

Direct observation of nanoindentation-induced punching-out of prismatic dislocation loops in tungsten

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While bulk bcc plasticity is traditionally governed by screw dislocation glide, prismatic dislocation loops are proposed as an essential pathway for strain accommodation under severe, local constraints. This experimental study on pure W, as a model bcc system, demonstrates that prismatic dislocation half-loops can develop and propagate in a triaxial stress field as imposed by nanoindentation. Using electron channeling contrast imaging, the Burgers vector of emitted dislocation half-loops is determined to be $1/2 \langle 111 \rangle$ and the punching-out process is studied during progressive stages of imposed strain. Long arrays of dislocation half-loops are emitted during the initial displacement burst (pop-in) and continue to propagate during subsequent loading. Although energetic considerations indicate that loop formation alone cannot account for the total plastic deformation energy—implying a concurrent contribution from glide mechanisms—these findings reveal that plastic flow under severe hydrostatic stress gradients can be accommodated via non-Schmid loop punching. Crucially, the unique slip system multiplicity of the bcc lattice yields highly mobile, circular loop geometries that stand in stark contrast to the faceted loops typical of fcc metals.

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The mechanism of prismatic dislocation loop (PDL) punching, first conceptualized by Seitz [1], represents a fundamental pathway for crystals to accommodate highly localized volume displacement. While PDLs are conventionally associated with nonconservative processes such as irradiation, they can also form conservatively through complex mechanical shear and cross-slip reactions under extreme localized stress. In face-centered-cubic (fcc) metals, this mechanical emission of PDLs has been extensively documented both experimentally [2–6], via molecular dynamics (MD) [5,7–10] and discrete dislocation dynamics simulations (DDD) [8,11–13]. In contrast, isolating this behavior in body-centered-cubic (bcc) metals remains exceptionally challenging due to their high Peierls barriers and nonplanar dislocation core structures, which dictate distinct thermally activated glide kinetics [14]. Consequently, because these intrinsic properties govern highly dynamic, subsurface defect interactions, the exact mechanisms of incipient plasticity and loop punching in bcc systems have largely remained confined to computational modeling. MD [15–19] and DDD [20] simulations of nanoindentation have repeatedly predicted that complex stress fields trigger stable PDL emission. Distinct formation pathways, such as local cross slip of shear loops

[17] and twin-induced formation [18] have been found computationally. However, direct experimental confirmation has proven elusive. Although recent nanoindentation studies on pure W observed elongated dislocation configurations, their exact identity could only be conjectured to be prismatic loops [21,22].

In this work, we provide direct experimental evidence of deformation-induced PDL formation in bcc metals. Using W as a model bcc material, we demonstrate that the complex, high-stress fields during nanoindentation punch out distinct arrays of PDLs. By systematically utilizing electron channeling contrast imaging (ECCI), we clearly resolve the character and Burgers vectors of these loops in bulk specimens, thereby bridging the gap between classical geometric predictions and modern multiscale computational simulations. Furthermore, by tracking the structural evolution of the PDL arrays across progressive indentation loads, we provide key insights into a fundamental nanoscale mechanism for strain accommodation in metals.

The material used was high-purity (> 99.999 wt%) single-crystalline W having [011] surface normal direction. The sample was mechanically polished with diamond suspensions down to particle sizes of $1 \mu\text{m}$ and then electrolytically polished with an ice-cold aqueous 2 wt% NaOH solution. Nanoindentation was performed on a TI950 TriboIndenter (Bruker, USA) using a blunt diamond Berkovich tip and a sharp diamond cube-corner tip. Tip radii were calculated by the Hertz elastic contact model [23] to be approximately 700 and 40 nm, respectively. Indentation using the Berkovich tip was conducted at a loading rate of $1000 \mu\text{N/s}$ to peak loads of $4500 \mu\text{N}$ and $10\,000 \mu\text{N}$ at which the load was held constant

