocation nucleation. By calculating the local dislocation density in an effective plastic region under indenters with different sizes, a unified mechanism is proposed to interpret the pop-ins, that is, the global plastic instability is induced by collective and sufficient contributions of nano-scale instabilities due to dislocations, and it is an intrinsic size-independent factor. The central role of local dislocation density is further confirmed by discussing the effect of nucleation stress.

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