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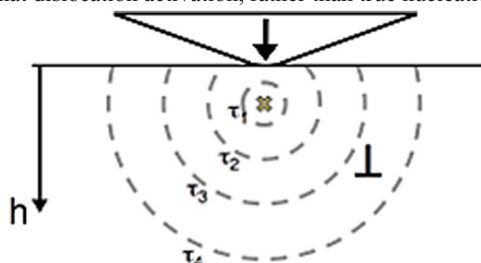
## Discontinuous Yield Behaviors Under Various Pre-Strain Conditions in Metals with Different Crystal Structures

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Instrumented indentation was performed to determine the statistics of discontinuous yield behavior of Co and Ni pre-strained to various levels. In both materials, increasing pre-strain decreases the frequency of indentations that exhibit discontinuous yield. Yield events in Co occurred at nominally the same stress independent of pre-strain, suggesting dislocation nucleation dominates during contact loading and that the existing dislocations are strongly pinned in Co. However, in Ni yield occurred at lower loads with increasing pre-strain, suggesting that dislocation activation, rather than true nucleation, dominates the yield mechanism.



**Keywords:** Nanoindentation, Plasticity, Dislocation Nucleation, Theoretical Shear Strength

**Introduction** Incipient yield, often referred to as pop-in, is a phenomenon unique to micro- and nanoindentation tests in relatively dislocation free volumes of crystalline material.[1] The stress at which pop-in occurs often approaches the theoretical shear strength of the material and on oxide-free metallic surfaces, pop-ins are often assumed to be due to the nucleation of new dislocations in the material.[1–4] Simulations showing dislocation avalanches when a characteristic load and associated shear stress is reached do corroborate this assumption.[5–8] However, experimental evidence and some simulations suggest that the pop-in load (or stress) may not be a representative of the theoretical shear strength, which would make it not a characteristic material property, since this load is subject to considerable variation, and in-fact is often not observed.[9–13] Researchers have studied the sources of variation in

the pop-in load. Several groups have demonstrated that the dislocation density of the underlying crystal influences the pop-in load.[12,14–16] Lodes et al. studied pop-in with experiments and simulations in CaF<sub>2</sub> single crystals in deformed and undeformed conditions. They found that when the pre-strained material is indented the motion of pre-existing dislocation loops assists the initiation of plasticity.[12] Catoor et al. [17] studied the initiation of plasticity in single crystals of Mg via spherical indentation and transmission electron microscopy (TEM). Their experiments showed that in the case of this hexagonal close packed (HCP) single crystal, pop-ins were always caused by nucleation of  $\langle a \rangle$ -type dislocations. Simulations of contact loading have shown that pop-in can be influenced by the presence of defects in the crystal lattice,[6–8] while experiments have also observed a dependence on grain orientation and tip

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geometry.[18] Lund et al. demonstrated that the pop-in load can vary with temperature or imposed strain rate (of course, strength may also vary with temperatures and strain rates).[19]

Since the literature reports many causes for differing pop-in load, in the present study, we have attempted to isolate the effects of pre-strain and crystal structure on the incidence of pop-in in materials with two different crystal structures but similar shear moduli to shed further light on the mechanism by which pop-in occurs. Further, this investigation has been conducted using a statistical approach towards indentation investigations to span multiple grains in multiple samples and examine if this testing methodology is robust enough to imply mechanisms which are appropriate for a wide range of orientations.

**Methods** Cobalt (Co) and Nickel (Ni) have nearly identical shear moduli, and thus should have similar theoretical shear strengths, but different crystal structures at room temperature. The magnitude of the Peierls stress is similar for face-centered cubic (FCC) and basal slip HCP metals and on the order of  $10^{-5}G$  to  $10^{-6}G$ , where  $G$  is the shear modulus.[17] The Co used in this study was 99.9 + % purity (Alfa Aesar). Prior to any preparation, the Co had been stored at room temperature for at least 10 years. The Ni obtained was more than 99.999% pure and having been treated by multiple zone refining passes can be considered to be well annealed.

