Unexpected temperature and velocity dependencies of atomic-scale stick-slip friction

I. Barel and M. Urbakh^{*}

School of Chemistry, Tel Aviv University, 69978 Tel Aviv, Israel

L. Jansen

Center for Nanotechnology (CeNTech), University of Münster, Münster, Germany

A. Schirmeisen

Institute of Applied Physics (IAP), Justus-Liebig University Giessen, Giessen, Germany (Received 23 June 2011; published 14 September 2011)

We report experiments of atomic stick-slip friction on NaCl that were performed over a wide range of surface temperatures and scanning velocities. As in previous experiments, we found a nonmonotonic relation between the time-averaged friction and temperature. In contrast to the previous works, here atomic-scale stick-slip friction was resolved for all measured temperatures and velocities. We further introduce a model that explicitly includes a periodic structure of a multi-asperity contact, and we demonstrate that the simulations reproduce the experimental observations. The presented results show that analysis of mean friction force only is not enough for an unambiguous understanding of the friction mechanisms, and measurements of force traces at different temperatures and velocities provide important additional information.

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I. INTRODUCTION

The field of nanotribology evolved around attempts to understand the relationship between frictional forces and microscopic properties of systems. In particular, studies of the temperature dependence of friction provide unique information on the kinetics of rupture and formation of atomic contacts at sliding interfaces, and thus they are of crucial importance for understanding microscopic mechanisms of friction. Experiments of nanoscopic friction under controlled vacuum conditions over a wide temperature range have revealed a nonmonotonic enhancement of dry nanoscale friction at cryogenic temperatures for different material classes.^{1–3} This temperature dependence has been accompanied by an unexpected velocity dependence of friction, F(V), as seen by a decrease of friction with V for low temperatures and increase of friction with V for higher temperatures. These observations are inconsistent with the common paradigm according to which the interfacial friction should decrease with temperature provided no other surface or material parameters are altered by the temperature changes. $^{4-10}$

Two theoretical models have been recently proposed in order to explain a peak-like enhancement of mean friction force at cryogenic temperatures: (i) the Prandtl-Tomlinson model,^{11,12} and (ii) the mechano-kinetic model, which describes friction through a thermally activated rupture and formation of molecular contacts.^{2,3} The Prandtl-Tomlinson model predicts that the friction force may exhibit a peak in the interval of temperatures (*T*) that corresponds to a transition from a multiple-slip regime of motion at low *T* to the single-slip regime at higher *T*. Under this condition, an interplay between the reduction of the slip length with *T* and thermally activated jumps over potential barriers may lead to a nonmonotonic temperature dependence of friction. In the mechano-kinetic model, the friction peak emerges from two competing processes acting at the sliding interface: the

thermally activated formation as well as rupture of an ensemble of atomic contacts.

Previous experiments,¹⁻³ which revealed a friction peak at cryogenic temperatures, focused on the temperature and velocity dependencies of the time-averaged friction forces and could not measure force traces with atomic resolution. This was achieved later on a graphite sample,¹³ where the explicit temperature and velocity dependencies of atomic friction were shown to be consistent with the conventional model of thermally activated atomic jumps of the tip (Prandtl-Tomlinson model). In particular, for atomic friction on graphite, the previously reported nonmonotonic friction-temperature relation with the peak-like enhancement was absent. This apparent contradiction may be due to the limited temperature range investigated on graphite, or there may be conceptual differences when measuring atomic stick-slip friction on various surface materials. We would like to note that a friction peak was also absent on a soft metallic Au(111) surface.³

In order to understand these discrepancies, here we investigated the temperature and velocity dependence of atomic stick-slip friction on the ionic crystal NaCl(001), which is another widely used material for atomic friction studies. As in previous experiments,¹⁻³ we found a nonmonotonic relation between friction and temperature. However, in this case, atomic stick-slip was resolved at all measured temperatures and velocities. We extended the mechano-kinetic model to explicitly include a periodic structure of a multi-asperity contact, which allowed us to reproduce the main experimental observations: (i) the peak-like enhancement of the mean friction force as a function of temperature, (ii) unexpected velocity dependence of friction, and (ii) variation of the stickslip traces with temperature. The presented results show that an analysis of time-averaged mean friction force is not enough for an unambiguous understanding of the friction mechanism, and that measurements of force traces at different temperatures and velocities provide important additional information.

