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Electron Fluctuation Induced Resonance Broadening in Nano Electromechanical Systems: The Origin of Shear Force in Vacuum

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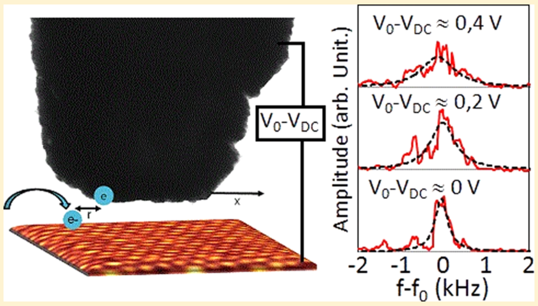
Supporting Information

ABSTRACT: This article presents a study of the poorly understood "shear-force" used in an important class of near-field instruments that use mechanical resonance feedback detection. In the case of a metallic probe near a metallic surface in vacuum, we show that in the 10−60 nm range there is no such a thing as a shear-force in the sense of the nonconservative friction force. Fluctuations of the oscillator resonance frequency, likely induced by local charge variations, could account for the reported effects in the literature without introducing a dissipative force.



KEYWORDS: *Fluctuation, shear-force, dissipation, nanowire, nanomanipulation*

The "shear force" is widely used for the feedback control of the probe/surface distance in near-field instruments,[1] most notably for the scanning near-field optical microscope (SNOM).[2,3] This effect, acting over distances as large as hundreds of nanometers, has been termed shear-force because it appears when the displacement of the vibrating probe is parallel to the surface and it leads to a measurable increase in the dissipation proportional to probe velocity and area similar to the action of a viscous force. This effect should not be confused with a simple frequency shift that appears with an additional conservative force nor with short-range noncontact friction forces that vanish when metallic surfaces are separated beyond few nanometers.

Measurements of the shear force made in air or other fluids can be well explained by considering hydrodynamics or a liquid meniscus and other adsorbed layers. However, the shear-force is also reported in vacuum and in this environment many authors have disagreed over its physical origin. Proposed explanations include occasional knocking on the surface,[1,4] surface friction,[5] tip bending[6] or interactions mediated by a third body.[7]

In clean vacuum and without contact there are only three known noncontact friction forces: van der Waals friction, electrostatic dissipation, and phononic friction.[8] In a recent publication, Kisiel et al.[9] have successfully separated phononic friction from electrostatic dissipation using the supraconducting transition, but certain controversial results are still being debated. Saitoh et al. claimed to have measured gigantic phononic friction[10] that matches results of Karrai et al.[7] Dorofeyev et al. report large van der Waals friction[11] although their results have been contested.[12,13]

In any case, these noncontact friction forces are expected to be of small intensity and short-range for the interaction between a metallic probe and a metallic surface and thus cannot explain the reported long-range shear-force in vacuum. In this paper, the existence of the shear-force is studied using a nanoelectromechanical system (NEMS) in vacuum that consists of vibrating a conducting nanowire in the vicinity of a metallic surface in a setup that has never been exploited for this kind of measurement before.

In the first part of the article, we demonstrate that under the right condition no dissipative forces are found down to the tens of nanometers range even though our force sensitivity is well beyond the required level. In the second part, we discuss why our setup allows us to observe frequency fluctuations while others report dissipation and thus the existence of a shear force.

Among the experimental difficulties in studying and understanding dissipation of a mechanical oscillator near a surface, three are of prime importance: (i) Measure accurately the probe-substrate distance. The use of piezo actuators is the easiest and most common method to control the probe surface distance. However the absolute distance between probe and sample is difficult to determine exactly, relying usually on the sudden blocking of the cantilever vibration.[9,14] Some authors have used the tunneling effect[7,10] even though the zero was set arbitrarily for a given tunnel current. (ii) Determine the real motion of the tip with respect to the surface that in principle can have different movement directions, modes, and irregular vibrations. (iii) Detect any possible contamination build-up between the tip and the surface that can lead to varying distances, friction and uncontrollable local effects.

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To overcome all these difficulties, we have used a nanowire resonator manipulated close to the surface of interest in a transmission electron microscope (TEM). It was then possible to measure the resonator *Q*-factor while observing directly the distance and the tip motion to determine whether or not there was contamination or contact.

