

Low-Temperature Wear of Silica Induced by Diamond Single Asperities

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Atomic force microscopy is used to analyze the temperature dependence of nanoscale wear occurring during reciprocal line scanning of a SiO₂ surface using diamond atomic force microscopy tips. Unexpectedly, our experiments show a wear rate that decreases with temperature. Thereby, the results cannot readily be reconciled with the common model of atomic scale wear, which assumes an abrasive process of thermally activated and shear-assisted atom-by-atom removal. Instead, shear-assisted tribochemical processes are taken into account. This is done based on a multibond model for single-asperity tribochemical wear as well as recent analysis of contact aging driven by bond formation for silica and diamond systems. We consider how thermally activated bond formation at different temperatures is met by temperature-dependent probabilities for bond rupture accompanied by wear. This allows us to rationalize the experimental results and link the temperature dependence of wear to the way an increasing number of interfacial bonds are ruptured at lower temperatures. Ultimately, our experiments thereby expand the emerging multibond model of single-asperity nanofriction and tribochemical wear to the temperature domain.

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Understanding the atomistic origins of nanoscale wear is currently a very active topic in nanotribology. Interest is not only spurred by the general aspiration to understand wear mechanisms on the atomic level, but is also motivated by the ongoing progress in the development of nano- and microelectric mechanical systems, where classical, macroscopic wear laws, like, e.g., Archard's law [1], are no longer applicable. Consequently, a continuously growing number of studies have been focusing on nanoscale wear. In particular, the sharp tip of the atomic force microscopy (AFM) cantilever can be considered as an ideal model system for a single asperity, i.e., the most elementary building block of realistic multiasperity contacts [2–4].

Nanoscale wear can be generated by repeated AFM tip “scratching” of the surface [5]. Extremely low wear rates can be achieved, giving access to fundamental mechanisms of wear, down to the removal of single atoms or clusters. Numerous aspects of nanoscale wear have so far been addressed including the influence of normal force [6–8], sliding velocity [9], and environmental conditions [7,10],

as well as reattachment and ripple formation [11–13], suppression of wear [14,15], or material-specific tribochemistry [8,16–19].

Tribochemical wear is especially intriguing, since it considers the complex behavior of interfacial bonds that are first formed and subsequently broken, often including transfer of atoms. Such processes can be crucial for interfaces formed between silica surfaces [20–24] or between silica and diamond [25–29], with considerable technological impact.

Tribochemical processes are characterized by the energy barriers for formation and breaking of bonds [8]. Experimental parameters like sliding velocity, normal force, and temperature allow one to probe the complex interface dynamics. So far, nanoscale wear experiments have mostly been focused on analyzing velocity and load dependence [8], while the influence of temperature remains largely unexplored.

However, the general importance of temperature for tribochemical processes has been demonstrated for nanoscale friction experiments within the so-called multibond model. Here, energy dissipation resulting from breaking newly formed bonds could explain a nonmonotonic temperature dependence of friction observed between Si AFM tips and Si(111) wafers [30–33]. The multibond scenario represented a significant further development of the thermally activated Prandtl-Tomlinson model, which assumes a periodic surface potential and predicts a monotonic decrease of friction with temperature [34,35]. In addition, tribochemical processes have been identified as main

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driving factors in nanoscale contact aging studies, where the hold-time dependence of static friction observed during slide-hold-slide experiments was linked to the gradual formation of interfacial bonds [20,36,37]. These studies were then also complemented by temperature variations, which allowed one to linearly tune the contact aging rate [24,38].

Still, temperature-dependent nanoscale wear experiments have so far been limited to systems like, e.g., NaCl and KBr, where interfacial bonds were not considered. Wear was sufficiently described by a thermally activated and shear-assisted process of atom-by-atom removal [6,39]. Correspondingly, the wear rate was found to increase monotonically with temperature [7,40].

Our experiments in this Letter show that the wear rate for silica surfaces in sliding contact with diamond AFM tips instead decreases with temperature in a range from 95 to 300 K. We explain this surprising behavior by considering shear-assisted bond formation, previously found to be responsible for an increased number of interfacial bonds at low temperatures [24,38]. This leads to the counterintuitive effect of an increased wear rate at low temperatures, even though the energy barrier for bond breaking is lower than for the removal of adjacent surface atoms. Lastly, these experiments highlight the wear process of SiO₂ itself, adding to previous studies of diamond wear due to formation of Si—C or Si—O—C bonds [27–29].