II. EXPERIMENTAL DETAILS

Nanoscale friction experiments on the ionic crystal NaCl(001) were performed under clean ultrahigh-vacuum (UHV) conditions with an atomic force microscope as a function of sample temperature at cryogenic temperatures from 105 K to room temperature. Furthermore, we measured the pull-off force at all temperatures, allowing us to monitor strong changes of the tip-sample adhesion, which would indicate significant alterations of the tip geometry. The sample surface was a NaCl(001) single crystal, which was freshly cleaved in air and then quickly inserted into the vacuum chamber. Subsequently, the sample was annealed in UHV at around 520 K for about 1 hour. This procedure resulted in atomically flat and clean terraces on which the friction was measured, avoiding any step edges.

The friction force experiments were performed with a commercial UHV atomic force microscope (VT-AFM, Omicron Nanotechnology, Germany, Taunusstein). All measurements were done at a base pressure of $P = 5 \times 10^{-10}$ mbar. As force sensors, we used single crystalline, rectangular silicon cantilevers (LFMR, Nanosensors, Germany), with a nominal tip radius of 20 nm. We applied the procedure described in Refs. 14 and 15 to determine the normal ($c_n = 0.05$ N/m) and torsional ($c_t = 6.0$ N/m) spring constants of the cantilevers. The lateral forces were calibrated after the method described in Ref. 16. The lateral force maps were measured on a small area of $5 \times 5 \text{ nm}^2$ of the NaCl(001) surface, where each map consisted of 300 scan lines. The average friction was calculated from the area encompassed by the individual friction loops (i.e., the forward and backward scan) divided by the scan size. After the measurements were completed at one temperature, the tip-sample adhesion was determined by measuring the pull-off forces. Experiments were performed in two consecutive runs with the same cantilever, run 1 at temperatures 105 K, 151 K, 197 K, 246 K, and 295 K, and run 2 at 127 K, 176 K, 223 K, and 274 K.

The results of the mean friction as a function of temperature are presented in Fig. 1(a), which reveals a nonmonotonic relation between friction and temperature. The adhesion forces in Fig. 1(b) do not vary much and can be regarded as roughly constant throughout the temperature range. In these experiments, atomic resolution was achieved at all measured temperatures and velocities, and Fig. 2 shows some examples of the obtained force traces. The traces exhibit three characteristic features: (i) For all measured temperatures and velocities, stick-slip behavior was observed; (ii) the slip length was almost unaffected by temperature; and (iii) there was a nonmonotonic behavior of the minimal, $\langle F_{min} \rangle$, and maximal, $\langle F_{max} \rangle$, values of the instantaneous spring forces with temperature.

III. THE MODEL

Both the Prandtl-Tomlinson and mechano-kinetic models,^{2,3,11} which explain the peak-like enhancement of the mean friction force at cryogenic temperatures, certainly fail to reproduce the characteristic features of force traces observed on NaCl. Within the Prandtl-Tomlinson model, the peak in temperature dependence of friction is accompanied by a significant change in the slip length that is inconsistent with the experimental results presented in Fig. 2. The mechano-kinetic model predicted that the stick-slip oscillations would cease to exist for temperatures above the peak temperature and the motion would become chaotic, which are also in conflict with the experimental observations.