Several samples of Co were used in this study and were prepared as follows. The as-received material was cut in two. One piece was annealed at 1,000°C for approximately 2 h in a 95%-Ar 5%-H atmosphere and allowed to slow cool in the furnace over a period of 4 h. This should relieve any cold work in the material and grow the grain size, but it should be noted that the transformation from the high temperature FCC to the low temperature HCP phase at 417°C [20] leaves transformation partial dislocations and stacking faults, and perhaps as much as 30% of metastable FCC regions.[21,22] The transformation from FCC to HCP phase below the transformation temperature can be induced by straining the material.[23] Next, both pieces were ground with silicon carbide paper then cut into smaller pieces approximately  $10 \times 10 \times 10$  mm using a diamond saw. This resulted in two samples that had been annealed, and four that were in the as-received condition. One as-received sample was rolled to each of 5%, 10%, and 20% additional strain, while one was left with no additional strain. Prior hardness experiments on the as-received samples indicate that they were at approximately 2% strain relative to our well-annealed piece, meaning that these samples were deformed to about 2%, 7%, 12%, and 22% strain after rolling. One annealed sample of Co was rolled to 5% strain and the other left with no additional strain.

Three samples of Ni were cut from the purified rod. One sample was cold rolled to each of 7% and 11% strain and one left in the unstrained condition. All samples were then mechanically polished with diamond and alumina through  $0.05 \mu\text{m}$ . The Ni samples were electropolished in a mixture of 37% phosphoric acid, 56% glycerol, and 7% water. The Co samples were electropolished in pure phosphoric acid. The electropolishing should remove the surface material with any residual stress and defects in the lattice from the mechanical polishing steps. A Hysitron Triboindenter (Eden Prairie, MN) fitted with two different Berkovich probes was used to indent the samples. The first probe was used to collect hardness measurements and was in new condition, having an apex radius of about 150 nm. The second probe, used for the pop-in tests, was worn and had a relatively blunt apex geometry. The effective radius of this probe was determined by fitting Equation (1) [24] to the nominally elastic portion of nine indents displaying pop-in behavior in a single crystal of tungsten.

$$P = \frac{4}{3}E_r\sqrt{Rh^3}. \quad (1)$$

Here,  $R$  is the radius of the probe,  $E_r$  is the reduced modulus of the sample,  $h$  is the displacement of the indenter from the point of initial contact, and  $P$  is the load at which the pop-in occurred. The effective tip radius of the blunt probe was 980 nm.

Before testing for pop-in, all samples were subjected to checks for elasticity in a manner consistent with a prior study.[13] The samples reliably displayed pop-in without detected plasticity prior to the pop-in event; likely due to the electropolishing step having removed the vast majority of damage induced by mechanical polishing. Each sample was subjected to 36 load-controlled indentation tests to determine the pop-in load distribution. The load-depth curves were analyzed for pop-in using a freely available software.[25] Only pop-ins greater than 3 nm were counted in this analysis. For each test, the load at which the pop-in occurred was recorded. For tests where no pop-in occurred, the load was recorded as zero.

**Results and Discussion** Typical load versus depth curves for the two materials are shown in Figure 1. Four curves are shown, one each from the following four conditions: Ni at 0% pre-strain, Ni at 11% pre-strain, Co at 2% pre-strain, and Co at 22% pre-strain. A test that displays pop-in and a test the displays ‘no pop-in’ are shown for each material.

Hardness values for all of the samples tested are given in Figure 2. The hardness was collected between 50 nm and approximately 300 nm depth. Little to no hardness change is generated by this amount of strain for the Co, but the Ni displays a consistent increase in

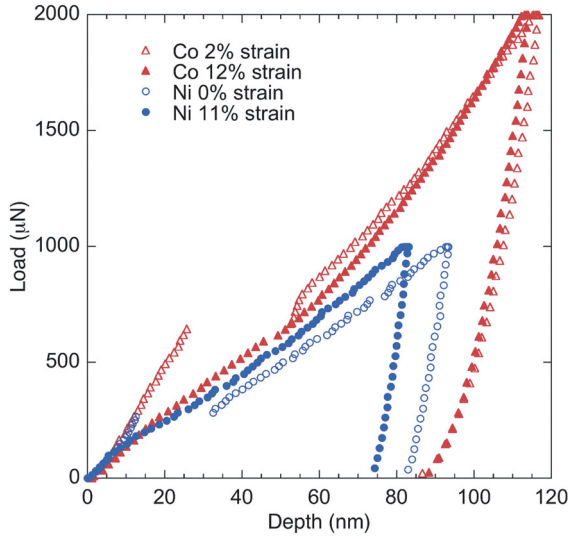


Figure 1. (Colour Online) Typical load versus depth curves for Co and Ni. Two different strain conditions are shown to demonstrate the material behavior of pop-in and ‘no pop-in’.

hardness with increasing strain. Applying an Orowan relationship to infer the dislocation spacing from the hardness suggests that for the Co samples, the dislocation density is relatively unchanged with additional pre-strain. By contrast, when considering the Ni samples, the dislocation spacing is reduced with additional pre-strain.

