STM-QCM STUDIES OF VAPOR PHASE LUBRICANTS

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Abstract:

Vapor phase lubricants have been studied for well over 40 years, generally within the context of macroscopic system performance. Nonetheless, they may well prove to be of critical importance to the tribological performance of MEMS devices, since the vapor phase may ultimately prove to be an effective, and perhaps exclusive, means to deliver and/or replenish lubricants. With the intent of developing a realistic laboratory test set-up for actual MEMS contacts, we have combined a Scanning Tunneling Microscope (STM) with a Quartz Crystal Microbalance (QCM). The STM-QCM allows unique and detailed investigations of the simple nanomechanical system formed by a contacting tip and surface. Both STM images of the contact and the response of the QCM are monitored throughout the course of the measurements, which are performed in realistic sliding conditions of over 1 m/s. We report here on both (vapor phase) lubricated and non-lubricated contact.
1. INTRODUCTION

Studies of the atomic-scale origins of friction and adhesion have undergone rapid progress in recent years with the development of new experimental and computational techniques for measuring and simulating tribological phenomena at atomic length and time scales.\[1\] Employing established technologies, such as ultra-high vacuum, for the preparation of crystalline samples, nanotribologists have been gathering information in situations where the nature of the contacting surfaces is determined in advance of the measurement. They have collectively measured friction forces per unit true contact area which span twelve orders of magnitude,\[1\] with no wear or damage occurring at the sliding interface. Faster computers have in turn allowed large scale molecular dynamics (MD) simulations of condensed systems to be performed for physically significant time periods, enabling numerical results to be increasingly comparable to experiment.\[2\]

If the precise nature of the contacting asperities between macroscopic objects in sliding contact could be determined (such studies do in fact represent an area of high research activity within the tribological community), then the results of nanotribological studies could begin to be directly implemented into mainstream tribological considerations. Meanwhile, the results are most applicable to friction at the interface between liquid and solid materials,\[3,4\] where the complicating factors associated with asperity contacts are less of an issue, and to the MEMS community,\[5\] where machine components with astoundingly small dimensions are rapidly approaching the length scales routinely probed by the nanotribological community. Indeed, solid surface nanocontacts abound among MEMS devices, and a myriad of device complications and failures are associated with their friction, adhesive and wear characteristics.\[6\] Because each of these contact areas is small, perhaps a few tens of atoms in extent,\[7\] both the topology and the mechanics of the contacting asperities must be investigated at the atomic scale in order to optimize device performance.

Current issues of importance to this area include: (1) Development of realistic laboratory test set-ups which are both well-controlled and relevant to operating machinery, (2) Understanding the chemical and tribochemical reactions which occur in a sliding contact, and the energy dissipation mechanisms associated with such a contact, (3) Merging and coordinating information gained on the atomic-scale with that observed at the macroscopic scale, (4) Characterization of the microstructural and mechanical properties of the contact regions between the sliding materials, and (5) Development of
realistic interaction potentials for computer simulations of materials of interest to tribological applications.

We focus here on points (1), (2), and (3), and describe herein our efforts to study the adhesion, friction and tribochemistry of atomic-scale contacts in a controlled environment by means of a Scanning Tunneling Microscope (STM) which is operated in conjunction with a Quartz Crystal Microbalance (QCM). A schematic of the experimental set-up is shown in Fig. 1. The STM allows a single asperity contact to be formed, and allows the buried contact to be imaged in both stationary and sliding conditions. The microbalance, whose surface is oscillating in transverse shear motion at speeds near 1 m/s, can be employed to measure the uptake rate and frictional properties of adsorbed lubricant species. The STM-QCM combination[8] allows access to a range of contact pressures and sliding speeds which are comparable to those encountered by actual MEMS devices.[9] Traditional instruments such as the Atomic Force Microscope (AFM) and the Surface Forces Apparatus (SFA) fail to access either the required sliding speeds or contact pressures, respectively. Furthermore, the use of STM avoids the problem of jump-to-contact associated with AFM, allowing the applied normal force to be varied continuously in a controlled fashion.