In this paper, we demonstrate that the observed variations of the mean friction force and force traces with temperature can be explained within a modified mechano-kinetic model. The mechano-kinetic model introduced in Refs. 2 and 3 describes an interaction between the AFM tip and the underlying surface through an array of contacts representing the molecular bonds. The contacts (*N* of them) are modeled by elastic springs, each of which has a force constant κ and a rest length $l^{(0)}$. As long



FIG. 1. (Color online) (a) The mean friction force as a function of sample temperature for two consecutive runs. Atomic stick-slip was observed for all data points, and the scan speed was 208 nm/s. (b) The corresponding adhesion forces determined from the pull-off force of the cantilever. (c) Friction as a function of sliding speed for run 1 at the five experimental temperatures.



FIG. 2. (Color online) Forward and backward lateral traces on NaCl showing atomic stick-slip behavior at all sample temperatures. The sliding speed was constant at 208 nm/s for all traces.

as a contact is intact (unbroken), it is stretched in the lateral direction with the velocity equal to the velocity of the tip, \dot{X} , while a ruptured contact relaxes rapidly to its equilibrium state. Therefore, from the interface, the tip experiences the force $F_c = -\sum_{i}^{N_s} f_i$, where $f_i = \kappa (l_i - l^{(0)})$, and l_i (t) is the time-dependent spring length. The tip with mass M and center-of-mass coordinate X is pulled along the surface with a constant velocity V through a linear spring of spring constant, K. The motion of the driven tip is described by the following equation:

$$M\ddot{X} + \eta \dot{X} - F_c + K(X - Vt) = 0$$
(1)

where η is a damping coefficient responsible for the dissipation of the tip kinetic energy to phonons and other degrees of freedom that are not considered explicitly here. The instantaneous lateral spring force, which is the main observable property in friction experiments, reads as F = -K(X - Vt), and its time average is equal to the friction force $\langle F \rangle$.

The rupture of contacts is considered as a thermally assisted escape from a bound state over an activation barrier $\Delta E_{\text{off}}(f_i)$, which is force dependent and diminishes as the applied

elastic force, f_i , increases (the contact is stretched).^{17,18} The formation of individual contacts is characterized by a further energy barrier ΔE_{on} , which is needed to initiate the contact. Contact formation as well as contact rupturing processes are thermally activated, and the interplay between them may lead to a complex dependence of friction on tip velocity and sample temperature.

The rates of rupture and formation of contacts, $k_{\text{off}}(f_i)$, can be approximated by the following equations:^{17,18}

$$k_{\text{off}}(f_i) = \omega_{\text{off}}^0 \exp[-\Delta E_{\text{off}}(1 - f_i/f_c)^{3/2}/k_B T] \qquad (2)$$
$$k_{\text{on}} = \omega_{\text{on}}^0 \exp[-\Delta E_{\text{on}}/k_B T] \qquad (3)$$

where ω_{off}^0 and ΔE_{off} are the characteristic attempt frequency and the height of potential barrier for unbinding in the absence of the external force, respectively; f_c is the critical rupture force at which the potential barrier, $\Delta E_{\text{off}}(f_i)$, vanishes; and ω_{on}^0 and ΔE_{on} are the attempt frequency and the barrier height for reattachment. The critical force can be estimated as $f_c = \Delta E_{\text{off}}/R_c$, where R_c is a characteristic length scale (width) of the binding potential. We note that a rate of spontaneous (thermal) unbinding is given by $k_{\text{off}}^0 = k_{\text{off}}(f_i = 0) = \omega_{\text{off}}^0 \exp(-\Delta E_{\text{off}}/k_BT)$.

Notice that contact stretching is renewed every time the contact reforms and, thus, the lengths, l_i , and elastic forces, f_i , are different for different contacts.

As it has been discussed in Refs. 2 and 3, the dynamics of friction in this model are determined by four characteristic frequencies (rates): the rate of spontaneous detachment of contacts, $k_{off}^0 = k_{off}(f_i = 0)$, rate of contact formation, k_{on} , rate of forced unbinding, KV/f_c , and a characteristic rate of the pulling force relaxation, $\omega_m = \max(K/\eta, \sqrt{K/M})$. The rates k_{off}^0 and k_{on} are defined by inherent microscopic properties of the system, and they depend on temperature, while the rates KV/f_c and ω_m are temperature independent and influenced by the pulling velocity V and mechanical parameters of the experimental setup, M and K. Under the experimental conditions we have, $KV/f_c < \omega_m$.