An in-house sample holder was designed to position the probe in vicinity of the surface during continuous observation (see Figure 1a). The voltage difference between the surface and the probe is controlled by a Keithley 6517A voltage generator. A three axis motion with a coarse control using micrometric screws and a fine control using piezo actuators allows perfect alignment between probe and surface as well as approaching the probe close to the surface. The three piezo actuators that move the probe are controlled with a three channel Agilent N6700B power supply. An independent piezoelectric actuator is used to excite the resonator. The probe excitation frequency is scanned with an Agilent N5181A function generator. Vibration amplitudes and tip−surface distances are measured by TEM imaging.

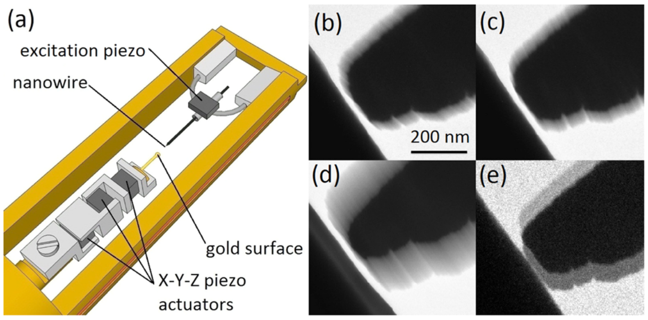

Figure 1. Experimental setup. (a) Sketch of the TEM sample holder used in this study. (b,c) TEM images of the two nanowire vibration polarizations visible after sample holder rotation (see text). (d) Large vibration 25 nm from the surface showing the parallelism achieved on one polarization. (e) Jump to contact from video recorded at 1.5 V difference between tip and surface (see text).

All measurements were made without any closed-loop feedback control of the distance. This gives a great flexibility to set the initial distance with TEM control whatever the other parameters are. The main drawback is thermal drift that was reduced by thermally isolating the whole electron microscope. However, a 0.1 °C variation on the 10 cm shaft translated into 100 nm drift of the tip−surface distance. Lateral drifts are smaller but are still in the 10 nm scale and they reduce the possibility to stay at the very same surface position for a long time. As we could not guaranty thermal stability beyond 0.1 °C, TEM images were recorded before and after measurements and data were discarded if tip to surface relative distance varied by more than 10%.

The pressure in the TEM was $\sim 2 \times 10^{-7}$ Torr, which rules out any viscosity based dissipation. The extremity of a 0.5 mm gold wire melted into a 1 mm ball was used as a surface. We chose a 25 μm long silicon carbide (SiC) nanowire coated with 40 nm of Au−Pd (80−20% at) as a resonator. The total diameter, including coating, was 230 nm and its effective mass $\sim 3 \times 10^{-15}$kg.

Figure 2 displays the mechanical resonances of the nanowire recorded far from the surface by scanning the excitation frequency and recording the vibration amplitude. A nanowire is often presented as a cylinder, however its cross-section is not perfectly circular but often slightly elliptical that yields two resonance frequencies along two perpendicular vibration directions, or polarizations. We find the first eigenfrequencies to be 199 and 201.5 kHz for its two respective polarizations and the *Q*-factor to be ∼1000 for both polarizations. We checked that the vibration amplitudes used in this study for our resonator are sufficiently small to be well within the linear vibration regime (i.e., harmonic oscillator) and that the two polarizations were sufficiently separated to avoid coupling and circular motion.[15]

A careful alignment of the probe and gold ball in the two transverse directions was first carried out in order to be able to observe the distances all the way down to contact with the surface (see details in Supporting Information). The tip was positioned in order to have at least one of its polarizations oscillating parallel to the ball surface (Figure 1d). The other polarization might not have been oscillating as parallel to the surface as we only had a two-dimensional image in TEM. For the measurements, the whole TEM sample holder was rotated

