The transition from elastic to plastic deformation in crystalline metals shares history dependence and scale-invariant avalanche signature with other nonequilibrium systems under external loading such as colloidal suspensions. These other systems exhibit transitions with clear analogies to work hardening and yield stress, with many typically undergoing purely elastic behavior only after "training" through repeated cyclic loading; studies in these other systems show a power-law scaling of the hysteresis loop extent and of the training time as the peak load approaches a so-called reversible-to-irreversible transition (RIT). We discover here that deformation of small crystals shares these key characteristics: yielding and hysteresis in uniaxial compression experiments of single-crystalline Cu nano- and micropillars decay under repeated cyclic loading. The amplitude and decay time of the yield precursor avalanches diverge as the peak stress approaches failure stress for each pillar, with a power-law scaling virtually equivalent to RITs in other nonequilibrium systems.

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reloading from irreversible plasticity. The plastic strain that occurs below the previous maximum stress is the yield-precursor strain.

As the occurrence of avalanches upon reloading is stochastic in small-scale crystals, we apply two types of stress-strain reconstruction to average all the reloading curves as a measure of the ensemble precursor deviation from the “peak stress” yielding. Figure 1(c) demonstrates the in-series and in-parallel reconstruction using the reloading process marked in Fig. 1(b). We first shift the origin of each reloading process such that the stress is zeroed at the previous maximum stress and the strain is zeroed at the beginning. We interpolate and average the reloading response $\sigma_r$ (in parallel) or stress $\epsilon_r$ (in series), along the monotonically increasing strain $\epsilon_0$ (in parallel) or stress $\sigma_0$ (in series). Figure 1(d) shows the reconstruction results obtained from displacement-controlled tests on seven identically prepared pillars for each size of micropillars. We have subtracted the elastic strain to emphasize the plastic precursor behavior (see Supplemental Material, Sec. S2, for details of the reconstruction procedure [27]).

In the experiments presented here, larger precursor strain is prevalently observed in smaller pillars. However, we observe that the larger pillars that are monotonically loaded under displacement control generally produce shorter avalanche strains [37,38] and are less frequently spontaneously unloaded by the instrument compared with the smaller pillars. The emergent effect of system size on precursor avalanche behavior, where “system size” refers to the overall pillar volume, might arise from the variable unloading conditions. We conduct load-controlled (LC) compression experiments with several prescribed unload-reload cycles interrupting the quasistatic compression to investigate the size effect. The maximum stress increases 5 MPa per cycle, which equals to a quasistatic ramping rate of $\sim$1.4 MPa/s. Figure 1(e) shows such unload-reload stress-strain response of representative 500 nm and 3 $\mu$m diameter Cu pillars, and Fig. 1(f) compares their reconstructed yield-precursor stress-strain response. The types of precursor avalanches that we observe during the deformation of small micropillars that extend over $\sim$10$^{-4}$ strains at precursor stresses that are $\sim$5% (20 MPa) lower than the previous maximum stress ($\sim$400 MPa) would pose significant corrections to Hookean elastic behavior if they persisted to macroscopic systems.

We numerically evaluate the energy dissipation per volume reduced by precursor avalanches in comparison with the conventional plastic behavior, the precursor dissipation, from an integral over the reconstructed stress-strain hysteresis, $U = - \int \sigma_r d\epsilon_r$, indicated by the shaded area in Fig. 1(f) for 3 $\mu$m diameter samples. We observe larger precursor dissipation of $\sim$60 kPa in the smaller 500 nm diameter pillars than the $\sim$4 kPa in the larger 3 $\mu$m diameter samples, which suggests that the precursor avalanches may disappear in macroscopic samples. This is different from finite-size effect in statistically averaged distributions, where individual avalanches are hard to resolve in bulk or in high-symmetry crystals [39]. Since we measure the ensemble hysteresis, which is in nature a sum of the dissipation, small avalanches below the resolution of the instrument will still be properly incorporated. Perhaps this explains why precursor avalanches have not been thoroughly examined in existing literature.