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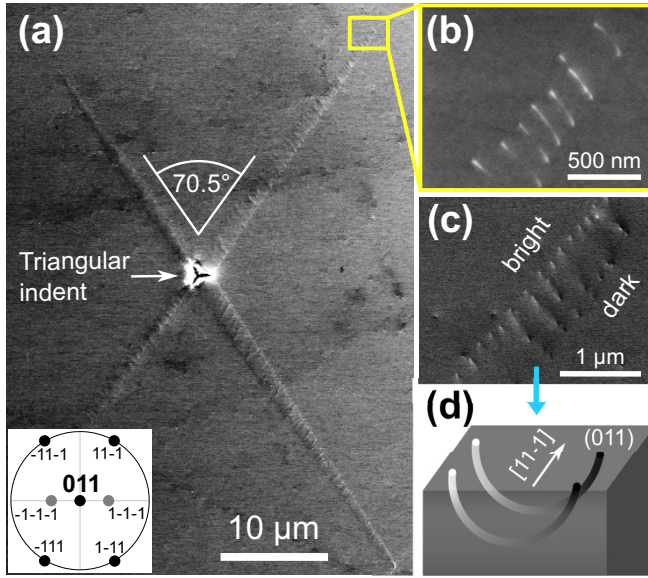


FIG. 1. Backscatter electron micrograph of (a) a Berkovich indent with emanating streaks of dislocations. The inset shows a stereographic projection with black dots representing $\langle 111 \rangle$ directions parallel to the surface and grey dots representing those pointing into the sample. (b) The tip of the streak marked in (a) in channeling condition, (c) opposite contrast of dislocation dipoles in a streak during out of ideal channeling condition and (d) schematic of two dislocation dipoles from (c).

for 10 s. Indents with the cube-corner tip were conducted at a loading rate of 1 $\mu\text{N/s}$ to various peak loads ranging from 100 to 1000 μN , each with a holding segment of 10 s. For each setup, machine compliance and tip area function were carefully calibrated prior to testing following the Oliver-Pharr method on a reference fused silica sample. Crystal orientations were determined via electron backscatter diffraction using an e-FlashHD detector (Bruker Nano GmbH, Germany) in a scanning electron microscope (SEM) Crossbeam 550 (Zeiss, Germany) featuring the Gemini II SEM column and applying a 20 kV acceleration voltage and 10 nA beam current. ECCI was done under the same current and 30 kV. For ECCI a circular four-quadrant backscatter electron (BSE) detector located below the polepiece was used with all quadrants turned on and gain set to medium. According to the required sample tilts a working distance between 5 and 10 mm was used. Approximate channeling conditions in the SEM were achieved via controlled ECCI [24] and position fine tuning done via comparison of observed electron channeling patterns with a simulated dynamical diffraction pattern as described in Sec. I of the Supplemental Material [25].

Figure 1(a) presents a BSE micrograph of the W (011) surface after Berkovich indentation with a peak load of 10 000 μN . The triangular indent is indicated by an arrow, and a stereographic projection showing the crystal orientation of the sample is inserted at the bottom-left corner of the image. Indentation resulted in X-shaped streaks extending more than ten times the indent size from the indent center. The four visible directions of propagation emanating from the indent follow the crystallographic $\langle 111 \rangle$ directions. Measuring

the included angle of the streaks resulted in $70.5^\circ \pm 0.5^\circ$ [Fig. 1(a)], perfectly agreeing with the ideal angle of 70.53° .

Close observation of the tip of the streaks in the area enclosed by the yellow rectangle in Fig. 1(a) is presented in Fig. 1(b). The image reveals pairs of visible points that are connected by lines as can be seen towards the end of the streak. The fading contrast with increasing depth, the fact that these features are invisible to secondary electrons and that they vanish under certain directions of observation are clear indicators that these must be dislocation lines. Accordingly, the observed streaks consist of dislocation half-loops.