For the temperature-dependent wear analysis, we used an atomic force microscope under UHV conditions (Omicron VT-AFM) in contact mode operation. All wear experiments were done on a Si wafer with a thermally grown oxide layer of about 300 nm. Tips were commercially available AFM cantilevers equipped with a durable diamond tip and a nominal force constant of 2.8 N/m for normal bending (Adama Innovations, Ireland). Since either bonds between two surface atoms or a bond between a surface atom and a tip atom need to be broken, it would rather be surprising if the effective frequency with which both bonds are strained would be the same, e.g., due to different atomic mass or different bond environment otherwise. As an example, for influence of atomic configuration on friction determining dynamics one might consider the publication by Cannara *et al.* [41] which considers nanoscale friction variations based on isotopic shifting of surface vibrational frequencies. In addition, a different attempt frequency for the formation and breaking of bonds was already assumed by Barel *et al.* in their ground-breaking publication on multibond dynamics for nanofriction [31]. Prior to the experiments, the SiO₂ has been heated to approximately 380 K for 1 h in order to remove adsorbates. The sample temperature was varied between 95 K and room temperature. Wear studies were done for up to eight different temperatures within this range for two sets of independent experiments. The sequence of temperatures was partially randomized to exclude influences of possible tip changes. In addition, all diamond AFM tips

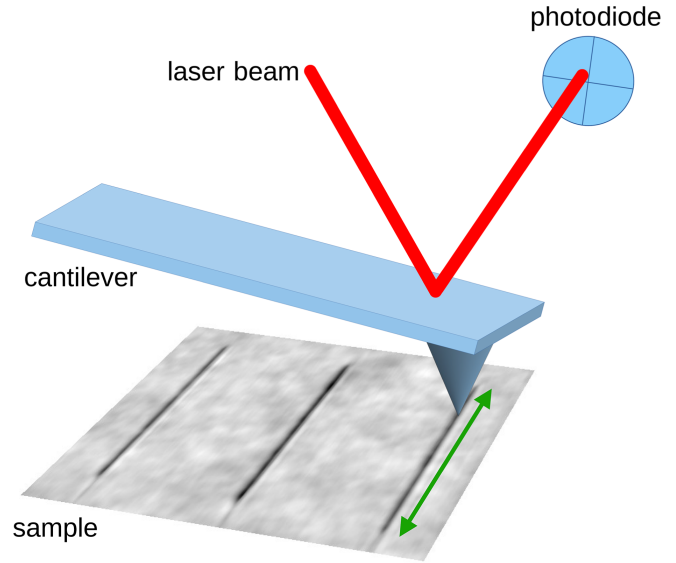


FIG. 1. Experimental approach for fabrication of wear scars on a SiO₂ substrate by reciprocal back and forth scanning together with an exemplary experimental result for a set of three wear scars produced at 95 K.

used in this Letter have been used considerably before the actual wear experiments in order to prevent sudden changes of the initially very sharp tips.

The experimental approach together with an exemplary result for AFM-induced surface modifications is shown in Fig. 1. As a first step, we produce wear scars by prolonged reciprocal scanning for 800 times using normal forces of 156 (series I) and 72 nN (series II). Typically, a set of three parallel tracks of 300 nm length is generated at each temperature, and a control image of these three tracks is recorded immediately afterward (Fig. 1). Please note that during the experiments temperature-induced drift can

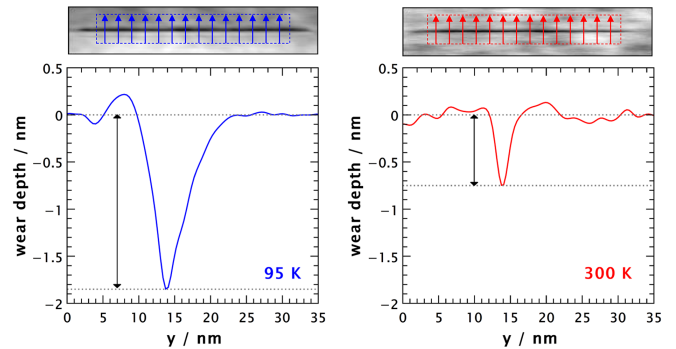


FIG. 2. Depth analysis of wear scars produced by reciprocal scanning at temperatures of 95 K (left) and 300 K (right). The shaded squares within the topography images indicate the width along which the individual profile lines perpendicular to the wear scar have been averaged. These average profile lines together with the resulting maximum wear depth are then shown below the topography images.

