

Effect of Amorphous-Crystalline Phase Transition on Superlubric Sliding

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Structural superlubricity describes the state of greatly reduced friction between incommensurate atomically flat surfaces. Theory predicts that, in the superlubric state, the remaining friction sensitively depends on the exact structural configuration. In particular the friction of amorphous and crystalline structures for, otherwise, identical interfaces should be markedly different. Here, we measure friction of antimony nanoparticles on graphite as a function of temperature between 300 and 750 K. We observe a characteristic change of friction when passing the amorphous-crystalline phase transition above 420 K, which shows irreversibility upon cooling. The friction data is modeled with a combination of an area scaling law and a Prandtl-Tomlinson type temperature activation. We find that the characteristic scaling factor γ , which is a fingerprint of the structural state of the interface, is reduced by 20% when passing the phase transition. This validates the concept that structural superlubricity is determined by the effectiveness of atomic force canceling processes.

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The tribological phenomenon of structural superlubricity is characterized by ultralow friction between atomically flat and incommensurate surfaces that can be achieved through collective force cancellations during relative motion. Understanding the mechanisms and, especially, limitations that determine the effectiveness of force cancellations is not only of fundamental interest in nanotribology, but also relevant to design superlubric interfaces for technical applications.

Consequently, a growing number of studies have analyzed superlubricity and the influence of different key factors both in theory and by nanoscale experiments [1–5]. In particular, interface contamination [6–12], interface size, shape, and orientation [13–19], interface relaxations and (local) commensurability [20–26], as well as the role of crystalline structure [13,14,27] have been considered as important factors for the efficiency of superlubric force cancellations. Concerning the crystalline structure, current models predict that well-ordered surfaces allow for more comprehensive force cancellations as compared to amorphous interfaces, where the statistical positioning of the interface atoms limits the potential for superlubricity [28]. Despite this fundamental character of this paradigm of superlubricity, a direct experimental verification is still lacking.

Previous experiments have linked amorphous and crystalline interfaces with different contact area scaling relations for gold and antimony particles [13]. However, open questions remained about the influence of the chemical nature of these two materials. A direct transition of the

interface structure without modifying the chemical nature of the surfaces, would give direct verification of the atomic origins of structural superlubricity.

Here, we are analyzing Sb-nanoparticles on HOPG for temperatures between 300 and 750 K. Sb nanoparticles on HOPG are known to exhibit an irreversible and controllable transition from amorphous to crystalline structure induced in the antimony particles by heating to temperatures of approximately 130°–170 °C [29–32]. Indeed, we find a clear fingerprint of this transition in our atomic force microscopy (AFM) nanomanipulation experiments. Comparison of friction levels at 300 K before and after heating allows us to quantify the change in superlubricity, which are compared to different concepts of interface changes. Ultimately, the observed friction changes of approximately 50% suggest a scenario where the superlubric friction reduction is limited by remaining structural irregularities such as the particle border and grain boundaries at a polycrystalline interface.

Friction between the nanoparticles and the substrate was measured using a nanomanipulation approach, based on detecting the torsional signal from the cantilever when the AFM tip pushes the particle across the substrate [3,6,11,13,27]. All nanoparticle-samples were prepared by thermal deposition of antimony onto highly oriented pyrolytic graphite. The HOPG was freshly cleaved in air before transfer to the ultrahigh vacuum (UHV) chamber with a base pressure of 5×10^{-10} mbar. To remove adsorbates, the HOPG was heated to 200 °C for 2 h.

Evaporation of antimony then took place at deposition temperatures of 400 °C for 5–10 min (see [27]). This procedure resulted in well-defined nanoparticles with atomically flat interfaces between particles and substrate [6,33,34]. Under these preparation conditions the Sb nanoparticles are amorphous, which is further validated by their size and compact shape [13,35].

After preparation and without breaking the vacuum, the samples were transferred to the UHV-AFM (VT-AFM, Omicron Nanotechnology GmbH). All nanomanipulation experiments have been performed as a three-step process: (i) To avoid scanning-induced movement of nanoparticles, the surface was first imaged in noncontact mode and a nanoparticle for manipulation was chosen [Fig. 1(a), top]. (ii) The AFM tip was placed next to the nanoparticle and the system was switched to contact mode. The particle was then pushed perpendicular to the long cantilever axis, while the lateral force signal F_L was recorded [Fig. 1(b)]. (iii) The particle displacement was verified by a control image using noncontact mode [Fig. 1(a), bottom].

