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Spatiotemporal slip dynamics during deformation of gold micro-crystals

G. Sparks [a], P. Sudharshan Phani [b], U. Hangen [c], R. Maaß [a, *]

[a] Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA
[b] Nanomechanics Inc., 105 Meco Ln, Oak Ridge, TN 37830, USA
[c] Hysitron, Inc., Dennewartstrasse 25, 52068 Aachen, Germany

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ABSTRACT

Intermittent plastic deformation in micro-crystals was resolved in both space and time, yielding velocity profiles that peak in a range from approximately 1 μm/s to 100 μm/s. The peak velocities exhibit a broad, close to scale-free distribution, with a scaling regime at high velocities that is compatible with a cubic decay. Slow slip dynamics in the μm/s regime show an approximately flat distribution. An apparent power-law scaling between peak slip-velocity and event size was also observed. The spatiotemporal dynamics of the dislocation-mediated plastic intermittency is discussed in terms of lateral and vertical slip-step growth velocities, where the vertical growth velocity is ~4 orders of magnitude slower than the lateral dislocation-group velocity, providing a rationale for the measured slow dynamics. In order to validate the experimental results, the response of the used nanoindenter is evaluated for the time during the plastic instability. Fracture tests on Si were conducted to determine the upper bound in dynamic response of the device. Finally, in-situ electrical contact measurements complete the suite of tests that unequivocally demonstrate that the used nanoindentation platform is capable of tracing the spatio-temporal slip dynamics during slip of small-scale crystals.

1. Introduction

Plastic deformation of crystals is mediated via the movement of dislocations. During application of an increasing stress, the underlying dislocation structure is known to respond intermittently, where the majority of the dislocation network remains immobile but a certain group of dislocations quickly rearranges to relax local stresses. These sudden rearrangements can be recorded as an acoustic emission pulse [1], or directly as discrete stress-strain data from ultra-high-resolution mechanical experiments on macroscopic samples [2,3]. Recent progress in nano- and micro-mechanical testing reinvigorated interest in this intermittent plastic flow because the stress-strain curves of nano- or micron-sized single-crystals exhibit a vast number of sudden strain bursts, which are associated with these moving groups of dislocations, also called dislocation avalanches [4]. Commonly, the micro-compression technique using a nanoindenter platform is employed to record intermittent stress-strain data (see Fig. 1) that can be analyzed with respect to the slip-size statistics. It has been shown that the event size probability distributions obtained from deforming micro- and nano-crystals exhibit some degree of scale-free power-law behavior with the scaling exponents typically around 1.5 [5–10], and recent discussions focus on the factors influencing the value of this scaling exponent [11–14]. In general, the experimental findings are well in agreement with theoretical predications [15] and dislocation dynamics (DD) modeling [16–18].

In our recent work, we explored the micro-compression method further, using data sampling rates of several kHz in order to probe the slip dynamics; that is, the transient phase between the onset and end of the slip event [19,20]. This allowed the assessment of the apparent slip-velocities during slip events in addition to the usual slip-size data. The obtained slip-size magnitudes displayed the expected probability distribution representing scale-free scaling, and furthermore the obtained slip-velocities showed a probability distribution scaling compatible with the predictions of 3D dislocation dynamics (DD) simulations [21] and theoretical analysis of avalanches near the depinning transition [18]. In addition to these results reflecting known properties of dislocation-mediated plasticity, no apparent scaling between crystallographic slip-velocity

* Corresponding author.
E-mail address: rmaass@illinois.edu (R. Maaß).

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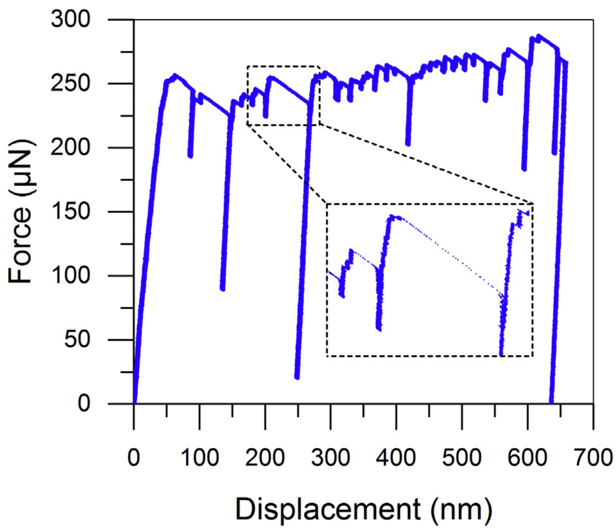

Fig. 1. Representative load versus displacement curve for micro-column compression testing. The zoomed inset shows the behavior of the load-displacement curve during strain bursts. Substantial elastic unloading after a strain burst is due to the controller reducing the applied force to counteract the sudden increase in displacement beyond the specified displacement rate in displacement control.