Figure 1. A schematic diagram of the STM-QCM apparatus.
In what follows, we first describe how a QCM acting alone can provide important information about lubricant layers adsorbed from the vapor phase. Next, we discuss our results obtained with the combined STM-QCM in three situations: (1) measurements of the amplitude of a vibrating QCM electrode with the STM tip in tunneling, but not physical, contact with the QCM, (2) unlubricated metal-metal contact of the STM tip with the QCM electrode, and (3) lubricated contact using a vapor phase lubricant which is known to reduce wear in macroscopic applications. In the case of unlubricated contact, we observe significant wear and evidence of increasing sliding friction with normal load. Application of molecularly thin quantities of lubricant to the same contact dramatically alters both the STM and QCM responses in a manner which is highly suggestive of the lubricant’s known friction and wear reducing properties at the macroscopic scale. Our measurements also reveal a potential tribochemical reaction which is highly localized at the point of contact and is associated with the realistic rubbing conditions provided by the STM-QCM apparatus.

2. QCM STUDIES OF VAPOUR PHASE LUBRICANTS

The concept of lubricating high temperature bearing surfaces with organic vapors has existed for at least forty years, with substantial efforts beginning in the 1980's and continuing on to the present day.[10] Vapor phase lubrication occurs via three distinct forms: (a) organic films which are intentionally reacted with a surface to form a solid lubricating film, (b) vapors which condense to form a lubricating liquid film on the surface of interest, and (c) light weight hydrocarbon vapors deposited onto hot catalytic nickel surfaces. Vapor phase lubricants are advantageous for use at high temperature, (meaning that either the ambient temperature of the entire system is elevated, or the local surface “flash point” temperature due to frictional heating is elevated) and in situations where the vapor can be used as a reservoir for replenishment of areas where the lubricant has been depleted in the course of the bearing lifetime.

Several organic vapors have been identified which exhibit desirable tribological properties,[11] but a detailed and fundamental understanding of their surface chemical reactions in the tribological processes of interest is far from complete. In particular, an understanding of the specific surface reactions which occur, and how they are affected by tribological conditions such as temperature and/ or lubricant concentration level in the carrier gas remains inadequate. Modern nanotribological techniques can be brought to bear on these issues by examining in detail the properties of a known
(macroscopic-scale) vapor phase lubricant. The knowledge gained (if not the lubricant itself) is extremely likely to be applicable to NEMS/MEMS operations as well. Indeed, the vapor phase may ultimately prove to be the most effective, if not only, means to deliver and/or replenish a lubricant in the case of a submicron scale device.[12]

The vapor phase lubricant chosen for our study consists of a blend of tertiary-butyl phenyl phosphate (TBPP) molecules,[13] whose atomic constituents are carbon, hydrogen, oxygen and phosphorus. It demonstrates high quality performance at elevated temperatures [10] and exhibits oxidation inhibiting characteristics as well as a number of other desirable tribological properties, such as the reduction of wear. While the precise mechanisms for its beneficial properties are uncertain, it is believed that after reacting with the surface, the phosphate contained in the original lubricant molecule acts as a binder for graphitic carbon, which in turn may be the actual lubricant.

The QCM is particularly well-adapted for measurements of uptake rates of vapor phase lubricants.[14] It has been used for decades for microweighing purposes, and was adapted for friction measurements in 1986-88 by Widom and Krin.[15] Specifically, a QCM consists of a single crystal of quartz that oscillates in transverse shear motion with a quality factor near $10^5$. The driving force (supplied by a Pierce oscillator circuit) has constant magnitude and is periodic with frequency $f = 4$ to 10 MHz, the series resonant frequency of the oscillator. Two metal electrodes, which serve as the substrates upon which adsorption occurs, are deposited in vacuum conditions onto each major face of the crystal.

Film adsorption onto the microbalance produces shifts in both the frequency and amplitude of vibration, which are simultaneously recorded as a function of pressure. Amplitude shifts are due to frictional shear forces exerted on the surface electrode by the adsorbed film (or alternatively by a three dimensional vapor or fluid phase). Our microbalance in its present arrangement can detect shear forces in excess of $2.5 \times 10^{-7}$ N.[15]

Figure 2 depicts a typical QCM response for the uptake of TBPP lubricant on an iron surface at room temperature. The uptake is relatively slow, corresponding to a total of 4 monolayers in approximately one hour’s exposure to 234 mTorr of the vapor. At the pressures we have studied, the rate is independent of TBPP gas pressure. Moreover, the uptake rate returns to its $t = 0$ value when the sample is left for a day with no exposure to further TBPP, even though the TBPP is not observed to desorb from the surface. These observations are consistent with slow diffusion of the TBPP or fragments thereof into the iron substrate, consistent with previous studies.[10] The fact that amplitude shifts are in fact observed indicate that
the film is either slipping on account of the oscillatory motion of the microbalance, or else exhibiting an internal molecular motion within the TBPP molecules themselves. (A solid film adsorbed on a surface with no slippage would produce no shift in the amplitude of vibration of the microbalance).