Referring again to the typical load versus depth curves of Figure 1, in indentations where pop-ins did occur, a range of pop-in length was exhibited in both materials; in general the length of the pop-in increases as the load at pop-in increases, as demonstrated in the literature.[3] In some tests, staircase yield was demonstrated, however, as previously mentioned, in this study, only pop-ins greater than 3 nm were counted, and only those that occurred first are a part of this analysis, omitting any latter staircase pop-ins. The majority of pop-ins were substantially longer than 3 nm.

Equation (2) [26] describes the shear stress in the material and was used to calculate the maximum shear stress ( $\tau_{\max}$ ) in the materials when pop-in occurred.

$$\tau_{\max} = 0.31 \left( \frac{6E_r^2}{\pi^3 R^2} \right)^{(1/3)} P^{(1/3)}. \quad (2)$$

Cumulative fraction plots of the maximum shear stress at pop-in for all tests in as-received Co, annealed Co, and Ni are shown in Figures 3(a) and (b) and 4, respectively. Tests displaying no pop-in have the shear stresses recorded as zero on the left side of the figure. From Figure 3(a), it can be seen that the average pop-in stress of those indentations in Co that do exhibit pop-in is not influenced considerably by the amount of pre-strain in the material. However, the number of no-pop-in tests does increase with increasing pre-strain. The coefficient of variation (CV) of pop-in stress (excluding no-pop-in

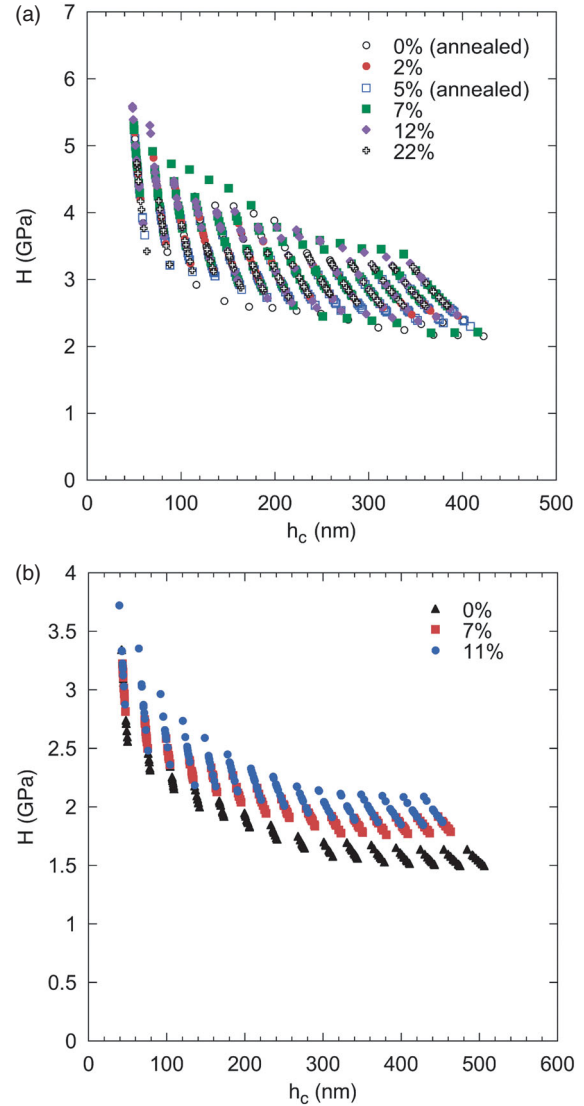


Figure 2. (Colour Online) Hardness versus depth for (a) Co and (b) Ni.

tests) for 2%, 7%, 12%, and 22% pre-strain are 0.052, 0.083, 0.17, and 0.076, respectively. For the annealed Co samples of Figure 3(b), the CV is 0.063 and 0.091 for the 0% and 5% strained samples, respectively. These annealed Co samples display more tests that do not pop-in than similarly strained samples that were not annealed. The range of pop-in stress in Co for all pre-strain levels is approximately the same, with the exception of the 2% pre-strain sample, which spans a slightly smaller stress range.

