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Quantifying early stage irradiation damage from nanoindentation pop-in tests☆

K. Jin [a], Y. Xia [b], M. Crespillo [b], H. Xue [b], Y. Zhang [a,b], Y.F. Gao [b,a], H. Bei [a,*]

[a] Materials Science & Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA
[b] Department of Materials Science & Engineering, University of Tennessee, Knoxville, TN 37996, USA

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ABSTRACT

Early stage irradiation effects on incipient plasticity are quantitatively investigated in single-crystalline molybdenum using nanoindentation pop-in tests. Defects produced under low-dose ion irradiations, even when they are hardly detected by ion-channeling technique, can significantly reduce the critical stress for the elastic-plastic transition, through acting as heterogeneous dislocation nucleation sources. The density and strength of defects are derived using a unified model convoluting homogeneous and heterogeneous mechanisms. In addition to the increased defect density, defect strength is found to decrease with increasing irradiation dose, suggesting a growth in defect size, which is further evidenced by combined analyses between pop-in and hardness tests.

Quantitative characterization of early stage defect production and evolution in ion-irradiated materials is essential to both fundamental materials science and various application fields [1–3]. For example, it bridges atomistic simulations, which primarily focus on the low dose regime, to the experimentally observed microstructure and property evolution after long-term irradiations [3–5]. In addition, it is required in developing and validating novel design concepts of irradiation-resistant materials that suppress early stage damage accumulation [6, 7].

Nonetheless, quantification of low-dose ion irradiation damage has been a long-standing challenge, mainly attributed to the small defect size and the limited penetration depth. The former leads to difficult statistical analyses in microscopic studies; the latter makes thin film samples required for electrical resistivity measurements [8], which usually brings a tedious or challenging sample preparation process, especially when specific material microstructures and configurations are required.

Pop-in phenomenon refers to the sudden displacement excursion on the load-displacement curves during nanoindentation tests, and arises from the onset of elastic-plastic transition [9–13]. In crystalline materials, pop-in originates from homogeneous dislocation nucleation at the theoretical strength, if no lattice defects are initially present. Pre-existing defects can serve as heterogeneous dislocation nucleation sources that require lower initiation stresses [9, 13], see Fig. 1a; the defect density and strength can both be derived from the cumulative probability distribution of the pop-in loads using a statistical model [9]. Pop-in load is very sensitive to crystal damage, which probably leads to the fact that pop-in has hardly been observed in previous nanoindentation studies on irradiated materials, without quantitative knowledge on how pop-in behavior progresses under irradiations [14, 15].

In this study, we investigate the impact of ion irradiation on the incipient plasticity of single crystalline Molybdenum (Mo), and demonstrate nanoindentation pop-in test as an effective quantification method in studying early stage irradiation damage. Utilizing a statistic model, pop-in tests can quantify not only the density of defect clusters, but also the change in defect strength, which is further correlated to the defect size. Moreover, the damage accumulation is compared with that characterized using ion channeling, a widely used technique for irradiated single-crystals [16].

Pure Mo (99.99% purity) with (100) surface was homogenized at 1600 °C for 4 h under vacuum. The samples were ground and polished using a standard metallographic procedure, and then electro-polished in a solution of 12.5% H_2SO_4 and 87.5% CH_3OH at a DC voltage of 10 V to remove the surface mechanical damage. The samples were irradiated with 10 MeV Ni ions at room temperature, with fluences from 2.5×10^{12} to 1.6×10^{14} cm^{-2} and a constant flux of 3.5×10^{11} cm^{-2} s^{-1} [17]. The profiles of displacement and implanted ions were estimated using SRIM

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* Corresponding author.
E-mail address: hbei1@utk.edu (H. Bei).

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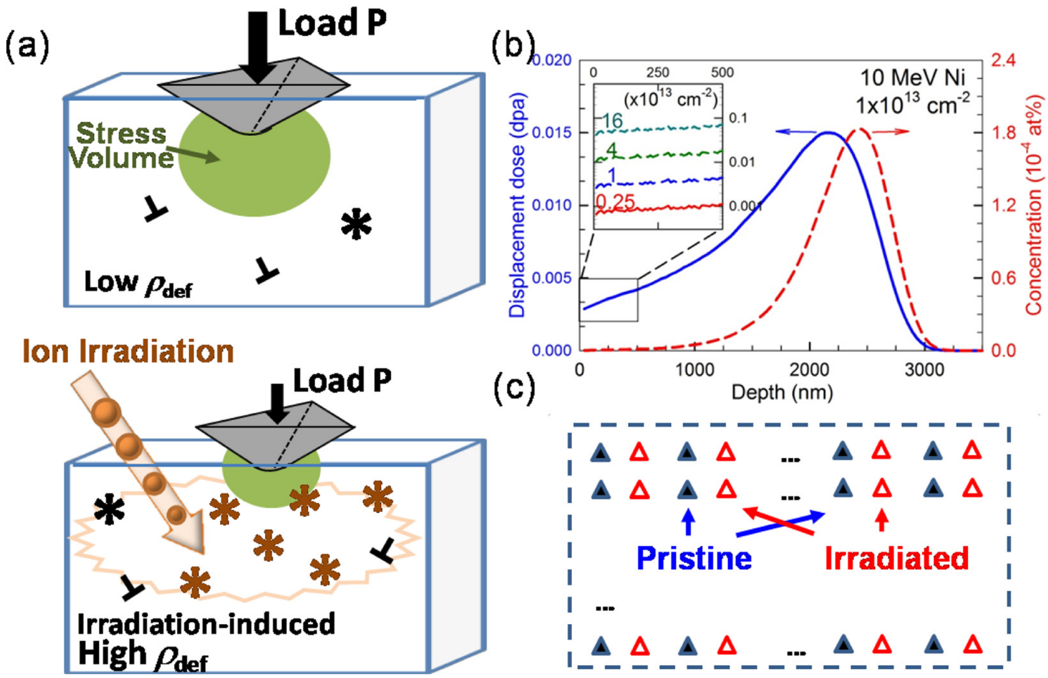

