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Supporting Information

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Independence of Slip Velocities on Applied Stress in Small Crystals

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Independence of slip velocities on applied stress in small-scale crystals

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Introduction

The main article presents data obtained during discrete slip events of deforming gold microand nano-crystals. During a slip event, both the used device as well as the sample are elastically relaxing in a very abrupt way, and due to the sudden deviation of the true displacement rate, the feedback control of the used device (Hysitron Triboscope) responds. This response leads to the retraction of the compression platen relative to the sample, and gives the obtained force-displacement data its characteristic shape with the linear elastic force (stress) drop segments evident in Fig.1 in the main article, or outlined in Ref. [S1]. Whilst this linear elastic load drop is purely a machine effect occurring after the slip event of interest, it is not obvious to know if the contact between the compression platen and the crystal is maintained during the slip event. Several arguments against a loss of contact are presented in Ref. [S2], but a direct verification is still lacking. In the next section, some aspects of the sample-device coupling are discussed, and experimental evidence is provided that demonstrates that the sample remains in contact during the slip event.

Sample-Device Coupling

Besides the simplicity of this technique, there are a few instrumental factors that need to be considered. In the present work all experiments were conducted with displacement-rate control by using a device that is inherently force-rate controlled. We note that the feedback loop frequency will affect the absolute magnitude of the measured slip magnitude. A lower

feedback frequency allows for larger deviations from the pre-described displacement rate, which in turn allows larger displacement jumps prior to adjustment. In this work a Hysitron Triboscope with a Performec control is used. This device has a feedback frequency of 78 kHz and is expected to correct any deviation from the programmed displacement rate 156 times faster than a device operating at for example 500 Hz. As a result, the displacement jump will be shorter. In the limit, an infinitely high feedback frequency would result in undetectable slip size magnitude, similarly as an infinitely stiff displacement driven machine. Another factor that may influence the absolute magnitude of the displacement jump is the compliance of sample-machine assembly, since it is known that the observed displacement jump Δd can be a function of both the axial sample compliance C_S (ca. 44 nm/mN for a 2 μ m crystal), and the machine compliance C_M (ca. 5000 nm/mN) according to: $\Delta d = \Delta F (C_s + C_M)$, where ΔF is the associated force drop during the event. Such a relationship is based on treating the machine and sample as two springs in series, but is found to overestimate the magnitude of the actual event size Δd by a factor of two, since the feedback loop leads to an apparent stiffening of the transducer spring. Thus, the total displacement measured during a slip event in any mechanically coupled experiment with a finite spring stiffness will always be proportional to the total compliance of the assembly. It is thus important to bear in mind that the absolute values can vary depending on the testing system.

Sample-Platen Contact During Slip

In order to directly probe if the compression platen remains in contact with the slipping sample, we have conducted in-situ nano-electrical-resistance measurements with the nanoECR add-on of the used Triboscope nanoindenter. Further specifics of the set-up can be found in Ref. *[S3]*. Care was taken to sample the current under application of a constant

voltage of 500 μ V with the same data sampling rate as was used for the displacement and force signal (7 kHz). During displacement-controlled (DC) compression the change in current was monitored, and displacement-time close-up view (similar to that shown in Fig.2 in the main manuscript) is displayed in Fig.S1a in combination with the current signal. Figure.S1b is a close-up view of the force-displacement-current data for the corresponding displacementtime-current data of the same displacement jump as in Fig.S1a. The data acquired during testing displays a current value that oscillates around a mean value of about 1.5 µA within the displacement range of the displacement jump. Whilst there is considerable variation of the current signal along the quasi-static phase of the compression experiment, there are no notable deviations of the current during the discrete event (displacement jump). In particular this can be seen in the force-displacement-current trace, where the current signal shows some oscillations but no permanent decay to a zero current. At the end of the displacement jump, the feedback loop of the DC-mode responds, and the tip is retracted to correct for deviations between the actual and predefined loading rate. This is indicated with the arrow in Fig.S1b. During the elastic unload after the slip event, the current signal reduces linearly, which is seen by the dense number of data points at the end of the slip event. Based on the data in Fig.S1 it is therefore concluded that, within the resolution of the experiments, there is no detectable loss of contact between sample and compression-tip during a crystallographic slip event. We further conducted similar experiments on metallic glass columns that display displacement jumps of axial velocities of ca. 60 µm/s which are well above the maximum velocities encountered in the present crystalline work. Similar values for metallic glasses have been published elsewhere [S4]. We thus can also conclude that the forward velocity of the indenter tip has not reached its limit for the data displayed in the main article. Continued research will investigate the mechanical coupling between crystals and tip during plastic instabilities of the type studied here.



Figure S1: displacement-time-current trace (a) and force-displacement-current trace (b) for the same slip event recorded during displacement controlled testing. The axial velocity was determined to be ~5.9 μ m/s in the displacement range between 35-55 nm. The arrow in b) indicates the elastic unload due to the acting feedback loop.

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References

[S1]	O. L. Warren, S. A. Downs, T. J. Wyrobek, Z. Metallk. 2004, 95, 287.
[S2]	R. Maass, P. M. Derlet, J. R. Greer, Scripta Materialia 2013, 69, 586.
[S3]	S. Ruffell, J. E. Bradby, J. S. Williams, O. L. Warren, J. Mater. Res. 2007, 22,
	578.
[S4]	Z. Y. Liu, Y. Yang, C. T. Liu, Acta Materialia 2013, 61, 5928.