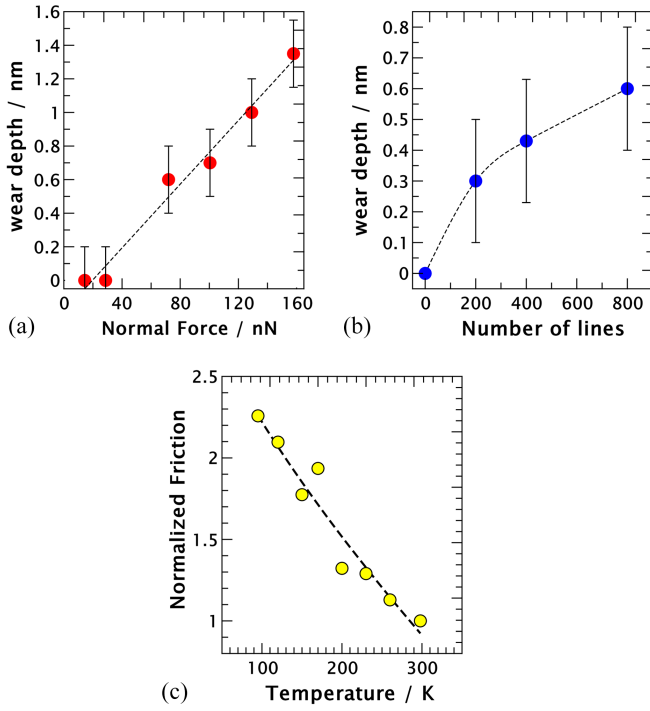


FIG. 3. Dependence of the wear depths on applied normal force (a) and number of reciprocal line scans (b) for a diamond AFM tip sliding on SiO_2 at a temperature of 150 K. A constant number of 800 lines was scanned for each data point in (a), while a constant normal force of 72 nN was used in (b). In addition, (c) shows the temperature-dependent friction obtained from the first scan lines of each wear experiments (yellow spheres). The friction values have been normalized by the value obtained for 300 K. In (c), the dashed line marks the theoretically expected decrease of friction with rising temperature, although it does not correspond to a full quantitative fit of the thermally activated Prandtl-Tomlinson model.

become a problem due to the time-consuming process of reciprocal scanning. Incomplete wear scars were not included in the analysis.

The depth of the wear scars can be used as a measure for the amount of wear [11,40]. Each of the tracks was analyzed by averaging the line profiles perpendicular to the track. From this average profile, the maximum depth is taken as a measure to quantify the wear of the substrate. This is demonstrated in Fig. 2, where two wear scars obtained at 95 and 300 K have been analyzed yielding depths of 1.91 and 0.62 nm, respectively. Figure 2 shows surprisingly low wear rates of up to 0.001 nm per passing of the tip. Therefore, our experimental regime is markedly different from previous studies, where DLC-coated AFM tips have been used to produce similar wear scars on Si wafers [42].

The depth of the wear scar can be controlled by variation of both the normal force and the number of scan-line repetitions, as shown in Fig. 3. In both cases, we find a monotonic albeit not linear increase of the wear depths.