and applied stress could be established across a range of more than 400 MPa [19]. This was rationalized on the basis of microplasticity in which fluctuations of an internal stress field form a static component, and dynamic interactions with other dislocations dominated the dynamic response of the avalanche. In view of the classical result of a strongly stress dependent dislocation velocity [22–24], v_d, empirically known as $v_d = v_0(\tau/\tau_0)^m$, with the materials parameters v_d, τ_0 and $m \gg 1$, it is expected that the dislocation velocity at the size-affected stresses will be very high. Given that a slip event (dislocation avalanche) is mediated by dislocation movement, it could be expected that the slip-velocity should also be very fast, and questions arise as to what extent the data truly represents a dislocation-mediated process. This is indeed a valid question, which urges for additional investigations on the validity of slip-kinetics measurements done with a nanoindenter (here a Hysitron TriboIndenter). In fact, if indeed the measured slip-kinetics are entirely unrelated to the underlying plasticity, then it is not clear why the distribution of slip-sizes or slip-velocities scale as expected from theoretical predications and as shown in DD-modeling of intermittent plasticity.

The main problem lies in the unclear link between the measured slip-velocity and the underlying dislocation velocity [20]. Clearly, the slip event (dislocation avalanche) represents a collective motion, where the slip-size magnitude can be understood as the lower bound of the net Burgers vector content of the instability. Taking a typical slip-size of 2 nm in a 2 μm sized crystal, and the Burgers vector for Au, this results in at least 10 dislocations that are deposited onto the surface during slip. The flow stress level of such a micro-crystal is ca. 95 MPa [20]. Using the classical picture of linearly stress dependent dislocation velocities for the high-stress/high-velocity regime, $v_d = \frac{b\tau}{B}$ (where B is a material constant representing viscous drag on the dislocation) [25] gives $v_d > 100$ m/s for $\tau = 95$ MPa, which leads to the conclusion that the approximate slip-duration for the consecutive motion of 10 dislocations (constantly emitting source) would be ca. 200 ns or less. This is clearly incompatible with earlier results that showed that slip-durations are of the order of some hundred μs to several ms when sampled at 7 kHz [19,20]. With this large discrepancy between the expected underlying dislocation velocity and the measured slip-velocity (when large enough sampling rates are used), one would need to conclude that during the majority of the measured slip-duration no actual dislocation activity is taking place, but that the slipping crystal primarily is at rest or in a quiescent avalanche state. The simple estimate above would yield an inactive phase of about 1 - (200 ns)/(2 ms) = 99.99%; an inconsistency that was already articulated during the 1960s, when velocities of individual or grouped dislocations were measured [26]. Either the slip event is indeed fast, but broken up in a series of alternating active or inactive stages that are beyond the experimental resolution, or the number of dislocations is substantially underestimated, or finally it may be that the device is not able to trace the slip event properly. In the latter case, the indentation tip would be expected to lose contact with the slipping crystal, which would need to continue its dynamic phase without any applied stress, and one would have to question whether the slip-sizes derived from such measurements are reliable. Elastic relaxation of the column could allow for contact to be maintained even if the tip cannot move at sufficient speed to follow the plastic deformation of the column, but given the observed applied forces and pillar stiffness, the elastic relaxation (length increase) can be at most ~13 nm, which is insufficient to maintain contact for the larger event sizes. Thus, the situation is unsatisfactory, and the aim of the following work is to shed further light onto the underlying collective dislocation dynamics during a slip event measured in a small-scale deformation experiment with a nanoindenter, and also to support the view that it can be excluded that the device is losing contact with the slipping crystal.

We will first follow the approach of Hay et al. [27] that modeled the nanoindenter as an underdamped linear harmonic oscillator, which allows us to estimate the indenter dynamics. The modeled dynamics of the device can then be compared to the measured values during slip events, the results of which imply that the force from the sample on the indenter tip does not fall to zero at any point during the slip events. Secondly, the peak velocities of the indenter tip measured during events in various materials are discussed and compared with those from a known loss-of-contact scenario that was evaluated by tracing the indenter response after fast fracture of silicon micro-samples. Thirdly, in-situ electrical contact measurements are done on micron sized crystals, with the aim at exploring if a loss of current can be recorded during the slip phase. The summarized findings of these efforts clearly indicate that contact is not lost between the slipping crystal and the nanoindenter.

Following this, further analysis is done on the characteristics of the slip events themselves. The force-displacement behavior of the sample during slip events is compared to that during elastic loading and unloading, demonstrating that the effective sample stiffness during slip events is lower than the axial quasi-static stiffness, and thus suggesting that the samples are indeed plastically deforming during the displacement jump representing the slip event.

Inspection of the velocity profiles in some events suggests that they may be multiple temporally overlapping slip events, and an algorithm designed to separate or discard such events results in the elimination of the two-branch structure seen in earlier work by removing all of the "slow" events [19,20]. This carries the implication that such events may simply be multiple slip events that could not be well separated during initial data extraction and analysis. Inspection of the peak velocities during slip events in FCC Au shows a power-law correlation with event size, which is compared with that found for BCC Mo [5] and theoretical values from mean-field theory [18]. Finally, we propose that the large discrepancy between collective dislocation velocities and the here measured slip-velocities finds its origin in the large ratio between laterally and vertically growing slip-steps — a view compatible with classical findings on slip-line cinematography [22].