Figure 1(c) shows an ECCI micrograph of an area on the streak about 20 μm from the indent, where only dots are visible. ECCI reveals dislocation contrast up to ~ 100 nm below the surface [24], explaining why full lines appeared visible at the streak ends where the half-loops were shallow and small, while larger loops closer to the indent extended deeper into the material so that only their near-surface interception points remained visible as dots characteristic of dislocation dipoles. Moreover, the crystal could be tilted out of perfect channeling conditions so that dipoles showed opposite bright-dark contrast as seen in Fig. 1(c), indicating the opposite sign of the line vectors of corresponding threading dislocations.

The dislocation half-loops were parallel to each other and perpendicular to the streak direction as shown schematically in Fig. 1(d). For the 10 000 μN indent they exhibited a mutual spacing of about 200–500 nm close to the indent and mostly 100–200 nm at the streak tip. The loop diameter decreased from roughly the indent size down to 300 nm at the streak tip. These values are highly reproducible. The streaks also show an increased surface elevation on the order of 1 nm, as reported previously [22]. Accordingly, material transport occurs not only laterally but also vertically to a small extent.

Burgers vectors $\mathbf{b}$ were evaluated by a transmission electron microscopy (TEM)-style diffraction contrast analysis based on the invisibility criterion, stating that dislocations become invisible when the condition $\mathbf{g} \cdot \mathbf{b} = 0$ is met [26], where $\mathbf{g}$ represents the diffraction vector. While TEM offers the advantage of rotational freedom, accurate alignment to two-beam conditions and thus clear $\mathbf{b}$ determination, it is limited in the sample volume that can be probed, and requires sample preparation via focused ion beam, which might risk the alteration of surface-near dislocations. Moreover, creating free surfaces in the stressed volume around the indent by material removal may lead to relaxation and annihilation of the dislocation loops. Therefore, an analysis approach via ECCI was employed, which follows the same physical principles as in TEM and additionally allows for quick analysis of multiple indents.

Figures 2(a)–2(d) show four diffraction conditions employed to unequivocally identify $\mathbf{b}$ at one end of a dislocation streak. Due to symmetry considerations the findings apply to all four directions of observed streaks. The dislocation contrast is clearly visible in Fig. 2(a) for $\mathbf{g} = (-200)$ and in Fig. 2(b) for $\mathbf{g} = (01-1)$. Conversely, dislocations are invisible in Fig. 2(c) for $\mathbf{g} = (-21-1)$ and in Fig. 2(d) for $\mathbf{g} = (3-21)$. The expected visibilities of possible $\mathbf{b}$ in bcc metals and the observed visibilities are summarized in Sec. II of the Supplemental Material [25]. It is apparent that only $\mathbf{b} = 1/2 [11-1]$ satisfies these invisibility conditions. Since this $\mathbf{b}$ is

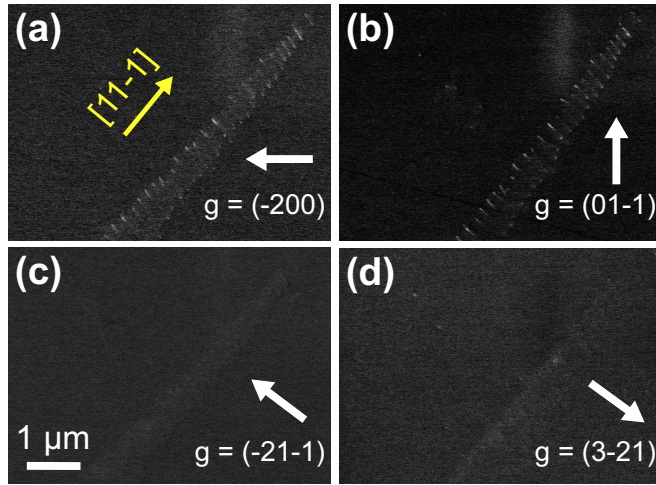


FIG. 2. Electron channeling contrast micrographs of the same location (end of a dislocation streak) for Burgers vector $\mathbf{b}$ determination. Dislocations are visible in (a), (b) and invisible in (c), (d). The yellow arrow shows the direction of $\mathbf{b}$; white arrows represent the respective diffraction vectors $\mathbf{g}$ for the set of diffracting planes (hkl).