*Figure 2. Uptake of TBPP on iron: The frequency drop indicates mass adsorption. Shifts in amplitude are related to adsorbate motion, either slippage or internal molecular motion of the adsorbed species.*

The frequency shifts observed during the uptake of TBPP in Fig. 2 are negative, simply due to the added mass of the film to the oscillating system. In Section 4, we discuss the need for more sophisticated modeling of the STM-QCM geometry to account for the frequency shifts observed with this apparatus, which may be either positive or negative.

3. **STM IMAGING OF A MOVING QCM ELECTRODE**

A remarkable consequence of the STM-QCM combination is that the amplitude of vibration at the surface of the QCM may be directly measured using the STM images.[16] Such measurements are an important prelude to our studies of friction with this apparatus, allowing accurate determination of the maximum sliding speed of the tip-surface interface. We note that the QCM frequency response to applied forces plays no role in this measurement. In fact, we observe no frequency shift upon engaging the tip into tunneling contact, to within the resolution of +/- 0.1 Hz out of 5 MHz. This indicates that there is no observable normal load on the crystal when the
tip is in tunneling contact, provided that both the tip and electrode surfaces are bare metals in high vacuum ($\sim 10^{-8}$ Torr in our experiments). As will be discussed in Section 5, the presence of even a thin adsorbed layer on the tip and electrode surfaces results in a QCM frequency shift during tunneling contact, due to the normal load required to squeeze the two metal surfaces sufficiently close together for tunneling.

The ability to image a vibrating surface with an STM, while unexpected, may be attributed to the three widely separated time scales involved, and the exponential dependence of the tunneling current on tip-surface separation. The characteristic frequencies of the scanner (Hz), the feedback loop (kHz), and the QCM (MHz) are each separated by three orders of magnitude. Conventional STM operation relies on the feedback loop being much faster than the scanner (in constant-current mode). For STM-QCM, the fact that the vibrations of the QCM are in turn much faster than the feedback loop causes the tip to be held at a separation from the surface which on average (over many surface oscillation cycles) gives the desired tunneling current (typically 1 nA). Qualitatively, the closest approach of the surface to the tip during each cycle is weighted most heavily in this average, due to the exponential dependence of the tunneling current on separation, so the tip is held at an altitude sufficient to avoid direct contact of the tip and surface. This allows the STM tip to image the vibrating surface without crashing into it, as is evident from a lack of damage to the surface.

The amplitude measurements proceed as follows: We employ an AT-cut quartz crystal for our QCM, which oscillates in transverse shear mode whereby displacements are in the plane of the surface. The STM tip is held in tunneling contact (in constant-current mode) with the QCM electrode (see Fig. 1). Simultaneous operation of STM and QCM is accomplished using the electrical circuit detailed in Ref. [16]. The amplitude of vibration is measured by observing the lateral smearing of features along the shear direction as the drive voltage of the QCM is increased.

Figure 3 displays a pair of STM images of the QCM surface. The surface is that of a copper electrode deposited in situ in high vacuum. The same 500 nm $\times$ 500 nm region is shown while the QCM is stationary (a) and vibrating (b). In image (b), the crystal is shown oscillating at its fundamental frequency of 5 MHz, with a quality factor of 54,000, and a peak drive voltage of 0.93 V applied across its electrodes. The horizontal elongation of features apparent in this image depicts the lateral extent of the surface vibrations. By comparison with image (a), the amplitude was determined to be 75 nm. A full exploration of the dependence of vibrational amplitude on drive voltage and quality factor is presented in Ref. [16]. In the present case, the maximum
speed of the surface is \( v = A \omega = 2.3 \text{ m/s} \), a direct verification that the STM-QCM apparatus achieves realistic interfacial sliding speeds.

\[ \text{(a)} \quad \text{and} \quad \text{(b)} \]

\textit{Figure 3.} A pair of 500 nm \( \times \) 500 nm STM images of the surface of a QCM. The full vertical scale is 40 nm. The same region on the QCM is shown both stationary (a) and oscillating (b). The apparent horizontal elongation of features in (b) indicates the lateral extent of surface vibrations. By comparison with (a), the amplitude of vibration was measured to be 75 nm. This results in a maximum sliding speed of 2.3 m/s at the interface of tip and surface.