The effective particle friction was calculated from the difference of the pushing force during sliding and the lateral force experienced by the AFM tip on the HOPG before reaching the nanoparticle [Fig. 1(b)]. Lateral force calibration was based on the method by Bilas *et al.* [36]. We used

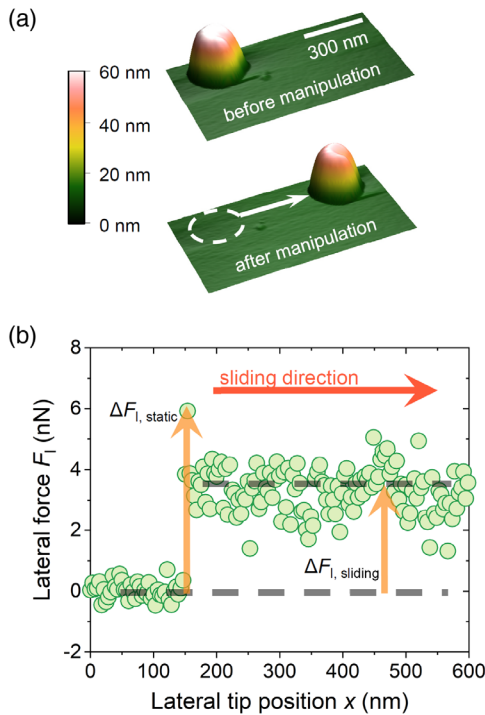


FIG. 1. Lateral manipulation of an antimony nanoparticle on graphite. (a) Top: AFM topography image before manipulation. Bottom: Topography image after manipulation. (b) Lateral force signal along the path of manipulation. The AFM tip reaches the nanoparticle at about $x = 150$ nm and the lateral force signal increases to $\Delta F_{L,static}$ before $\Delta F_{L,sliding}$ then corresponds to the sliding friction of the nanoparticle.

typical normal loads of $F_n = 75$ nN in the first set of experiments and 101 nN in the second, with a constant sliding velocity of 417 nm/s. The contact area A of each particle was determined from AFM topography images.

The first set of experiments was performed for a group of three nanoparticles of similar contact area and at temperatures between 300 and 750 K. Within this range, the temperature was progressively increased in steps of 50 K resulting in friction values for ten different temperatures. To ensure that the exact same particles were measured at each temperature, thermal drift during temperature changes was compensated based on continuous imaging of the relevant sample area.

Figure 2 displays a similar temperature dependence for all three particles, where three different regimes can be

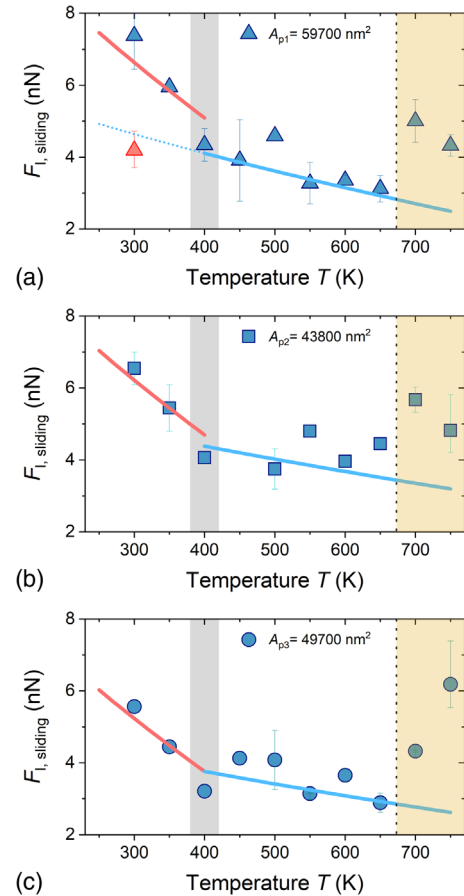


FIG. 2. (a)–(c) Temperature dependence of sliding friction measured for three different nanoparticles for continuously increasing temperatures between 300 and 750 K (blue symbols). The red data point in (a) was recorded after cooling particle 1 from 750 back to 300 K. For increasing temperatures below 700 K the temperature dependence can be divided into two regimes as indicated by the theoretical curves based on thermally activated stick slip (continuous lines). The transition corresponds to the typical temperature range for structural phase transitions of nanoscale antimony as indicated by the grey background color. In addition, a high temperature regime with increased friction emerges once the sample temperature exceeds 400 °C.