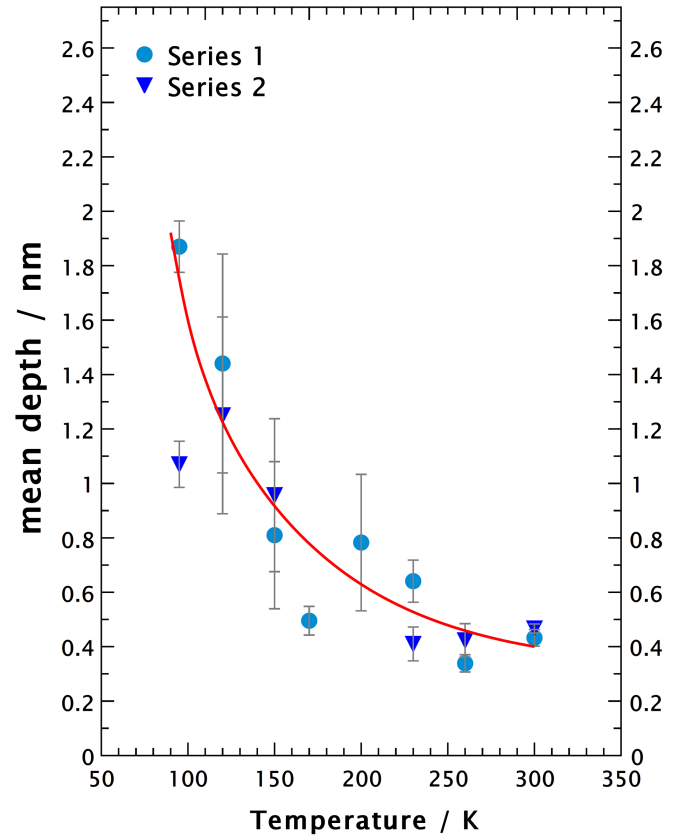


FIG. 4. Temperature dependence of nanoscale wear for two sets of experiments obtained on SiO_2 using two different diamond AFM tips. Normal forces of 156 (series 1) and 72 nN (series 2) have been used, while in both cases the lines have been scanned reciprocally 800 times. In addition, a theoretical curve to describe the data is shown as a continuous line. This curve has been obtained using the following parameters: $E_{\text{on}} = 0.3$ eV, $E_{\text{off}} = 0.38$ eV, $E_{\text{wear}} = 0.32$ eV, $f_{\text{on}} = 5 \times 10^5 \text{ s}^{-1}$, $f_{\text{off}} = 5 \times 10^7 \text{ s}^{-1}$, $f_{\text{wear}} = 2 \times 10^6 \text{ s}^{-1}$, and $\alpha = 0.1$ eV.

For the normal force, we find a minimum threshold $F_N > 20$ nN that needs to be exceeded before discernible wear effects occur. For the scan line dependence, we observe that the overall wear rate decreases and becomes linear only after the first 200 lines, reminiscent of a nanoscale run-in process. In addition, Fig. 3(c) shows the temperature dependence of relative friction $F_{\text{rel}}(T)$ averaged from the first few scan lines during each wear experiment. We find monotonic decrease of friction with temperature, in agreement with the conventional model of thermally activated friction (Prandtl-Tomlinson model) [34]. The lateral force profile during back and forth scanning always remained flat. Because of the large scan range, stick slip characteristics or single wear events could not be resolved.

Figure 2 already suggests that wear effects are more pronounced for low temperatures. The complete temperature-dependent series consists of two sets of experiments shown in Fig. 4, using two different diamond tips. Wear scars of 300 nm length have been produced using scan

speeds of 1 $\mu\text{m/s}$. Roughly similar wear rates for both tips could be achieved by adjusting the normal forces, despite potential differences in tip radius. Consequently, comparable pressures and shear stresses are assumed to be acting at the interface and the same theoretical curve could be applied to describe the normalized results (see below).

The results in Fig. 4 are in clear contradiction to current models, where nanoscale wear is viewed as a process of thermally activated atom-by-atom removal. Experiments on ionic crystals like NaCl and KBr [7,40] showed that nanoscale wear increases with temperature, as the model predicts.

However, we also have to consider that contact aging was recently reported for diamond tips sliding on SiO_2 [38] which potentially allows for alternative wear mechanisms [8,43]. The aging process was rationalized based on interfacial bond formation, similar to the concepts applied to aging of silica nanojunctions under both ambient [20,36,37] and UHV conditions [24,38]. Already in these temperature-dependent contact-aging studies performed under UHV conditions, the increase of wear with decreasing temperature was briefly mentioned as a reason for a limited temperature range for the contact-aging experiments [24,38]. This previous qualitative observation prompted the quantitative wear study presented here, which now allows us to examine the underlying wear mechanisms with temperature as the decisive parameter. In particular, we can extend the emerging multibond model of single-asperity nanofriction and tribochemical wear into the temperature domain, thereby adding a crucial new dimension to the overall understanding of the interfacial processes that drive wear.

To include the effects of interfacial bond formation into the process of wear generation, we now consider a model as discussed by Shao *et al.* [8], that focuses on the breaking of newly formed bonds at the interface. As illustrated in Fig. 5, this breaking can occur either within the substrate or directly at the interface between slider and substrate. While the former process contributes to the generation of wear, the latter one is wear-free and no material is removed.