IV. RESULTS AND DISCUSSION

Previous simulations^{2,3} have been performed under the assumption that atomic-scale contacts are randomly distributed over the contact area, which reflects the amorphous nature of the tip. In this configuration, the stick-slip regime of motion can be observed only when the reattachment rate is lower than the rate of force relaxation, $k_{on} \leq \omega_m$. As the rate k_{on} approaches ω_m , the spring force does not have time to relax during a slip, the stick-slip motion becomes more and more irregular, and for $k_{on} > \omega_m$, the force traces become completely erratic.^{2,3}

For realistic values of system parameters $(M, K, \eta, \Delta E_{on}, \omega_{on}^0)$, the condition $k_{on} \leq \omega_m$ ceases to be true for temperatures above 100–200 K, and as a result, previous simulations of the mechano-kinetic model^{2,3} predicted a chaotic motion above the peak temperature for the mean friction force. This conclusion is certainly inconsistent with the results of AFM measurements on NaCl, where the regular stick-slip oscillations have also been observed for room temperature.^{9,19}



FIG. 3. Schematic sketch of the multi-atom contact between periodic substrate and tip surfaces.

In order to describe the results of friction measurements on NaCl, a distribution of the binding sites at the tip and substrate surfaces should be taken properly into account. In scanning probe microscopy experiments on NaCl, it is known that nanocrystals of NaCl are frequently transferred to the scanning tip (e.g., Refs. 20 and 21), thus providing a multiatom contact between two NaCl single crystals. In order to mimic this configuration, we assume that binding sites are periodically distributed along the substrate and tip surfaces. In the general case, the effective lattice spacing of tip and surface atoms could be different, since the NaCl cluster on the tip is usually not in perfect alignment with the sample. However, the experimental observations of regular stick-slip motion indicate that the NaCl cluster on the tip is in perfect alignment with the surface. This configuration is schematically shown in Fig. 3. Microscopic contacts can be formed between the binding sites at the tip and substrate, and the maximum possible number of contacts is given by a number of sites at the tip, N. It should be noted that the contacts can be created not only between the tip and substrate sites, which are located one above the other, but also between more distant sites. However, formation of contacts between distant sites requires an additional elongation of the contact, Δx , and as a result, the energy barrier for this process grows with increasing distance between the sites. The energy barrier for formation of a contact between the sites separated by the lateral distance Δx can be calculated as

$$\Delta E_{\rm on} = \Delta E_{\rm on}^0 + \kappa (\Delta x)^2 / 2 \tag{4}$$

where ΔE_{on}^0 is the energy barrier for formation of a contact between the sites located one above the other. Correspondingly, the rate of reattachment is given by the equation

$$k_{\rm on} = \omega_{\rm on}^0 \exp\left[-\left(\Delta E_{\rm on}^0 + \kappa (\Delta x)^2/2\right)/k_BT\right]$$
(5)

Figure 4 shows the results of calculations of the temperature dependence of the time-averaged spring force, $\langle F \rangle$, and time series of the spring force, which were obtained for the case of periodically distributed binding sites. We found the same friction enhancement peak at low temperatures as in previous simulations for amorphous surfaces;^{2,3} however, the force traces exhibit a very different temperature dependence. Contrary to the earlier calculations,^{2,3} which exhibit stick-slip only for temperatures below the peak temperature, here we found regular stick-slip oscillations in the whole range of temperatures under investigation In this case, the slip length is given by the period of the binding site lattice, and it is almost temperature independent.