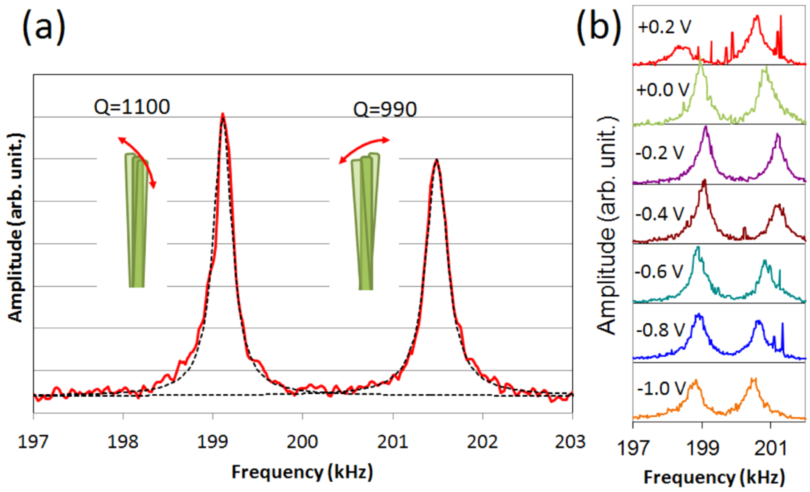

Figure 2. (a) Nanowire resonance peaks recorded far from the surface. The two perpendicular vibrations have comparable *Q*-factors (∼1000). (b) Influence of a DC voltage between tip and surface on the nanowire resonance frequency. The downshift is a signature of electrostatic pull-in and shows that we need about −0.3 ± 0.1 V to compensate the workfunction difference.

in order to observe the two polarizations (Figure 1b,c), the oscillation amplitude was set to about 60 nm and the driving frequency was scanned as the probe was positioned at various distances.

Experimentally, a first required step consists in annihilating electrostatic coupling between the tip and the surface. As this coupling requires a nonzero voltage difference between the two objects, one might think that grounding tip and surface will reduce this coupling to zero. A key point is that contact voltage differences exist even for zero-applied voltage difference. They can arise from workfunction differences between the probe and surface that are usually made of different materials. Typical contact voltage differences are several hundreds of millivolts. Note also that even identical materials will exhibit some local voltage variations.[13,16,17]

To set the voltage difference to zero, we use the so-called "pull-in" effect where at low probe/surface distances the probe senses asymmetrical lateral forces, particularly when a voltage difference is applied. The pull-in creates frequency downshifts and small additional lateral movements that are sometimes unstable and which can cause jump-to-contact (see Figure 1e). As electrostatic pull-in will downshift the resonance frequencies of our resonator when close to surface, we searched to minimize the frequency downshift to estimate the contact voltage difference between tip and sample (Figure 2b). This contact voltage difference was found to vary according to the location on surface, but typical values are found to be −0.3 ± 0.1 V on the nanowire with respect to the surface.

Although electrons from the microscope strike and could induce voltage drops along the nanowire, the low nanowire resistance (estimated to ~10 Ω) combined with the weak current emitted by the TEM gun (5 μA, of which a minute fraction hits the nanowire), ohmic potential drops will be negligible. Magnification and brightness were nevertheless kept constant during measurement, so that voltage differences induced by the electron exposure, if any, were at most a constant offset included in the compensation. Once the contact voltage difference is compensated, the resonator amplitude should stay constant while approaching the surface in the absence of a dissipative force.

To test this, the nanowire was excited at its resonance frequency far from the surface and the vibration amplitude was then measured while approaching the surface. To minimize thermal drift issues, the tip−surface approach was recorded on video while keeping a constant excitation frequency and driving amplitude. The surface approach time can thus be reduced to a few seconds and vibration amplitudes are extracted from the video record. In this configuration, both dissipation or frequency shift will translate into amplitude reduction. The drawback of this method is that without a full resonance spectrum, one cannot know which of the two creates the amplitude reduction. On the other hand, if the amplitude does stay constant, there is no added dissipation nor frequency shift. Video imaging also reduces the frame exposition time and while the tip to surface distance can still be accurately determined, determination of the vibration amplitude suffers from blurring. We apply a conservative 15 nm error bar to our amplitude measurements.

Figure 3 shows the tip vibration amplitude versus distance extracted from some of these videos available in Supporting Information. The free tip motion amplitude (i.e., far from the surface) was set to 160 ± 15 nm, and the resonator was still in the linear mechanical oscillator regime (i.e., harmonic oscillator). The important result is that with the best voltage compensation and within the video record accuracy neither increased dissipation nor frequency shifts were observed while the tip−surface distance was reduced down to 10 nm. An amplitude decrease below 10 nm was detected and it can be attributed either to remaining local workfunction differences that will shift the resonator frequency (complete annulation of contact voltage effects for all distances remains impossible as the workfunction has been shown to be distance dependent[16,17]) or to short-range noncontact friction forces. For the later, van der Waals and phononic friction are expected to play a role within a few nanometers of the surface.[8]

