



## Regular article

## Shear-induced softening of nanocrystalline metal interfaces at cryogenic temperatures



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## ABSTRACT

We demonstrate inverse Hall-Petch behavior (softening) in pure copper sliding contacts at cryogenic temperatures. By kinetically limiting grain growth, it is possible to generate a quasi-stable ultra-nanocrystalline surface layer with reduced strength. In situ electrical contact resistance measurements were used to determine grain size evolution at the interface, in agreement with reports of softening in highly nanotwinned copper. We also show evidence of a direct correlation between surface grain size and friction coefficient, validating a model linking friction in pure metals and the transition from dislocation mediated plasticity to grain boundary sliding.

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Among the unique properties of nanocrystalline metals, their remarkably low friction coefficients remain poorly understood [1–3]. Defying wisdom that clean, bare metals invariably produce high friction [4,5] (coefficients > 1), recent reports [1–3] have shown highly pure, coarse-grained metals (e.g. gold, copper) can exhibit low friction (coefficients < 0.5) at low stresses in inert environments. This has been linked to formation of shear-generated ultra-nanocrystalline surface films [1,2,6,7]. We show that it is possible to drive self-mated, high purity copper to low friction at relatively high stresses by subjecting the contact to near cryogenic temperatures. By thermally limiting stress-driven grain growth [8,9], we enabled the formation of a reduced shear strength ultra-nanocrystalline layer [10] (grains ≤ 10 nm) exhibiting inverse Hall-Petch behavior, with grain boundary sliding as the dominant deformation mechanism [11]. The low friction is shown to be entirely attributable to this mechanism. We present a quantitative correlation between friction coefficient and surface grain size, which is in agreement with reports of grain-size dependent strength reductions for pure ultra-nanocrystalline metals [12–15]. Correlating friction to grain size establishes a new approach for materials design and investigation of stress-induced destabilization of nanocrystalline metals and alloys, an area at the forefront of materials science research [16,17].

Contact during sliding for rough, pure metals is fully plastic [18], and contact area is proportional to hardness. The friction coefficient can be represented as the ratio of interface strength ( $\tau$ ) and hardness,

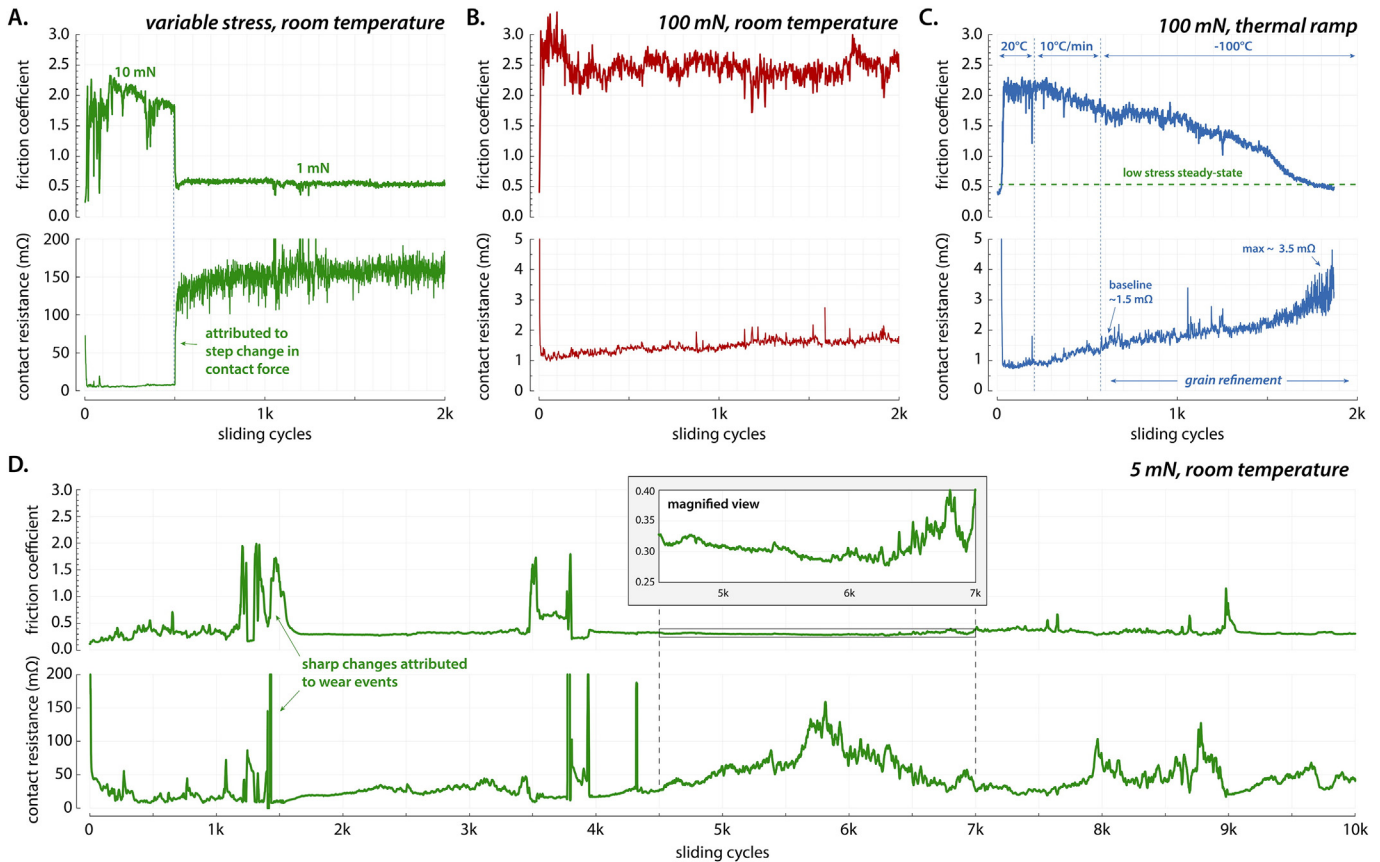
$$\mu \approx \frac{\tau}{H} \quad (1)$$