The model demonstrates that the peak in the temperature dependence of the mean friction force, $\langle F \rangle$, results from an interplay between two competing process that occur with increase of *T*: (i) a rise in the number of contacts reattached during a stick interval of motion, n_{max}^r , and (ii)

a reduction in detachment (rupture) force, F_{max} . Taking into account that n_{max}^r increases exponentially with T, $(n_{\text{max}}^r \propto \omega_{\text{on}} \exp(-\Delta E_{\text{on}}/k_B T))$, while F_{max} decreases according to a power low with $T(\langle F_{\text{max}} \rangle - F_c \propto -T^{2/3} \ln^{2/3} (BT/V)^{4,5})$, we find that the friction force peaks at $T = T_{\text{max}}$, which is only slightly below the temperature for which all N contacts are reattached during a stick interval. The peak temperature can be estimated from the following equation

$$N/k_{\rm on} \approx \langle F_{\rm max} \rangle / KV$$
 (6)

This consideration demonstrates that the peak in the friction force shifts to higher temperatures with an increase in scanning velocity, and this is supported by the results of numerical simulations in Fig. 4. A direct fingerprint of the peak shift with velocity is found in Fig. 4, which shows the calculated velocity dependence of friction at constant temperatures, T = 100 K, T = 200 K, and T = 300 K. At temperatures above the peak temperature, friction increases with scan speed, whereas at a temperature below the peak, friction decreases with velocity. The experiments show the same characteristic fingerprint in the friction-velocity curves in Fig. 1(c).

Equation (6) shows that the peak temperature decreases with a reduction of the barrier height for the reattachment, $T_{\text{max}} = \Delta E_{\text{on}} / \ln[\langle F_{\text{max}} \rangle \omega_{\text{on}} / (NKV)]$, and for very small values of ΔE_{on} , the peak may lie below the experimentally accessible range of temperatures. In particular, for $\Delta E_{\text{on}} =$ 0, the theory predicts monotonic decrease of friction with temperature, which is in agreement with the conclusions of the Prandtl-Tomlinson model.^{4,5,9} Thus, the monotonic decrease of friction with temperature that was found in experiments on graphite and Ag(111)^{3,13} may result from small values of the barrier heights for the reattachment of surface contacts in these systems. However, a definite answer to this question requires first-principles calculations of potential energy surfaces for the tip-surface junctions.

The proposed model includes seven unknown parameters: $\Delta E_{\text{off}}, \Delta E_{\text{on}}, \omega_{\text{off}}^0, \omega_{\text{on}}^0, \kappa, N, \eta$; however, only three of them, $\Delta E_{\rm off}$, $\Delta E_{\rm on}$, and N, were varied in order to get a qualitative agreement with the experimental data. For all other parameters, we used the values estimated in previous simulations of AFM experiments (see, for instance, a discussion in Ref. 22). The most important of the parameters are the barrier heights, $\Delta E_{\rm off}$, $\Delta E_{\rm on}$, which define the position of the peak in the temperature dependence of friction and the rate of decrease of $\langle F \rangle$ at high temperatures. The simulations show that the overall behaviors presented in Fig. 4 are robust and hold for a wide range of parameters. It should be noted that here we did not try to quantitatively fit the simulation results to experimental ones. At this stage of research, this is meaningless, because the model includes a large number of unknown parameters. A quantitative analysis of experimental data requires firstprinciples calculations of potential energy surfaces for the tip-surface junctions, which will allow us to determine the unknown parameters. The first steps in this direction have been already taken recently.²³

As shown here, accounting for a periodic structure of the binding sites, we can explain most of the features observed in friction experiments on NaCl: (i) temperature and velocity dependencies of the mean friction force; (ii) existence of