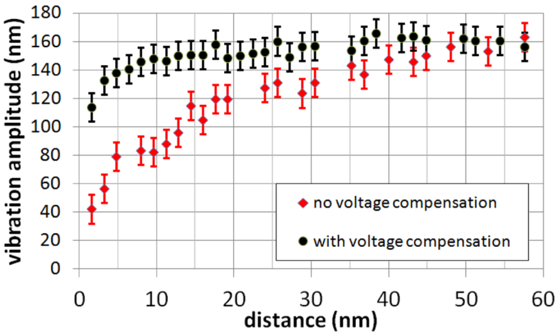

Figure 3. Amplitude of vibration recorded for a fixed excitation frequency while approaching the surface for different voltage difference. Without voltage compensation, a feedback on the amplitude will stop the approach ~20 nm from surface with a 25% amplitude reduction set-point. With voltage compensation and identical set-point, this distance will reduced to few nanometers.

In a SNOM system, feedback control often simply measures the vibration amplitude and working distances are set for a given amplitude reduction. Without voltage compensation (i.e., surface and tip grounded), we found that if a 25% vibration amplitude reduction was chosen as set point for an hypothetical feedback control, our probe-surface working distance would be ~20 nm, which is a typical value found in the SNOM literature.[18] Note that with the best compensation voltage the 25% amplitude reduction is reached for a distance of ~2 nm.

These measurements appear to rule out the existence of a long-range dissipative force in vacuum acting over 10−60 nm range when the experiment is properly set up. However before reaching a definitive conclusion, it is necessary to be sure that our setup has the required sensitivity. To calculate the sensitivity of our system, we use the general relationship $2\Gamma = \omega_0/Q$ to extract the free damping rate of our oscillator far from the surface, $\Gamma_{\mathrm{free}} \sim 500\ \mathrm{s}^{-1}$, use the effective mass for a cantilever rod and compute its free mass collision rate $M\Gamma_{\mathrm{free}} \sim 1.5 \times 10^{-12}\ \mathrm{kg \cdot s^{-1}}$. Even if we use a conservative 25% amplitude reduction as a minimum for dissipation detection, this would translate into an increase of the damping rate Γ_{visq} of $\sim 125\ \mathrm{s}^{-1}$ that yields an increase of mass collision rate with the tip induced by viscosity of $\gamma_{\mathrm{visq}} = M\Gamma_{\mathrm{visq}} \sim 4 \times 10^{-13}\ \mathrm{kg \cdot s^{-1}}$. Using our nanowire tip velocity, the force sensitivity is determined to be in the ~20 fN range with our setup.

According to several other authors,[5,6] the shear-force is expected to be in the nanonewtons range in vacuum although it will depends on tip area and velocity. Karrai[7] gives a more detailed analysis proposing a "third body hypothesis" and reports a viscosity of $\mu \sim 25$ Pa·s from which we can compute that our nanowire will be exposed to a viscous force of ~10 μN at 10 nm from the surface which is orders of magnitude higher than our oscillator force sensitivity.

Note that our increase of damping rate and mass collision rate calculated above for 25% vibration amplitude reduction (i.e., 2 nm from the surface with best voltage compensation) are comparable to those reported by Kisiel et al.[9] at similar distance and are consistent with electrostatic dissipation.

The above observation demonstrates that if we compensate for electrostatic difference, no dissipative force is observed down to distances as small as 10 nm from surface in vacuum. The problem is now to explain reported effects at larger distances (e.g., 15−20 nm[1,7,13]). We propose that probe resonance frequency fluctuations could be at the origin of observed resonance peak broadening. In a recent Letter, Dykman et al.[19] studied the effect of random frequency jumps on the resonance curve of an oscillator. According to their calculation, resonance spectra will be broaden if $W/\Gamma > 1$ with W being the jump rate between frequency and Γ the oscillator decay rate. However, they also predicted that the resonance will split into several peaks if $W/\Gamma < 1$ providing than the characteristic frequency jump Δ is larger than intrinsic resonance width. In other words, the oscillator frequency fluctuations W have to be slower than the oscillator decay time Γ (gives enough time to the oscillator to change its frequency) and the frequency change Δ has to be larger than the peak resonance width (to be visible as a separate peak).