Both materials, when tested at 0% strain, exhibit mean applied contact stresses which are nearly identical. Due to the similarity in expected theoretical shear strength based on the similar elastic modulus of the Co and Ni, this directly implies that random sampling of many of grains (the statistical approach taken in this work) is sufficient to capture the full distribution of the material behavior.

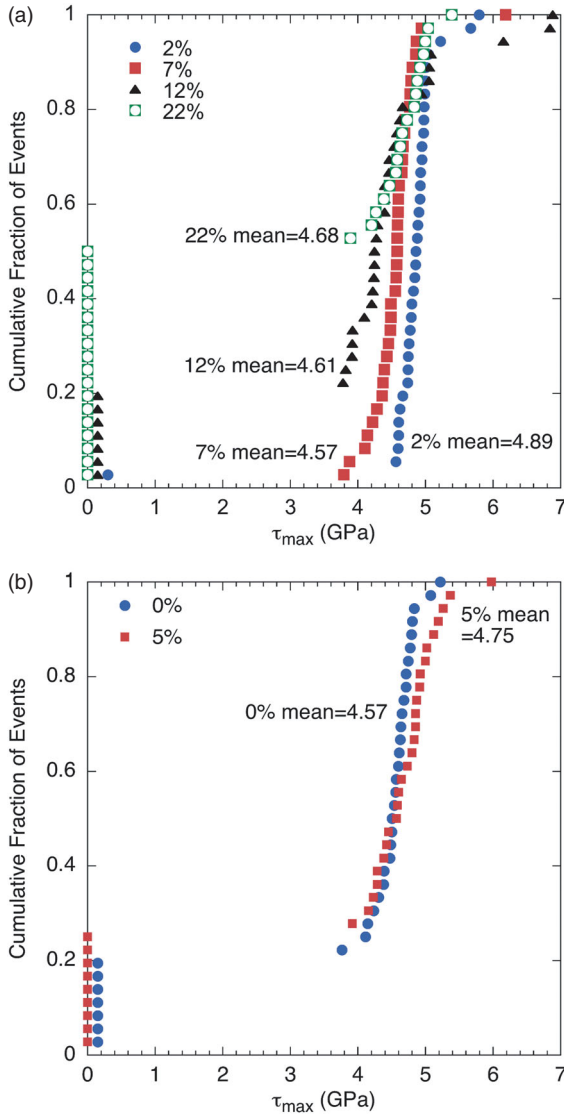


Figure 3. (Colour Online) Cumulative fraction of maximum stress at first pop-in for (a) as-received Co and (b) annealed Co.

The behavior is different in the Ni sample. Here, the average pop-in stress changes considerably with the amount of pre-strain. In addition, the number of tests not displaying pop-in is much more heavily influenced by a given amount of pre-strain than in Co. The CV of pop-in stress for the 0% and 7% pre-strained samples are 0.16 and 0.07, respectively. It should be noted that the CV for the 11% pre-strain sample is not a good indicator of variability, since only two tests can be included in the calculation.

The differing cumulative fraction curves can be explained by considering the dislocation structure and slip behavior of the two materials. To understand this, a few assumptions need to be made:

- (1) Hardness is a measure of the intrinsic lattice resistance (shear stress) to activate existing

sources and move dislocations through existing defect structures.[27]

- (2) The tested materials initially contain both glissile and sessile dislocations, with some spacing that decreases with added plastic strain in the system.
- (3) Glissile dislocations will move when the Peierls stress is reached on the dislocation's glide plane.
- (4) Glissile dislocations that have been pinned can be unlocked by a suitably high stress (a value greater than the Peierls stress).
- (5) Sessile dislocations cannot be moved by conservative processes unless plastic deformation has occurred in the material allowing the dislocation to glide on a newly created glide plane. Sessile dislocations may act as defects that reduce the stress required to nucleate a dislocation, or as obstacles to dislocation motion.