perpendicular to the whole segment of the dislocation line, the observed half-loops can be identified as edge dislocations, which explains why they could propagate far outward from the indent. Furthermore, regarding the travel direction parallel to $\mathbf{b}$, the observed loops are almost certainly PDLs. Contrarily, shear loops formed purely by glide would lie within a specific slip plane and would almost inevitably contain mixed and screw segments. The punching-out of prismatic loops is also in agreement with accommodating the volumetric deformation imposed by the indenter.

To understand the temporal evolution of the streaks, Berkovich indents were carried out at different peak loads. Figures 3(a) and 3(b) show BSE images of the sample surface after indentation with peak loads of 10 000 and 4500 μN , respectively. Corresponding load-displacement curves are presented in Fig. 3(c). All indents at these loads exhibited pronounced pop-in at around 4500 μN . It should be noted that the pop-in event occurred during the holding segment at peak load in the case of Fig. 3(b), indicating that the streaks were formed at pop-in. A comparison of streak lengths for the two different loads and the four observed directions is provided in Fig. 3(d). Indents with the same peak load featured similar streak lengths. While streaks were shorter for lower peak loads, streaks were still present despite pop-in occurring during the holding segment. Since the pop-in load was naturally the same for different peak loads but the streak length differed with peak load, subsequent plastic deformation after pop-in likely dominated the final streak length. Therefore, we suggest that PDLs were instantaneously emitted at pop-in and subsequently traveled during further penetration of the indenter tip.

Moreover, the streak length varied with orientation relative to the indent geometry. The length was almost double if the streak was emitted from the side of the indenter [streaks to the right side on Fig. 3(a)] as compared to emission from a

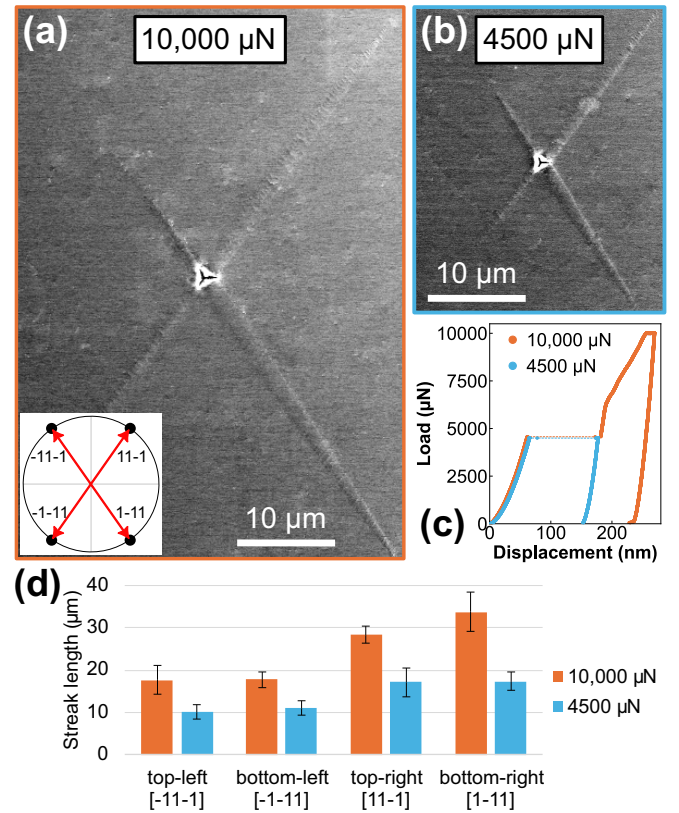


FIG. 3. Berkovich indents at peak load of (a) 10 000 μN and (b) 4500 μN . The inset in (a) shows a stereographic projection where red arrows denote the $\langle 111 \rangle$ streak directions. (c) shows the respective load-displacement curves; and (d) dislocation streak lengths across identical indents and indents at different peak loads. Error bars denote standard deviations for 10 measured indents at each load.

corner (streaks to the left side). Tests with indents rotated by 45° confirmed this correlation and corroborated that streak direction depends solely on crystal orientation (Sec. III of the Supplemental Material [25]).