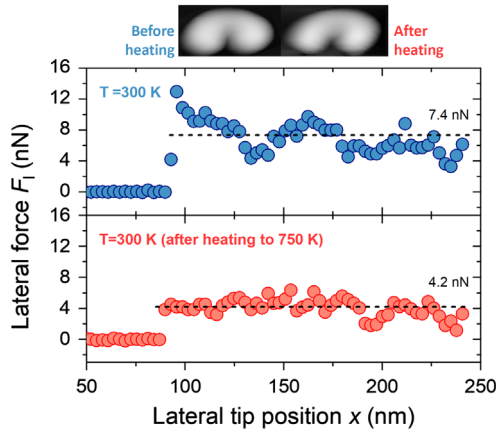


FIG. 3. Friction forces measured during manipulation of nanoparticle 1 [$A = 59\,700\text{ nm}^2$, Fig. 2(a)] at 300 K before and after heating. A significant reduction in friction by a factor of 1.76 is found. In addition, the AFM-topography images (top) verify that particle size and shape have not changed during exposure to high temperatures. In particular, no significant evaporation of antimony occurred.

distinguished. Over the whole temperature range, sliding friction varies by a factor of 3.3 for the first nanoparticle, 2.6 for the second nanoparticle, and 2.8 for the third nanoparticle. The red data point in Fig. 2(a) refers to the sliding friction of particle 1 ($A = 59\,700\text{ nm}^2$) when it cooled back to 300 K after exposure to high temperatures. This documents a friction reduction by a factor of 1.76. Figure 3 shows the friction traces recorded during manipulation of this nanoparticle at 300 K before and after the high temperature treatment. This result suggests that a temperature induced irreversible change of the interface has occurred. While such changes can be ruled out for HOPG, phase transitions of antimony are well-known occurrences in nanoscale systems [29–31,37–45]. The crystalline structure depends on the system size, and transitions from amorphous to crystalline structure can occur once a critical film thickness or particle size is exceeded [34,42,46,47]. At the same time, temperature also plays an important role for phase transitions in nanoscale antimony structures [37,40], and sufficient temperature can induce a transition from amorphous to crystalline state in thin films [41,43], nanowires [29,30], or nanoparticles [31,32] with typical transition temperatures around 400–420 K.

To verify the occurrence of a temperature induced phase transition for our nanoparticles, we prepared two new samples, one of which was heated to 513 K for 5 h. Both samples have then been analyzed by SEM EBSD (scanning electron microscopy electron backscatter diffraction, see, also, the Supplemental Material [48]). The SEM images [Figs. 4(a) and 4(b)] illustrate the general topography, while Figs. 4(c) and 4(d) show standard EBSD IQ (image quality) maps. In these maps, the brightness of each pixel represents a relative measure for the diffraction

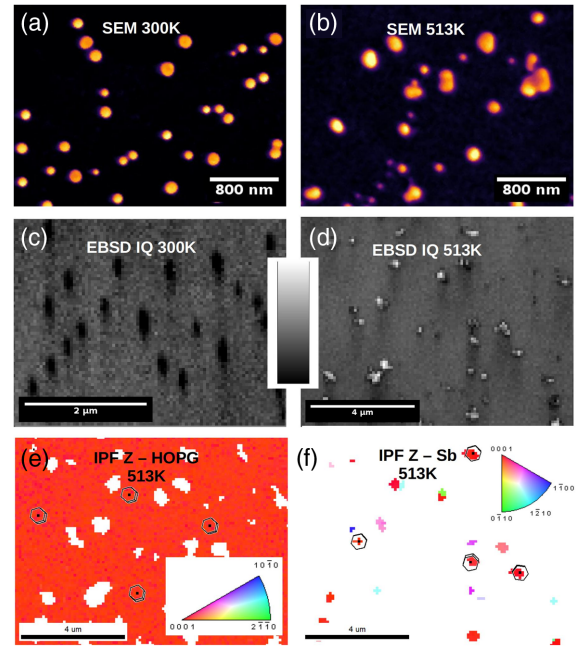


FIG. 4. SEM and EBSD analysis of Sb particles on HOPG. (a), (b) SEM surface morphology of the unheated sample (a) and the sample heated to 513 K (b). (c),(d) SEM EBSD IQ maps of the unheated sample (c) and the sample heated to 513 K (d). (e),(f) EBSD inverse pole figure maps (Z out of plane) for HOPG (e) and the heated Sb particles (f). The orientation of hexagonal unit cells is shown for selected points.

pattern quality that is calculated from local EBSD patterns as average of the peaks in the Hough Transform space [49,50]. Comparing the crystalline HOPG substrate with the nanoparticles, we find that the intensity of the HOPG far exceeds that of the unheated particles, which appear as dark spots in Fig. 4(c) due to their amorphous structure. On contrary, the heated nanoparticles [Fig. 4(d)] appear as bright spots since their crystalline structure and high mass number result in high IQ. Additionally, the detailed diffraction patterns (see Supplemental Material [48]) also corroborate the structural difference.