In principle, the model by Shao *et al.* is an enhanced multibond model where wear is considered as an additional competing rate process [8]. In this model, three different energy barriers E_{on} , E_{off} , and E_{wear} are taken into account to describe the formation and breaking of bonds as thermally activated process with a corresponding attempt frequency.

Here, the index “on” is related to the formation of bonds, while “wear” and “off” refer to the energy barriers for wear-inducing and wear-free bond breaking, respectively. Based on this approach, wear rates can be calculated for different regimes of normal forces (which effectively reduce the energy barriers) and sliding velocities.

In our case, we find that the experimental wear rates are extremely low, and we assume that only very few bonds are formed at the interface. In this case, the “low effective

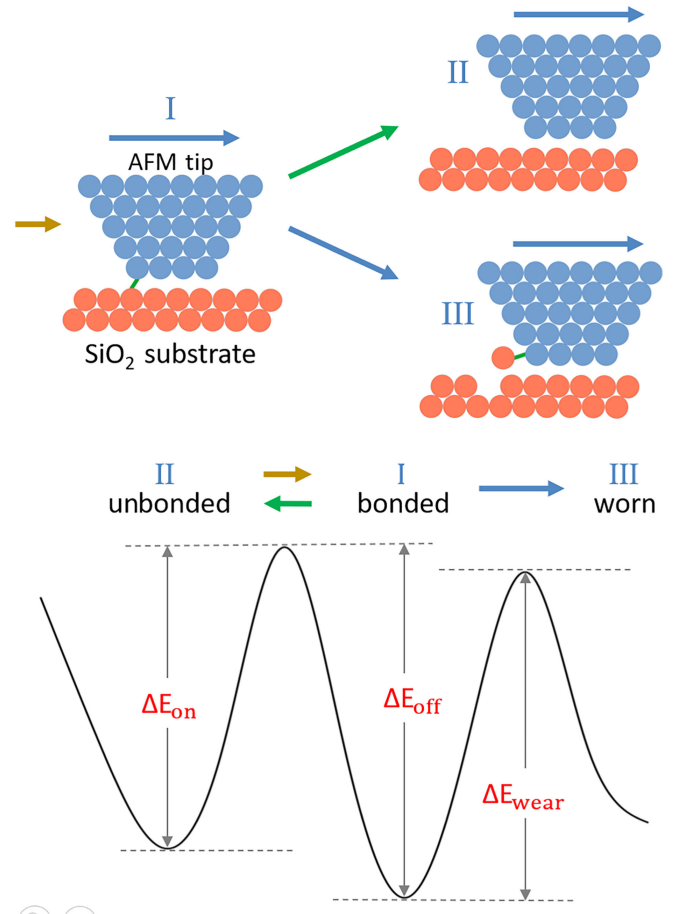


FIG. 5. (a) Simplified concept of nanoscale wear involving bond formation and rupture. (b) Potential energy landscape for the different processes involved (cf. [8]). Once a bond between tip and substrate is formed (process I), it must be ruptured when the tip continues to move. This rupture can either leave tip and surface unchanged (II) or remove material from the substrate (III). All processes involve energy barriers as shown in (b) (tip wear is not considered in this scheme).

normal stress regime” appears appropriate, and we describe the temperature-dependent wear rate Γ as

$$\Gamma(T) = \frac{f_{\text{on}} e^{-E'_{\text{on}}(T)/kT} f_{\text{wear}} e^{-E'_{\text{wear}}(T)/kT}}{f_{\text{on}} e^{-E'_{\text{on}}(T)/kT} + f_{\text{off}} e^{-E'_{\text{off}}(T)/kT} + f_{\text{wear}} e^{-E'_{\text{wear}}(T)/kT}}. \quad (1)$$

This model thereby simplifies the complex dynamics at the silica-diamond interface by assigning a single, discrete value to the energy barriers of the different processes.

Compared to Shao *et al.*, Eq. (1) is modified by two aspects: First, we now consider different attempt frequencies f for the different processes. Since either bonds between two surface atoms or a bond between a surface atom and a tip atom need to be broken, it seems reasonable to assume different frequencies here with which both of the bonds in different environment are strained, similar to the

influence of isotopic shifting of surface vibrational frequencies on nanoscale friction observed by Cannara *et al.* [41]. In addition, different attempt frequencies for the formation and breaking of bonds was already assumed by Barel *et al.* in their early publication on multibond dynamics for nanofriction [31].