We conduct LC cyclic training experiments to study how the precursor hysteresis changes under repeated loading to
the same maximum stress, analogous to experiments on other nonequilibrium systems [7,8]. We choose 3 \( \mu \)m diameter single crystalline Cu pillars as the primary experimental system because it is sufficiently large amongst the “small-scale” counterparts to exhibit failure under quasistatic loading as well as relatively deterministic precursor avalanche behavior. Figure 2(a) shows the estimated true stress-strain data from one representative training experiment on the left along with the scanning electron microscope (SEM) images of a typical pillar pre- and postcompression on the right. The failure stress, \( \sigma_c \), or the stress beyond which the samples are no longer able to support additional applied load, is defined as the global maximum stress at ~390 MPa. Above this stress, the sample continually deforms plastically at a constant stress [40]. In the representative experiment, we prescribe five cyclic stress steps with maximum engineering stress from 228 MPa (~0.57\( \sigma_c \)) to 452 MPa (~1.15\( \sigma_c \)) at equal intervals of 56 MPa (~0.14\( \sigma_c \)). In each stress step, we apply 100 unload-reload cycles, during which the sample is loaded to the same maximum stress and unloaded to a minimum of 56 MPa to maintain contact between the compression tip and the sample. We investigate the yield precursor dissipation evolution over all cycles at each stress step. Figure 2(b) shows the second, fifth, and eighth cycles of drift-corrected data (see Supplemental Material, Sec. S4, for details [27]) cycled to ~320 MPa in Fig. 2(a), with precursor dissipation indicated by the shaded areas.

We apply the multistep cyclic load function spanning the stress range 0.5~1.0\( \sigma_c \) to 24 identically prepared samples. It is reasonable to assume that for a cycle at a specific maximum stress, the intrinsic precursor dissipation behavior is equivalent across all samples within statistical variation. Figure 3(a) shows the average and standard error of the precursor dissipation as a function of cycle number for increasing maximum stress. These plots unambiguously demonstrate the training phenomenon: the precursor hysteresis decays with cycling. Increasing the maximum stress triggers new precursor avalanches and new training cycles. Below the catastrophic failure stress \( \sigma_c \), the precursor dissipation virtually vanishes. Post the failure stress, the hysteretic dissipation continues beyond the prescribed 100 stress cycles, which indicates that the training is incomplete.

We characterize the decay of precursor dissipation, \( U_f \), versus the number of cycles, \( n \), using a fitting function \( U_f(n) \) [8],

\[
U_f(n) = (U_0 - U_\infty)e^{-n/\tau}n^{-\delta} + U_\infty,
\]

where \( U_\infty = U_f(n \to \infty) \) is the estimated steady-state dissipation. \( U_0 \) is the initial dissipation. The power-law decay of \( U_f \) hints at the fluctuation behavior near the critical point. This analysis reveals that the catastrophic failure stress \( \sigma_c \), in these experiments can be associated with the RIT critical stress. This association is corroborated by the nonzero limiting dissipation \( U_\infty \) for a maximum stress amplitude of \( \sigma_{\text{max}} \geq \sigma_c \). We approximate the long-term decay at the step at \( \sigma_{\text{max}} \sim \sigma_c \) as critical behavior and fit the precursor dissipation \( U(n) \) using the simple power-law function, \( U_f(n) = U_f(n; \tau \to \infty, U_\infty \to 0) = U_0n^{-\delta} \), and estimate the exponent \( \delta \) be 0.68. A separate fit for \( \delta \) at different maximum stresses gives an average exponent with standard deviation fluctuation \( \delta = 0.70 \pm 0.18 \). We apply the fitted power-law exponent \( \delta = 0.70 \) to determine \( \tau \) for the remaining stress steps. Additional fitting details are provided in the Supplemental Material, Sec. S5.

We find, unlike granular systems [41] but like the colloidal systems, that the dislocation avalanches mostly disappear during the unloading branch (and hence, at the reversibility transition, also on the loading branch). This observed behavior could simply reflect a typical dislocation pinning stress large compared to the failure stress. Modifying the mean-field model, which studies hysteresis in a granular system [42], we incorporate the exponential decay rate \( \tau \), and predict \( \delta = 1 \) (see the Supplemental Material, Sec. S6, for details [27]). The
Figs. 3(b) and 3(c) from the expected power-law divergence timescale of the system. The deviation of the final point in (b) and the corresponding points in (c) from the expected power-law divergence is probably due to our method for estimating the steady-state value (see the Supplemental Material, Sec. S7[27]). (c) A direct comparison of dislocation RIT behavior gleaned from the Cu micropillar compression experiments with that reported for a colloidal particle system in a sheared suspension [8], which provides evidence for a divergence of necessary cycle time $\tau$ to reach a reversible state, close to the critical failure stress $\sigma_c$.