A more detailed analysis of the heated nanoparticles was done by EBSD inverse pole figures (IPFs) (Z out of plane) studies [51], which show crystallographic orientations by color-coded maps [Figs. 4(e) and 4(f)]. The IPF map on HOPG confirms the (0001) orientation [Fig. 4(e)]. Figure 4(f) then shows the orientation distribution for different nanoparticles. In the majority of cases, we find (0001) orientation, which means that sliding mainly occurs at the (0001)Sb// (0001)HOPG interface (cf. Supplemental Material [48]).

Based on the EBSD analysis, we assign the change in friction in our Sb sliding experiments to a thermally triggered phase transition. The observed transition temperature at about 400 K (Fig. 2), is consistent with the above findings. In addition, the absolute friction values for the amorphous state are well in line with previous results for Sb

particles on HOPG in the superlubric regime, where a link between particle friction and diffusion energy barrier was possible [13].

Next, we model the experimental friction F_L as a thermally activated Prandtl-Tomlinson process [52–55]

$$F_L(T) = F_c - \left[\beta k_B T \ln \left(\frac{v_c}{v} \right) \right]^{2/3} \quad (1)$$

with a critical velocity v_c of

$$v_c = (2f_0\beta k_b T)/(3c_{\text{eff}}\sqrt{F_c}). \quad (2)$$

Here, f_0 is the attempt frequency, c_{eff} the effective contact stiffness and the factor β is a measure for the curvature of the potential energy corrugation.

The key parameter for describing the different superlubric states for crystalline and amorphous particles is the effective energy barrier. For an ideal sinusoidal energy landscape the effective energy barrier E_0 is linked to the critical force F_c by $\beta_{\text{sin}} = (3\pi F_c)/(2\sqrt{2}a)$ and $F_c = \pi E_0/a$ with a as the lattice constant of the substrate. Since superlubric force canceling for an amorphous interface is less effective than for a crystalline interface, we expect a lower energy barrier for the crystalline state.

A number of experimental works using friction force microscopy (FFM) [56] indicated a significant deviation between β and β_{sin} . Factors of approximately 2–6 have been found for the ratio $\delta = \beta/\beta_{\text{sin}}$ during velocity and temperature dependent FFM on mica or HOPG [54,55]. Therefore, we treat β as an additional fit parameter. We find that the phase transition is not only reflected in the critical forces F_c but also in β (Table I). This emphasizes the different mechanisms of force cancellation in the two phases. The critical forces F_c between the temperature regimes differ by a factor of 2. Extrapolation of the crystalline theoretical curve for particle 1 also yields a good approximation for the friction value measured at 300 K after heating [Fig. 2(a)]. Finally, we look at the experimental observation of increasing friction for the highest temperatures ($T \geq 700$ K), which might be tentatively linked to increased interface

TABLE I. Parameters to calculate the theoretical curves in Fig. 2. The table shows only varying parameters. Additionally, $a = 2.5$ Å and $f_0/c_{\text{eff}} = 1.4 \times 10^{11}$ m/Ns were used for all temperatures and particles.

Particle	T range	F_c/N	δ
1	Low	13.5×10^{-9}	5
1	High	7×10^{-9}	1.5
2	Low	13×10^{-9}	5
2	High	6.5×10^{-9}	1
3	Low	11.8×10^{-9}	5
3	High	5.8×10^{-9}	1

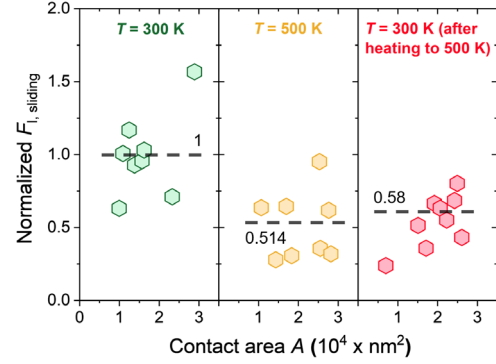


FIG. 5. Irreversible friction changes for Sb nanoparticles after heating to 500 K. The friction values were obtained at three different temperatures from nanomanipulation experiments for three different groups of nanoparticles of similar size. Left: friction measurements at 300 K, Middle: friction measurements after heating the sample to 500 K, Right: friction measurements at 300 K after the sample was cooled down again. After heating, the nanoparticle friction is reduced by a factor of approximately 2.

relaxations when measuring at temperatures suitable for sublimation and approaching the solid-liquid transition at 630 °C.