For improved resolution of the PDL emission event, a sharper tip was required to enable plasticity at lower loads. BSE images of indents produced by a cube-corner tip at peak loads of 1000 and 100 μN are displayed in Figs. 4(a) and 4(b), respectively. As the indent dimensions decreased, the streak length, dislocation loop diameter and loop spacing scaled down. The magnified region in Fig. 4(a) highlights the streak tip, revealing a PDL diameter of approximately 100 nm. Notably, this loop diameter is only a third of that induced by the blunter Berkovich tip in Fig. 1(b).

With decreasing tip radii, the load-displacement curves of bcc metals typically transition from a single pronounced pop-in to a staircase behavior, where the intervals between the displacement bursts behave elastically [27]. For the indent shown in Fig. 4(b), between two and nine PDLs are visible depending on the examined direction.

The corresponding staircase-shaped load-displacement curve, presented in Fig. 4(c), displays nine distinct displacement bursts prior to unloading. While it seems plausible that each discrete displacement burst may directly correspond to the emission of at least one surface-visible PDL, it is

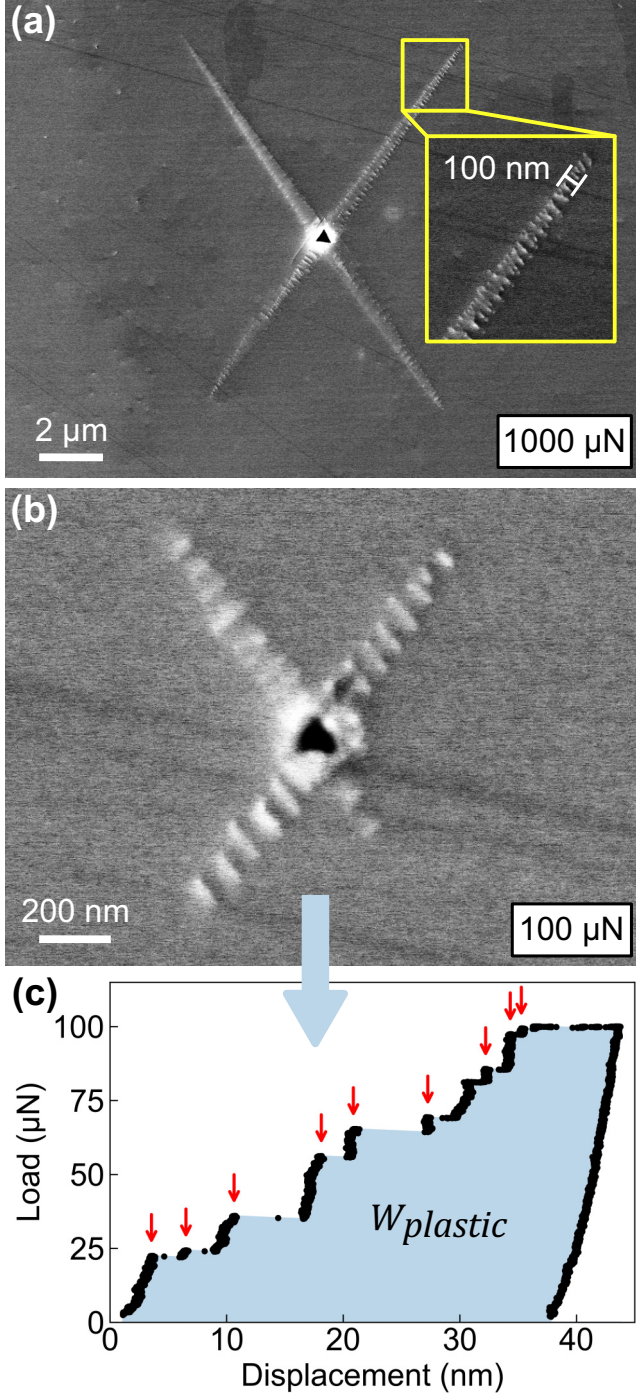


FIG. 4. Backscatter electron micrographs of sharp cube-corner indents at peak loads of (a) 1000 μN , with the enlarged area indicating the dislocation loop diameter at the streak tip, and (b) 100 μN . (c) Load-displacement curve of the indent in (b), where the shaded area represents the integrated plastic deformation energy W_{plastic} . Pop-ins are marked with red arrows.

conceivable that the major resistance against deformation may stem from subsurface dislocation nucleation or a dislocation network evolution below the indenter. In this scenario, plastic strain may be accommodated by the other two potential $1/2$ $[1-1-1]$ and $1/2$ $[-1-1-1]$ Burgers vectors pointing into the

depth directions [see inset in Fig. 1(a)], rendering the emitted surface PDLs a secondary byproduct.

The high reproducibility of even the smaller indents coupled with sufficient resolution of individual dislocations allows for an energetic consideration to estimate the contribution of the visible dislocations to the total strain. From basic dislocation theory the energy of a dislocation loop is the sum of its self-energy and its interaction energy with other dislocation loops [14]. Tsuru *et al.* [28] have derived a relation for the total energy W_D of a line of parallel dislocation loops with similar spacing d_D and loop radius a ,