Fig. 1. Illustration of concepts and experiments. (a) Schematic illustration of the dominant mechanisms of nanoindentation pop-in before and after ion irradiations. "⊥" represents the Frank-Read sources, e.g. dislocations, and "*" represents the defect clusters acting as heterogeneous dislocation nucleation cores. (b) SRIM predicted damage and ion profiles after 10 MeV Ni ion irradiations in Mo at the fluence of 1×10^{13} cm^{-2}; the inset is the enlarged depth region of 0–500 nm at all four fluences. (c) The arrangements of indents of nanoindentation tests.

[18] simulation in the Kinchin-Pease mode, assuming a threshold displacement energy of 33 eV [19], as shown in Fig. 1b. The damage peak is located at ~2.2 μm, and the local displacement dose only gradually changes in the surface regime to a relatively small extent (inset of Fig. 1b). Since pop-in occurs within a few tens of nanometers from surface (as will be shown below), the surface dose values, rather than the peak values, are used in the present study, ranging from ~7.5×10^{-4} to 4.8×10^{-2} displacements per atom (dpa). Almost no implanted Ni ions were introduced in the surface region. Ion channeling measurements were performed using 3.5 MeV He ions with a 155° scattering angle [17].

Nanoindentation tests were performed using Nanoindenter XP (Nano Instruments Innovation Center, MTS Corporation, Knoxville, TN), in the same region before and after irradiations in an alternating pattern for each dose as shown in Fig. 1c. A Berkovich triangular pyramid indenter with a blunt tip was used, and the effective tip radius was calibrated as $R = 310$ nm using a standard tungsten sample [20]. The tests were conducted in the continuous stiffness mode (CSM) [21], at a constant $\dot{P}/P = 0.05$ s^{-1}, where P is the load. About 100 indents were performed at each condition for low uncertainty. The indents were separated by at least 20 μm to avoid interference.

Typical displacement-load curves that correspond to the average pop-in load are shown in Fig. 2a for the pristine and irradiated samples. Before pop-in, the load-displacement, *P*-*h*, curves fit well the Hertzian equation,

$$P = \frac{4}{3} E_r \sqrt{R} h^{3/2}, \tag{1}$$

where E_r is the reduced modulus, indicating a pure elastic response. As shown in the inset of Fig. 2a, pop-in load is very sensitive to irradiation damage, and is dropped by 70% after irradiation to the lowest fluence,

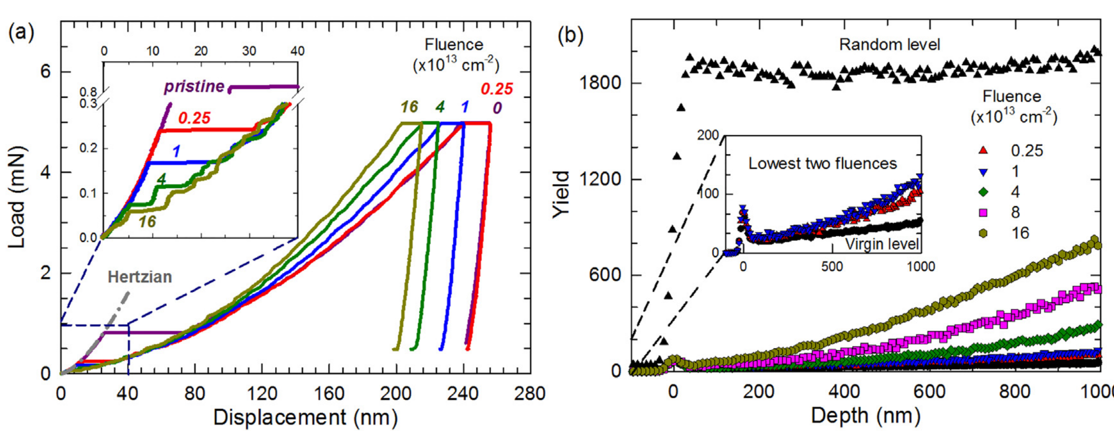

Fig. 2. Load-displacement curves and ion channeling spectra. (a) Typical load-displacement curves of Mo after irradiations with fluence from 2.5×10^{12} to 1.6×10^{14} cm^{-2}. Inset is the enlarged area with displacement below 40 nm. (b) RBS/C spectra of Mo from 2.5×10^{12} to 1.6×10^{14} cm^{-2}. Inset is enlarged for the lowest two fluences.