Second, we also consider that all energy barriers are effectively reduced by a temperature-dependent shear stress $\tau(T)$ to $E'(T) = E - \tau(T)/V_a$ with V_a a characteristic activation volume [6,40]. This shear stress originates from sliding friction and, therefore, mirrors the temperature dependence in Fig. 3(c). The additional energy $\Delta E(T)$ can, thus, be described as $\Delta E(T) = \tau(T)/V_a = \alpha \cdot F_{\text{rel}}(T)$, with α a constant summarizing the contact geometry. Using Eq. (1), the experimental results can then well be reproduced using the parameters listed in the figure caption.

The experimental results can best be approximated when the absolute values of the energy barriers are of similar magnitude, while we find larger differences between the attempt frequencies. On one hand, this might be related to general differences in bond characteristics. However, the difference between f_{off} and f_{wear} might also reflect the fact that only a smaller amount of weakly bond surface atoms might be suitable for removal at all. In addition, the relatively low value of f_{on} might be related to the fact that not every surface position is suitable for bond formation.

At this point, we discuss the origin of $F_{\text{rel}}(T)$, which is the interfacial friction and in first order is independent of the atomic wear process itself. This separates two kinds of interface interaction, namely, van der Waals forces and stronger bonds. Since the number of bonds is very low, the sliding process itself can well be described by the thermally activated Prandtl-Tomlinson model, which results in a temperature-dependent shear stress acting on the interface. Parallel to that, formation and breaking of bonds takes place and is affected by the shear stress through reduction of energy barriers. As a first step, no further link between the two interaction mechanisms is considered. (Please note that this approach is further simplified by not taking into account any kind of wear of the durable diamond tip.)

In this picture, the wear rate can then increase with decreasing temperature, since the combined influence of temperature and shear stress does not only affect the ratio between wear inducing and wear-free bond breaking, but also the bond formation rate can increase.

Strictly speaking, the theoretical approach is valid only for very low wear rates with negligible forced unbinding [8]. While the wear rates always remain low over the whole temperature range, forced unbinding cannot completely be ruled out at lower temperatures. However, the experimental results can still be described well by thermally activated bond formation and bond breaking.

In addition, the experimental results can phenomenologically be reconciled with the previous analysis of temperature-dependent contact aging in diamond and silica systems by slide-hold-slide experiments [24,38]. Here, the initial contact stiffness, i.e., a measure for the number of

interfacial bonds, was found to increase with decreasing temperature for all hold times. If this trend is not overcompensated by a temperature-dependent shift between wear inducing and wear-free unbonding, larger wear rates at low temperatures can be expected.

For even lower temperatures, which are not accessible when using liquid N₂ as a coolant, one might speculate that the wear rate will eventually drop off again, since very low temperatures will prevent bond formation even in the presence of increasing shear stress.

In conclusion, our results showed a temperature dependence of nanoscale wear, where a surprising decrease of wear with increasing temperature was experimentally found. This decrease of the wear volume with temperature cannot be reconciled with the well-established concept of thermally activated and shear-assisted atom-by-atom removal [6,39,40]. Instead, a modified multibond model which takes into account the shear-assisted formation of interfacial bonds and their subsequent rupture along two different paths can qualitatively explain the general temperature dependence of wear between diamond and SiO₂. Nonetheless, thermally activated and shear-assisted atom-by-atom removal cannot be ruled out completely and might become dominant when even higher sample temperatures are considered.

In future works, it would now be interesting to achieve a more detailed picture of the interface processes, in particular, by complementary tip characterization (e.g., by SEM analysis). Temperature variations might then open pathways to control wear by limiting it to either tip or substrate. However, this far exceeds the simple model presented in Fig. 5 even if an additional channel for tip wear is introduced. Instead, both *ab initio* and MD simulations would be required to capture tribochemical and interfacial processes with full complexity. As a first step, it would also be helpful to solve the equation of motion when considering both van der Waals interaction and interfacial bond formation to see how their link can influence wear.

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Data availability—The data that support the findings of this article are not publicly available upon publication because it is not technically feasible and/or the cost of preparing, depositing, and hosting the data would be prohibitive within the terms of this research project. The data are available from the authors upon reasonable request.

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