When load is applied to the material via the contact of the tip to the sample, a stress field develops in the material, with stress contours of approximately hemispherical shape centered below the contact of the indenter (variation in the stress field would be due to elastic anisotropy). The stresses decrease with increasing distance from the contact, as in the simplified depiction of Figure 5, and are described by Hertzian contact theory.[26] The stresses, upon reaching a suitable magnitude (presumably the magnitude of the Peierls stress), which occurs nearly immediately in tests at strain rates commonly used for instrumented indentation testing, cause any unpinned glissile dislocations to move away from the contact with the length of their motion proportional to the applied stress. Evidence of this is provided in micropillar compression tests [28] and indentation tests showing dislocation motion prior to discontinuous yield.[13,29]

Once any unpinned glissile dislocations have moved far from the contact, only pinned glissile or sessile dislocations remain in the area of interest, and these dislocations experience a shear stress due to the load imposed by the indenter tip. In well-annealed crystals, the dislocation density is low and it is not likely that there are two dislocations approximately equidistant from the point of contact. With increasing tip penetration into the material, the pinned dislocation near the contact experiences a greater stress. If the dislocation is near the contact, it may experience a stress great enough to unlock it or force it past an obstacle before a dislocation is nucleated at the position denoted by the X in Figure 5. The unlocking of this dislocation triggers a dislocation avalanche and a pop-in occurs. However, if there is not a pinned dislocation near enough to the contact to be unlocked before the theoretical shear strength is reached, then a new dislocation will be nucleated, again triggering an avalanche

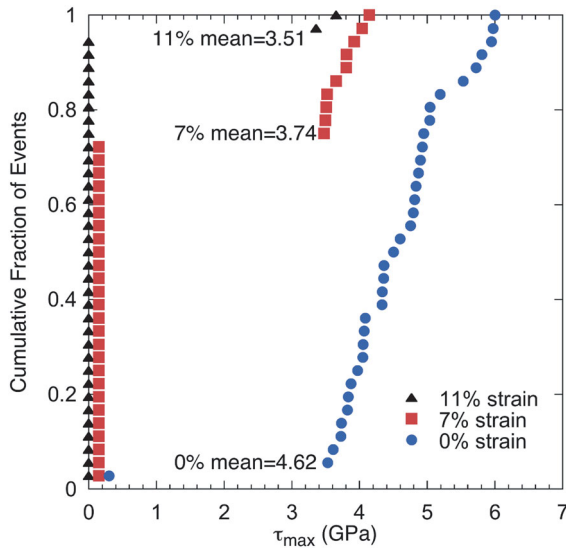


Figure 4. (Colour Online) Cumulative fraction of maximum stress at first pop-in for Ni.

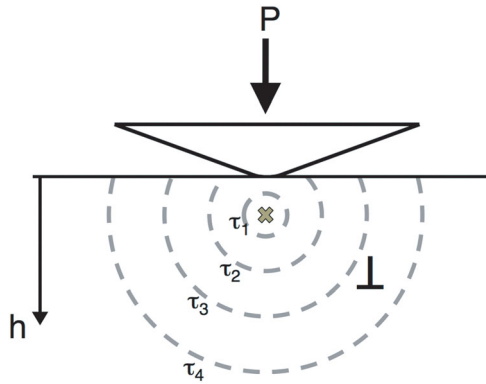


Figure 5. Schematic of indenter at first contact with the material and resulting stress contours. A dislocation exists in the material and experiences a stress of approximately  $\tau_3$ .

and causing a pop-in. This view is supported by the experiments on Co and Ni, which have nearly identical elastic moduli and consequently the same theoretical shear strength. The difference between their behaviors arises because of their differing crystal structures. Nickel is FCC and Co is HCP at the test temperature. FCC structures have five independent slip systems, while HCP have only four, with the preferred for Co being the basal plane.[30] The theoretical shear strength of both materials when taken as  $G/15$  is approximately 5 GPa. Referring again to Figure 4, it is observed in Ni that the mean pop-in stress and the number of pop-ins that occur are greatly reduced with increasing amounts of pre-straining in the material. As more pre-strain occurs, the density of dislocations in the material increases. No-pop-ins are the result of initially glissile dislocations that move under the initial loading, and multiply when they encounter other dislocations creating a Frank-Read source, or when there are already dislocations in a source configuration near the contact (pre-existing source as

described by Kreuzer and Pippan [27]). With increasing strain in the Ni, and its ability for dislocation cross-slip in the FCC structure, the number of pre-existing sources increases dramatically with more pre-strain and damage to the perfect lattice, leading to no discontinuous yielding if the volume of the material sampled by the indenter is such that existing glissile dislocations are readily available (i.e. at high pre-strains, there are always dislocations that can move in the stress field).