4. **UNLUBRICATED CONTACT**

In the previous section, tunneling contact was established between the STM tip and QCM electrode, whose bare metal surfaces were maintained in high vacuum. There was no QCM response or any significant wear associated with this type of contact. In this section, we establish unlubricated physical contact between the tip and surface, and describe the resulting wear (shown in STM images) and associated QCM response. In Section 5, we compare these results to the case of lubricated contact between the same tip and surface, using TBPP as the lubricant species. In this way, we study the action of a (macroscopic) lubricant on a known interface using the combined capabilities of the STM-QCM.

For our system, the hallmark of even light physical contact between tip and surface is the presence of deformation and wear (a situation familiar to many STM operators, and often referred to as crashing the tip!). Figure 4 shows a case of carefully controlled wear: the scratching or machining of a platinum surface (again deposited in-situ in high vacuum) using a tungsten tip. The region shown is 1700 nm \( \times \) 1500 nm. The central mound in image
(a) is roughly 50 nm high. Image (b) shows the result of making two horizontal scratches in the surface by scanning the tip across the mound at a velocity too fast for the feedback loop to move the tip vertically enough to prevent it from crashing into the mound (5 μm/s). We estimate that the tip was pressed 20 to 40 nm into the mound. The full vertical range of our scanner piezo is 800 nm, so only a small fraction of the available normal force was employed to produce these scratches. Similarly, various holes and depressions may be produced by holding the tip fixed, or by scanning, while pressing the tip into the surface. We observe that machining of this surface with the tungsten tip is always accompanied by a negative frequency shift of the QCM as the tip is forced into the surface. The frequency shift is increasingly negative as the normal load is increased. In the next section, we observe that after lubricating this contact, the frequency shift is increasingly positive as the normal load is increased, and we present a plot of the frequency shifts versus normal load for these different types of contact (see Fig. 5). The observation of both positive and negative QCM frequency shifts is not without precedent when considering different regimes of coupling for a point contact under shear stress.[17] In our geometry, an increasing normal load almost certainly results in an increase in sliding friction at the interface. We find that this is associated with an increase in the absolute value of the frequency shift.

Figure 4. A pair of 1700 nm × 1500 nm images showing the machining of a platinum QCM electrode by a tungsten tip. The central mound in image (a) was scratched horizontally in two places by scanning the tip quickly across the mound. The final result is shown in (b). The inset in (b) shows a line section across the two scratches. This unlubricated metal-metal wear is accompanied by a negative QCM frequency shift.
Clearly, careful modeling is needed to account for the various QCM frequency responses observed here. In general, positive frequency shifts are associated with increased stiffness of the system and negative shifts are associated with added mass or increased damping. In previous experiments where small spheres or wires were pressed into QCM’s operated in air, positive frequency shifts were exclusively observed.[18,19] These results were attributed to plausible mechanisms which would serve to increase the stiffness of the system in the geometry of probe and surface. Our results with STM-QCM in high vacuum conditions, exhibiting both positive and negative frequency shifts, show that more complete modeling is necessary to describing the competing effects of increased mass, stiffness, and/or damping in the coupling between tip and surface. For quantitative predictions, such modeling will likely need to incorporate a variety of factors, including the tip and surface materials, the presence or absence of intervening adsorbed layers, tip length, contact area, and normal load. For the time being, we focus on comparative studies where the exact same surface and tip are used before and after introducing an intervening lubricant layer. For instance, in the next section we present our results for a lubricated contact, employing the same tungsten tip and platinum surface as were used in our experiments with the unlubricated contact.

5. LUBRICATION CONTACT

We now study the case of lubricated contact between the tip and surface, employing the (macroscopic) lubricant and anti-wear additive TBPP, which was discussed in Section 2. This case is particularly relevant to the possibility of vapor phase lubrication of MEMS devices. Having characterized the unlubricated contact of our tungsten tip and platinum surface, as discussed in the previous section, we exposed the same system to TBPP vapor. The QCM registered an uptake of approximately 1 monolayer. The tip is expected to have adsorbed a comparable amount. The electrode and tip surfaces were at room temperature during deposition.