We investigated sliding contact between high purity copper (99.999%) with initial hardness,  $H \sim 1.5$  GPa (see supplemental). For self-mated pure metal contacts, in the absence of oxides and hydrocarbons, friction is effectively a measure of the strength required to shear cohesive metal junctions [19]. We show that it is possible to derive a direct and insightful correlation between friction coefficient and interface grain size, and validate this claim by sliding high purity copper against copper in an inert gas environment. We show that friction is influenced by varying contact stress and temperature, down to  $-100$  °C (Fig. 1). The evolution of friction coefficient and electrical contact resistance were measured concurrently [20] and then used to indirectly determine the evolution of surface grain size (method details in supplemental).

We correlate the reduction in friction from  $\mu \sim 2$  to  $\mu < 0.5$  with softening of the interface due to grain refinement into a regime where grain boundary sliding dominates [10,21]. We used sphere-on-flat contacts (1.6 mm radius), in linear reciprocating sliding at 1 mm/s, and contact forces in the range 1 to 100 mN. We first show that at room temperature it is possible to induce a transition in friction behavior from high-to-low

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**Fig. 1.** Friction coefficient and electrical contact resistance data for self-mated copper contacts in inert gas showing bimodal steady-state friction regimes achieved by changes to both stress and temperature. In (A) we show an immediate transition between the two regimes at room temperature achieved by sufficiently lowering contact force, in (B) we show baseline data for the high friction regime at 100 mN, and in (C) we show a thermally-driven transition between the friction regimes at 100 mN. In (D) we show an extended experiment at 5 mN, just below the room-temperature critical stress bound for copper [1], and an inset of friction coefficient and contact resistance behavior during a transient period showing inverse correlation attributed to microstructure evolution.

(Fig. 1A) by reducing contact stress, similar to what was shown for pure Au contacts in an earlier publication [1]. In both steady-state and ramped temperature experiments, during the first 10 cycles we observe run-in to friction coefficient,  $\mu > 1$  (Fig. 1A–C); during this run-in period, electrical contact resistance is consistent with the removal of native oxides. A fully-plastic contact stress solution (see supplemental) can be used to estimate contact area [22] which is more accurate when analyzing rough surfaces, contrasting with the elastic (Hertz) solution used in an earlier publication for smooth surfaces [1].

To make a prediction of friction based on microstructure requires a definition for the interface strength as a function of surface grain size. The Hall-Petch model has limited applicability at grain sizes smaller than about 10 nm [17,23–25], and it has recently been shown by various groups [2,3,6,7] for a variety of FCC metals in inert environments that a reduction in friction from high ( $\mu > 1$ ) to remarkably low ( $\mu < 0.5$ ) is linked to the formation of a thin, ultra-nanocrystalline (UNC, grain sizes  $< 10$  nm) surface film. This has also been observed in both initially coarse grained pure metals [1] and near-amorphous (initial grain size  $\sim 5$  nm) Ni-W alloys [7,26]. Whereas we previously showed that grain size is linked to friction and evolves toward a stress-dependent steady-state value [1], here we validate the prediction that steady-state friction is also a function of temperature. Specifically, the steady-state grain size is defined by the competing rates of grain refinement through deformation [19] and stress-driven coarsening [9]. By reducing temperature and thereby limiting grain growth kinetics [8], we postulated [1] that it should be possible to achieve a transition to low friction at contact stresses that otherwise generate immediate high friction, as shown in Fig. 1. We demonstrate that not only is it possible to achieve a stable, low friction UNC layer by reducing contact stress at 20 °C (Fig. 1A) – even for

prolonged periods of sliding (Fig. 1D) – but also by maintaining high surface stresses and reducing temperature. We now turn to an analysis of the thermal ramp data and show that it is possible to extract fundamental materials properties and demonstrate inverse Hall-Petch behavior through macro-scale sliding experiments.

To link friction coefficient to surface microstructure evolution, we must first understand the grain size-dependent strength of metals in the nanocrystalline regime. Much progress has been made to establish constitutive models for the grain size dependent strength of nanocrystalline metals [11,27–29]. By combining these with contact mechanics models, we show that it is possible to predict the shear strength of a sliding metal junction – and by extension, the friction coefficient – based on materials properties and contact parameters. Our experiments show that reducing the temperature of pure copper contacts to  $-100$  °C sufficiently slows grain growth kinetics and enables the formation of an UNC surface layer with greatly reduced shear strength. We propose a direct link between the observed reduction in friction coefficient at cryogenic temperatures and a transition from dislocation mediated plasticity to grain boundary sliding. Details of this transition are clear in Fig. 2, where we show the friction and contact resistance as a function of wear track position and sliding cycles. We validate the hypothesis that suppression of stress-driven grain growth enables grain refinement to dominate over grain growth at contact stresses that otherwise generate high friction [1] (Fig. 1A & B) using scanning transmission electron microscopy (STEM). In Fig. 3, we compare the UNC interface from the experiments at cryogenic temperatures to the microstructure from a room temperature experiment with persistent high friction. In Fig. 3A we see that the discrete UNC layer, where sliding is accommodated via shear deformation, does not correspond to the exposed surface on the TEM specimen;

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