By contrast, the average stress at pop-in for Co, shown in Figure 3 is essentially unchanged by pre-straining. The annealed Co samples display more tests that did not pop-in as compared to similarly pre-strained as-received Co. This is probably due to a higher fraction of residual FCC phase in the recently annealed samples. Evidence of this transition is also observed with the 2–7% strained as-received samples. The 2% shows fewer pop-ins. The storage time and additional strain act to decrease the amount of FCC phase present.

The theoretical shear strength of the two materials is the same, and it appears by comparing the mean stress at discontinuous yield of Co to the 0% mean stress of Ni, that pop-ins occur due to nucleation when indented in the relatively strain-free condition, because of a very low dislocation or source density (high dislocation spacing). When more pre-strain is introduced in Co, the number of tests which exhibit no discontinuous yield increases, but at a much lower rate as compared to the Ni. Hardness is a measure of the stress required to activate dislocation sources and/or move existing dislocations past obstacles. Co has a much higher inherent hardness than Ni and for this reason, it could be that the stress at pinned dislocations near the contact cannot reach the stress required to unlock them before the theoretical shear strength is reached at a location nearer the contact. The similar theoretical strength of all the Co samples, independent of the percent strain, indicates that pop-ins always occur due to nucleation. Any glissile dislocations in the stressed region are either not favorably oriented for the applied shear stress field or inherently more securely pinned than in Ni wherein there is a significant shift in average stress. It is also possible that few dislocations exist in regions away from grain boundaries. To answer this question, we can refer to existing TEM work indicating the structure change in Co and Ni with increasing strain.

Landau et al. studied dislocation boundaries in Ni at various levels of strain between 7% and 65%.[31] They found that the grain size of Ni is not influenced much by straining, but dislocation cells with equiaxed shape are formed, and that the cell size decreases with additional strain. The cell boundaries also become sharper. Both microstructural changes result from the dislocation density in the cells increasing.

Wu et al. used surface mechanical attrition treatments to induce strain in the surface of pure cobalt samples.[23] By examining the material at various

distances from the surface, it was possible to determine microstructural changes at different strain conditions. At low levels of strain (consistent with those in this work), they found that {10–11} deformation twinning was the main mode of deformation. In the interiors of grains, high-density (0001) basal stacking faults were observed. In either case, one would expect these defects to lower the onset of plasticity.

The increasing density of stacking faults observed in the work of Wu et al. suggests that the behavior of our Co is due primarily to securely pinned dislocations rather than a dislocation starvation mechanism. The limited number of slip systems and their orientations is then responsible for the fact that pop-ins either occur by nucleation or not at all. The existing dislocations can only move on certain planes, which are not favorable given the stress field around the indenter contact. This is consistent with the previously mentioned findings of Catoor et al., that dislocation nucleation is the mechanism of pop-in in Mg.[17] However, a critical feature of this current study is that the nucleation dominated behavior is prevalent in HCP materials. Their prior work emphasized the crucial nature of testing ‘perfect’ material to isolate nucleation; by sampling random orientations in the current work, we suggest the importance of nucleation in contact mechanics of HCP metals is broader than the narrowly defined conditions expressed in [17] and their conclusions are likely applicable to other HCP metals. The behavior is different for Ni, with the number and stress of the pop-ins being reduced by higher dislocation mobility and density as dislocation cell walls begin to thicken and occupy the interior of grains. It is likely that the stress at the actual avalanche site is the same, but is calculated as lower when the dislocation is nearer the point of contact.

**Conclusions** These experiments have demonstrated that pop-in can be a result of both homogeneous dislocation nucleation and the activation of pinned glissile dislocations originally in a locked condition under a suitably large shear stress. This behavior and model by extension can be used to understand the behavior of indentation load-depth curves that display many pop-ins, ‘jerky flow’ or staircase yield. In this case, it is presumed that the strength of the locks further from the indentation is in some instances stronger than in others or a more favorably oriented slip system reaches an activation stress. Yield during contact in Co (and we suspect other HCP systems) is more dominated by nucleation events even in the presence of pre-strain.

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