To further validate the irreversible phase transition, we performed manipulation experiments on a new sample for three different random groups of nanoparticles of similar size. First, the friction of a set of eight nanoparticles was analyzed at 300 K prior to any heating of the sample (Fig. 5, left). After that, the sample was heated to 500 K, and a second set of eight nanoparticles was analyzed (Fig. 5, middle). Finally, sliding friction was measured for a third set of nine nanoparticles after the sample cooled back down to 300 K (Fig. 5, right).

The results confirm the observations from Fig. 2(a). At low temperature, friction is highest and is reduced roughly by a factor of 2 when heating to 500 K. This friction change is then found to be irreversible, since the same level of friction is measured after cooling back to room temperature. In addition, the absolute friction values also compare well to Fig. 2.

Next, we want to correlate the relative friction changes in Fig. 5 with the superlubricity model predictions from “scaling law” [11,13,15,28]. In this concept, the effective energy barrier for sliding ΔE is linked to the number of interface atoms N by a power law as $\Delta E = \Delta E_0 \times N^\gamma$, where ΔE_0 represents the single-atom energy barrier. As a first approximation, we assume that the sliding friction values are directly proportional to the energy barrier ΔE and can be described as $F_{\text{sliding}} = F_0 \times N^\gamma$ with F_0 as the single atom friction.

In both cases, γ is a characteristic scaling factor that sensitively depends on parameters like crystalline structure, shape, or relative orientation between substrate and slider [13,14,17]. For amorphous sliders, the scaling factor

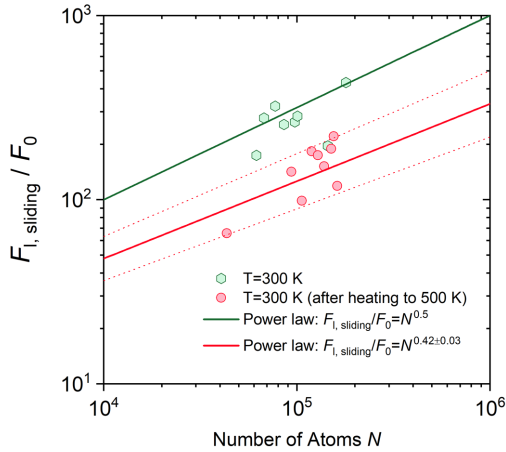


FIG. 6. Normalized sliding friction as a function of the number of antimony atoms at the interface measured for 2 ensembles of nanoparticles at 300 K before (green hexagons) and after (red circles) heating the sample to 500 K. The straight lines represent the scaling of friction vs the atom number for the two cases. The friction data has been normalized by the single atom friction of antimony on HOPG which, in turn, was derived from fitting $F_{l,sliding} = F_0 N^{0.5}$ to the dataset analyzed before heating. From the dotted lines, the error of γ' can be estimated.

is $\gamma = 0.5$ independent of shape and relative orientation. Thus, we have fitted the data of Fig. 5 obtained at 300 K before heating with a fixed scaling factor $\gamma = 0.5$, yielding the single atom friction value F_0 . This allows us to normalize the data of Fig. 5 obtained at 300 K before and after heating by F_0 . A fit to the normalized friction data after the heating cycle results in a scaling factor of $\gamma' = 0.42 \pm 0.03$, accounting for the temperature induced phase transition (Fig. 6). (Please note, that the fitting result is mostly determined by the absolute changes in friction.) By further considering a potential influence of β on the friction vs area scaling, an even reduced value of $\gamma' = 0.371$ can be estimated (see Supplemental Material [48]).

The scaling analysis validates the idea that superlubric force cancellations become more efficient after the phase transition from amorphous to crystalline. The round shape of the particles allows a theoretical minimum of $\gamma = 0.25$ [13,17]. The observed reduction of $\gamma = 0.5$ to $\gamma' \approx 0.37$ – 0.42 here might appear moderate but already compares well to previous experimental results, where a value of $\gamma = 0.33$ was found for crystalline gold nanoparticles sliding on HOPG. In our case, disordered grain boundaries and random grain orientations as well as defects might counteract the force cancellation process, which can increase friction and, thus, γ .

In summary, we find a characteristic and irreversible change in the sliding friction behavior of Sb nanoparticles when undergoing a structural phase transition from amorphous to crystalline. These results highlight the general importance of the atomic structure at the sliding interface in the superlubric regime. Experiments on idealized model

systems like this help our understanding of how forces of each atom at the sliding interface act synergistically to enhance or cancel their contributions. At the same time, applications of superlubricity are rapidly evolving [57–60], requiring a thorough knowledge of the underlying mechanisms.

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- information on the friction vs. contact area scaling and the EBSD results.
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