The theoretical exponent, however, is far outside our statistical errors for the collective fit, but within the fluctuations for $\delta$ fit separately for different $\sigma_{\text{max}}$.

Figure 3(b) shows that the decay time constant of precursor hysteresis $\tau$ increases with maximum stress $\sigma_{\text{max}}$. The inset shows that the estimated steady-state $U_{\infty}$ is close to zero below the critical stress $\sigma_c$ and abruptly increases to $\sim 2-4$ kPa when $\sigma_{\text{max}}$ reaches $\sigma_c$. The deviation of the final point in (b) and the corresponding points in (c) from the expected power-law divergence is probably due to our method for estimating the steady-state value (see the Supplemental Material, Sec. S7[27]). (c) A direct comparison of dislocation RIT behavior gleaned from the Cu micropillar compression experiments with that reported for a colloidal particle system in a sheared suspension [8], which provides evidence for a divergence of necessary cycle time $\tau$ to reach a reversible state, close to the critical failure stress $\sigma_c$.

At higher peak stresses, the dislocation rearrangements in one cycle may trigger a cascade of further avalanches in subsequent cycles. In small-scale crystalline plasticity, the RIT corresponds to the stress at which additional cycling continues to plastically deform the system with no additional applied forces, which corresponds to the failure stress. We can speculate about the relation between the critical behavior of the precursor avalanches observed here and the power-law distribution of dislocation avalanches observed in nano- and micropillars under monotonic loading. The precursor avalanches at an RIT usually diverge in size only near the failure stress. Plasticity avalanches under monotonic loading are debated to be associated with a “stress-tuned criticality” [44,45] or a jamming transition [46], either of which exhibit a power-law scaling with a cutoff in the avalanche size distribution that diverges only as the stress approaches the “failure stress”—precisely as one would expect for the approach to an RIT.

In this Letter, we bring attention to the overlooked signature of yield precursor avalanches in nanomechanical experiments. We show that the amount of dissipation due to yield precursor avalanches decays over repeated stress training cycles. We find that the characteristic decay time increases with the applied maximum stress. The apparent divergence of the time constant at a maximum stress near the quasistatic failure stress indicates that the flow transition of the dislocation system is fundamentally an RIT. This is the first time that this effect has been shown in any crystalline material experimentally. Prior studies have only focused on amorphous materials and attributed RIT behavior to many ordered or short-range ordered material systems. Our work extends the universality of RIT to include crystals. The training and RIT behavior has potential connections with cyclic fatigue and the transition from rapid hardening to saturation hardening at bulk scales, e.g., shakedown and ratcheting [47], wherein the dislocations microstructures evolve from mutual trapped bundles into distant loop patches [17,18]. However, we demonstrate that size effect is not negligible, which corroborates with the lack of prior research on training effects and precursor avalanches at large scales. Nanomechanical experiments have been intensively explored as a powerful methodology to study the fundamentals of crystal deformation, but the understanding of the dislocation plasticity in terms of RIT was hitherto lacking because people had only focused on quasistatic experiments which are not efficient in resolving history-dependent dissipative features of materials. Our Letter may inspire novel approaches to study plasticity, fatigue, and catastrophic failure in crystalline materials governed by complex dislocation dynamics.

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[40] The perfect failure mode is not observed in the LC compression experiments because a dynamic increase in engineering stress is required to compensate for the plastic deformation induced contact area change of the pillar loaded in (111) high-symmetry direction to maintain the true applied stress above the critical stress level.
[43] The true stress estimates are not reliable post the plastic collapse, as the true stress-strain conversion (see Supplemental Material, Sec. S1 [27]) holds only in the conditions that (i) the lattice orientation does not change significantly and that (ii) deformation remains approximately homogeneous on the pillar scale.