FIG. 4. (Color online) Results of the simulation for the multi-atom contact between periodic substrate and tip surfaces. (a) Representative parts of time series of the friction forces at different temperatures, scan velocity = 500 nm/s. (b) Time-averaged friction as a function of temperature for different scan velocities. (c) Friction-velocity curves for the different temperatures showing characteristic negative and positive slopes. Parameter values used in simulations: $\omega_{off}^0 = 10^8 s^{-1}$, $\omega_{on}^0 = 6 \times 10^8 s^{-1}$, $\Delta E_{off} = 0.23 \text{ eV}$, $\Delta E_{on} = 0.14 \text{ eV}$, $\kappa = 0.5 \text{ N/m}$, $f_c = 0.25 \text{ nN}$, $\eta = 3.5 \times 10^{-5} \text{kg/s}$, K = 6 N/m, $M = 5 \times 10^{-11} \text{ kg}$, N = 7.

regular stick-slip oscillations for all measured temperatures and velocities; and (iii) independence of slip length and temperature. However, in this model, the minimal values of the instantaneous spring forces, $\langle F_{\min} \rangle$, decrease monotonically with temperature,²⁴ which is in conflict with experimentally observed nonmonotonic variation of $\langle F_{\min} \rangle$ with T (see Fig. 2). In order to describe this effect, an additional contribution to friction should be included that does not significantly affect the regular stick-slip dynamics but introduces a new temperaturedependent channel of energy dissipation. This can be achieved assuming that the tip interacts with the substrate through two types of contacts with distinct dissociation energies, ΔE_{off} : strong contacts between periodically distributed binding sites at the substrate and tip, and weak contacts for which spatial distribution is unimportant. Then, the force-induced detachment and reattachment of strong contacts lead to regular stick-slip oscillations, while the dynamics of weak contacts result in additional temperature-dependent dissipation that only slightly affects the stick-slip patterns. The described behavior is shown in Fig. 5, where we present the results of simulations of the

model including two types of contacts. The results demonstrate that the proposed model indeed enables us to describe all experimentally observed features of the mean friction force and force trace.

An important and still unresolved question is: What is the physical nature of the atomic instabilities described here in terms of detachment and reattachment of weak contacts? Similar to the mechanism of energy dissipation in atomic force microscopy,^{25,26} the instabilities can be attributed to reversible jumps of surface atoms, flips of surface fragments, or transitions between different tip structures, which are induced by the tip motion along the surface. These nonconservative processes result in a bistable potential energy surface for the tip-surface junction, where the barrier separating the potential minima is continuously changed during the tip motion, and rates of transitions between the minima can be described by Eqs. (2) and (3). An unambiguous understanding of the nature of the instabilities and an evaluation of the microscopic parameters in Eqs. (2) and (3) require first-principles calculations of potential energy surfaces for the tip-surface junctions.



FIG. 5. (Color online) Results of the simulation for two kinds of contacts between periodic substrate and tip surfaces. (a) Representative parts of time series of the friction forces at different temperatures, scan velocity = 500 nm/s. (b) Time-averaged friction as a function of temperature for different scan velocities. Parameter values used in simulations: $\eta = 3.5 \times 10^{-5}$ kg/s, K = 6 N/m, $M = 5 \times 10^{-11}$ kg, (i) strong contacts: $\omega_{off}^0 = 10^{10} \text{s}^{-1}$, $\omega_{on}^0 = 10^7 \text{s}^{-1}$, $\Delta E_{off} = 0.375$ eV, $\Delta E_{on} = 0.002$ eV, $\kappa = 0.5$ N/m, $f_c = 0.43$ nN, N = 1; (ii) weak contacts: $\omega_{off}^0 = 10^{10} \text{s}^{-1}$, $\omega_{on}^0 = 10^7 \text{s}^{-1}$, $\Delta E_{off} = 0.175$ eV, $\Delta E_{on} = 0.075$ eV, $\kappa = 0.5$ N/m, N = 15.

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*urbakh@post.tau.ac.il

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pulling the tip in the direction of motion. This effect is significant in the sliding state (where the formation of contacts occurs), and it leads to a decrease of the minimal values of the instantaneous spring forces, $\langle F_{\min} \rangle$, with temperature, which was found in simulations.

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