$$W_D = n_s \frac{Gb^2}{1-\nu} a \left\{ \frac{N}{2} \left(\ln \frac{8a}{\rho} - 1 \right) + 2\pi \left(\frac{a}{d_D} \right)^3 \sum_{\alpha=1}^{N-1} \frac{N-\alpha}{\alpha^3} \right\}, \quad (1)$$

where G is the shear modulus, b is the magnitude of the Burgers vector, ν is the Poisson ratio, ρ is the dislocation core cut-off radius here taken as $\rho = b$, n_s is the number of active slip systems, and N is the number of dislocations in the array. We take the indent in Fig. 4(b) as proxy for present calculation. Four slip systems corresponding to the four streaks are activated (not taking into account the other two $\langle 111 \rangle$ directions pointing into the depth from the indent) and an average of seven dislocations per slip system. As half-loops instead of closed PDLs are present, we estimate the overall energy of the visible half-loops by taking 50% of W_D . The average loop diameter was measured to be about 50 nm with a mean PDL spacing of 70 nm. Taking G as 160 GPa, ν as 0.28 and b as 0.274 nm, a total energy of 6.9×10^{-14} J is obtained. If using the experimentally determined value for G from 100 indents of 124 ± 3 GPa instead, W_D results in an even lower value of 5.3×10^{-14} J. The discrepancy between theoretical and measured G most likely stems from an elastic contribution of the sample fixture. The W_D can be compared to the overall plastic deformation energy of the nanoindent W_{plastic} , by integrating the load P over the displacement h . Hereby, a possible influence from energy dissipation via machine compliance is already accounted for owing to the initial indenter calibration procedure. W_{plastic} is equivalent to the elastic and plastic work done during loading W_{loading} minus the recovered elastic energy during unloading $W_{\text{unloading}}$ [Fig. 4(c)] given as

$$W_{\text{plastic}} = \int_0^{h_{\text{max}}} P(h) dh = W_{\text{loading}} - W_{\text{unloading}}. \quad (2)$$

The resulting $2.3 \times 10^{-12} \pm 0.2 \times 10^{-12}$ J of total indentation work is more than 30 times larger than the energy of the observed PDLs. Even if streaks of complete PDLs are formed in a similar manner in the other two $\langle 111 \rangle$ directions, deformation energy cannot be accommodated only by PDL formation. Instead, PDLs appear to contribute to a cascade of strain evolution events that collectively require much higher energy levels than PDL formation alone. Graca *et al.* arrived at an analogous conclusion for PDLs in nanoindented Ni using force-based reasoning [4], which aligns with MD simulations that show required precursor rearrangement steps, such as shear loop nucleation and cross slip, prior to PDL emission [17].