The response of the QCM to the normal force of the tip undergoes a dramatic change as the lubricant layer is introduced. Figure 5 shows the frequency shift versus normal load for both lubricated and unlubricated contact between our tungsten tip and platinum surface. We find that upon adsorption of TBPP, the QCM responds to the normal force of the tip with a positive frequency shift rather than negative, as observed for unlubricated contact. Another contrast is that the frequency shift remains increasing and positive up to the maximum normal load for lubricated contact, whereas the
shift remains negative and decreasing over the range investigated for unlubricated contact. This striking change in the response of the QCM may well be a nanometer-scale signature of the friction and wear reducing properties of TBPP known from macroscopic observations.

![Graph showing QCM frequency shift versus normal load](image)

*Figure 5.* A plot of the QCM frequency shift versus normal load of the STM tip for lubricated, lubricated and annealed, and unlubricated contact. (The arbitrary units for normal load are obtained from the $Z$ voltage of the scanner piezo. The full range represents a deflection of 800 nm in the vertical direction in the absence of a contacting surface). The same tungsten tip and platinum surface are used in each case. The lubricated contact involves a molecularly thin layer of TBPP, a known lubricant on the macroscopic scale, on the tip and surface. The direction of frequency shift for lubricated contact is opposite that observed for unlubricated, metal-metal contact. This striking contrast may be a nanometer scale signature of the film’s lubricating properties. The magnitude of the shift is smallest by far for the lubricated and annealed contact, suggesting that sliding friction is most reduced in this case.

Additional insight into the properties of the lubricated interface may be obtained with the STM. Figure 6 shows a 350 nm × 350 nm STM scan of the surface immediately after exposure to the lubricant. We find that in order to obtain an image, it is necessary to run the QCM while scanning. No stable image is obtained when the QCM is off. We understand this result by considering the following: When the QCM is off, the tip-surface junction is quite insulating. The “$Z$” voltage, which controls the vertical position of the tip, is unstable while a constant tunneling current “$I$” is maintained through feedback. The so-called $I-Z$ dependence is as follows: A change in voltage
corresponding to a deflection of 100 to 300 nm into the surface is required to raise the tunneling current by 10 nA! Most likely, increases in the normal force and contact area are responsible for the increased current, not an actual change in tip-surface separation of such a large magnitude. (In contrast, for a clean metal-vacuum-metal tunneling junction, a change in Z voltage corresponding to a deflection of less than 1 Å changes the tunneling current by 10 nA). When the QCM is turned on and the tip engaged at 10 nA, the junction becomes more conductive over time (a few seconds or minutes) until an I-Z dependence close to that of tunneling through vacuum is achieved. This applies only to the region in close proximity to the tip. Changes in the Z voltage and QCM frequency indicate a decreasing normal load as the junction becomes more conductive. Under these conditions a recognizable image of the surface may be obtained. However, the junction returns to insulating as soon as the QCM is shut off, or if the tip is moved away from the region or raised up from the surface.

\[\text{Figure 6. A 350 nm} \times \text{350 nm image of the TBPP-lubricated platinum surface at room temperature. The rubbing action of the vibrating QCM electrode against the STM tip is required to produce a stable image. The rubbing appears to clear away the lubricant, producing localized holes in which the surface may be imaged. These holes fill-in with additional lubricant once the QCM is turned off or the tip is moved away from the region, indicating that the TBPP film is mobile at room temperature.}\]

Our interpretation of these observations is that the rubbing action of the vibrating QCM electrode against the STM tip effectively brushes the insulating lubricant layer aside to allow imaging of the underlying surface, similar to a windshield wiper except that the vibration of the QCM surface is critical in the process. Holes or clear areas produced in this way are not
permanent, however, and readily fill in with additional lubricant once the QCM is turned off or the tip is moved to a different position. We therefore conclude that there is a high degree of mobility associated with the TBPP layer at room temperature. This corresponds to the results of adsorption studies with QCM alone discussed in Section 2, in which the observed change in amplitude of the QCM during adsorption of TBPP indicated either wholesale slippage of the layer or intermolecular mobility. The ability of a lubricating film to replenish its depleted areas is indeed a known requirement for good lubrication.

Thus far, our studies of the lubricated interface have been performed exclusively at room temperature. TBPP is known to undergo a chemical reaction above 100°C which renders the film more conductive. In view of this, we have investigated the lubricated interface after annealing for several hours near 100°C. (The entire vacuum system was raised to elevated temperature). Indeed, we find that the TBPP-lubricated platinum surface is much more readily imaged after annealing. The tip-surface junction is conductive enough to maintain a stable tunneling signal with or without the QCM in operation. Figure 5 shows that the magnitude of the positive QCM frequency shift over the full range of normal load is significantly reduced. This suggests a reduction in sliding friction at the interface. Moreover, STM images of the annealed surface help identify a more subtle rubbing-induced response, which may provide evidence for a tribochemical reaction.

Figure 7 displays a pair of images of the lubricated surface after annealing. Image (a) shows an non-rubbed 70 nm x 70 nm region, for which the QCM has not yet been operated with the tip engaged. The image has a fringed appearance. (Due to the presence of the lubricant film, the appearance of the surface in these constant-current images is not to be interpreted literally as the topography of the surface. The imaging mechanism is unknown, and electronic effects are clearly present). Image (b) shows the same region after the QCM was operated for a few minutes with the STM tip held in tunneling contact at 2 nA. This short rubbing experiment produced two distinct regions in the vicinity of the tip. The majority of image (b) has a uniform appearance, but a region in the upper left remains which bears the fringed appearance of image (a). Interestingly, the two regions are accompanied by different QCM frequency responses. In both (a) and (b), the fringed region registers a small positive frequency shift when the tip is briefly engaged in tunneling (at 2 nA). The uniform region in (b) registers no frequency shift. In general, we find that if the tip is held in tunneling contact at a fringed region while the QCM is operated, a small positive frequency shift appears and decays over a period of a minute or so, leaving a uniform region with no frequency shift for the same tunneling conditions.
Figure 7. A pair of 70 nm × 70 nm images of the TBPP-lubricated platinum surface after annealing for several hours near 100 °C. QCM frequency shifts upon establishing tunneling contact at different places are shown in boxes. Image (a) shows a non-rubbed region. Image (b) shows the same region after a rubbing period of a few minutes, during which the QCM was vibrating with the tip engaged in tunneling contact. Two regions may be distinguished: non-rubbed regions have a fringed appearance and are accompanied by a small positive frequency shift when the tip is first engaged in tunneling. Rubbed regions have a more uniform appearance and exhibit no frequency shift when tunneling. The electrical properties of the tunneling junction suggest that the lubricant film is not simply worn away by rubbing. These results suggest a possible tribochemical reaction associated with the interfacial sliding conditions achieved by the STM-QCM.

This rubbing-induced effect is both localized to the region of rubbing and permanent. The uniform regions do not revert to fringed, and fringed regions are only converted to uniform by rubbing the tip against the vibrating QCM. The ability to distinguish rubbed and non-rubbed regions on the nanometer scale by both appearance and corresponding frequency response is intriguing, since improvements in the protective properties of lubricant films after interfacial sliding are well-known, yet poorly understood, on the macroscopic scale.[21] While the mechanism responsible for the change in frequency response observed here is unknown, we find that the tunneling junction never regains the conductivity (I-Z dependence) of a clean metal-vacuum-metal junction. This suggests that the film is not simply worn away by rubbing. Together these results provide evidence for a potential tribochemical reaction triggered by the interfacial sliding conditions attained with the STM-QCM.
6. CONCLUSION

In conclusion, we have combined a Scanning Tunneling Microscope (STM) with a Quartz Crystal Microbalance (QCM) with the intent of developing a realistic laboratory test set-up for MEMS contacts. The STM-QCM allows unique and detailed investigations of the simple nanomechanical system formed by a contacting tip and surface. We have discussed our results obtained with STM-QCM in three situations: (1) measurements of the amplitude of a vibrating QCM electrode with the STM tip in tunneling contact with the QCM, (2) unlubricated metal-metal contact of the STM tip with the QCM electrode, and (3) lubricated contact (before and after annealing) using a vapor phase lubricant which is known to reduce wear in macroscopic applications. In the case of unlubricated contact, we observe significant wear and evidence of increasing sliding friction with normal load. Application of molecularly thin quantities of lubricant to the same contact dramatically alters both the STM and QCM responses in a manner which is highly suggestive of the lubricant’s known friction and wear reducing properties at the macroscopic scale. Our measurements also reveal a potential tribochemical reaction which is highly localized at the point of contact and is associated with the realistic rubbing conditions provided by the STM-QCM apparatus.

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