Combining observations from the present study with findings from MD simulations [5,9–11,15,17,29], a plausible sequence for the emission and propagation of PDL arrays can be reconstructed. Indentation generates a triaxial stress field with large shear components that induce dislocation nucleation and interaction at the first displacement burst, during which a loop with $\mathbf{b} = 1/2 \langle 111 \rangle$ is produced. Once formed, the PDL experiences a driving force from the indentation stress field that pushes it away from the highly stressed zone via a classic loop-punching mechanism. The local microstructural configuration at the nucleation site can act as a stable source, sequentially emitting multiple loops. While the indenter stress field drives the initial movement of the PDLs, after a certain propagation distance, the stress field from adjacent loops dominates, pushing the array further outward [11]. Notably, the first emitted PDLs along the streaks exhibit smaller diameters than those emitted later. This trend indicates that the loop diameter at emission increases as the imposed volumetric change accumulates. Furthermore, because the dislocation lines are most visible—and thus closest to the surface—at the streak tips, these early PDLs likely glide outward with negligible changes in depth or diameter during propagation. Such stable, long-distance glide is expected for edge dislocations in high-purity single crystals due to their structural inability to change glide planes and the negligible resistance to climb in W at ambient temperatures.

Interestingly, PDLs are punched out along directions where macroscopic shear stresses are conventionally considered to be zero due to a vanishing Schmid factor perpendicular to the loading axis. This apparent violation of Schmid's law arises because PDLs glide along a three-dimensional cylindrical surface driven by severe localized hydrostatic stress gradients rather than uniform resolved shear stress on a single flat slip plane. Therefore, their propagation fundamentally eludes description by classical Schmid kinetics. Instead, the availability of $\langle 111 \rangle$ directions enables easy glide, that channels the plastic flow laterally away from the advancing indenter surface. The PDLs represent the elementary steps of material cylinders being displaced laterally to accommodate indenter volume. While this volumetric relaxation mechanism appears to be universally operative across bcc, fcc, and potentially hcp

structures, a distinct crystallographic divergence emerges in the loop morphology. Whereas fcc loops often feature faceted shapes bounded by less glissile dislocation jogs [6], the bcc PDLs observed here are strikingly circular as evidenced by the gradual fading of dislocation contrast in Fig. 1(b). This circular morphology is driven by line-tension energy minimization, facilitated by the high multiplicity of symmetrical, intersecting slip systems available in the bcc lattice. Consequently, this isotropic energy landscape likely enhances loop mobility, enabling the formation of the exceptionally long, extended streaks of punched-out PDLs.

In conclusion, direct observations demonstrate that mechanically driven PDL generation and punching are active mechanisms in bcc metals under severe, local constraints. Through ECCI of nanoindented pure W, we tracked the structural evolution of long dislocation streaks along the $\langle 111 \rangle$ directions. These defect arrays are generated at the initial pop-in burst and extend during subsequent deformation. Determining $\mathbf{b}$ via ECCI provides clear experimental evidence that validates prior microstructural conjectures and directly bridges the long-standing gap with atomistic and discrete dislocation dynamics simulations. Consistent with these simulations, the circular shape of the PDLs and their ease of glide appear to be characteristic features of the bcc lattice, while the mechanically induced punching mechanism is observed universally across the major crystal structures. Furthermore, energetic considerations indicate that loop formation alone cannot account for the total plastic deformation energy of indentation, implying that other mechanisms, such as classical shear loops, contribute concurrently. Ultimately, these observations resolve historical ambiguities regarding subsurface defect transport in bcc systems under highly confined deformation.

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Data availability. There are no publicly available research data or software supporting this manuscript. Requests for further information or data should be sent to the authors.

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