Figure 4 shows several spectra recorded in the 10 nm range with different applied voltages. Frequency offset due to electrostatic pull-in that appears when applying voltage has been subtracted for the sake of comparison. Curves have been offset but the amplitudes were not renormalized. We concentrate on the polarization for which the vibration was parallel to the surface. The lower spectrum (Figure 4c) corresponds to the best compensation voltage (surface grounded and −0.4 V applied to the tip. The local voltage difference was then ∼0 ± 50 mV, see Figure 2b). Several peaks are clearly present in the spectrum, mostly downshifted. Within the framework of Dykman's theory we can consider that our tip, close to the surface, sees a randomly fluctuating potential that changes randomly the nanowire frequency resonance. On the one hand, to resolve fluctuations in our spectra W/Γ should be smaller than unity, which implies $2W(Q/\omega_0) < 1$, which implies $W < 1000$ Hz. On the other hand, W should by higher than our frequency scan rate (12.5 Hz) otherwise several well-defined peaks could not have been observed. This gives an order of magnitude of ∼100 Hz for W. We can also estimate the characteristic frequency change Δ to be in the range 100−500 Hz from the resolved peaks in Figure 4. Note that the individual resolved peaks present a Q-factor of ∼1000, close to the value far from the surface that confirms the absence of dissipative force.

It is important to identify the source of these local fluctuations. At 300 K, no large surface diffusion is expected, so the local topography should be relatively stable. However, even though our tip and surface are metallic, small charges can be trapped in absorbed molecules or other surface defects and we propose that fluctuations of such charges are responsible for the observed effects.

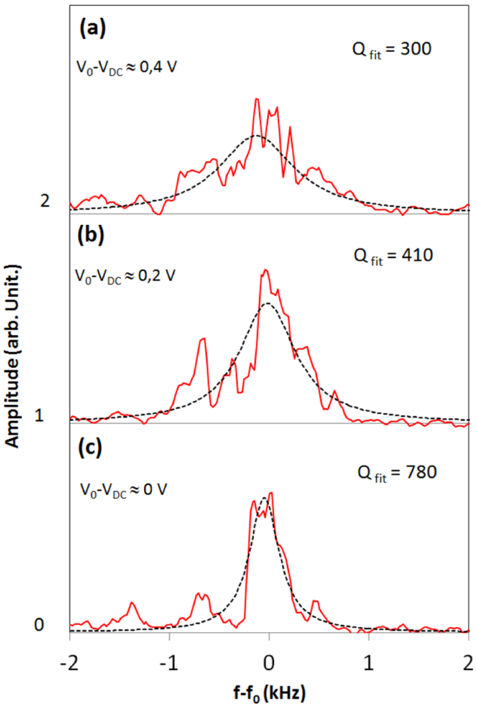

Figure 4. Resonance curves recorded in the 10 nm range with various applied voltages V_{DC}. The workfunction difference is noted V_0. Even with no voltage difference (work function difference compensated, i.e., $V_0 - V_{DC} \sim 0$), several peaks which we attribute to frequency fluctuations are observed where only one is expected. The individual resolved peaks have Q-factors of ∼1000 showing the absence of additional dissipation. Increasing the voltage difference increases the fluctuation jump rate and the number of peaks. Failure to resolve these frequency fluctuations might explain reported shear-force measurements in vacuum.

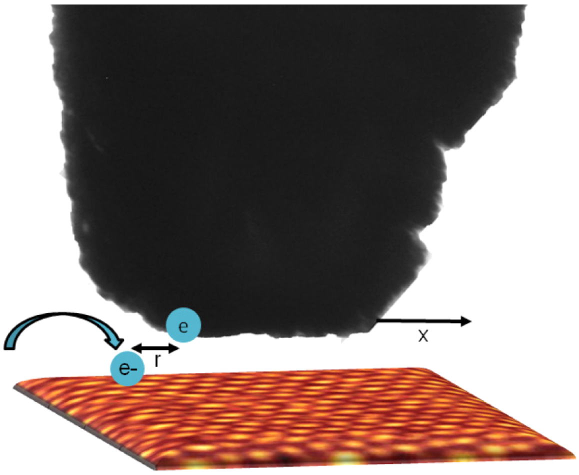

Figure 5. Model use to evaluate the influence on the probe resonance of fluctuating charges on surface. The relative frequency variation can be estimated from the spring constant variation. The effect of a single electron on surface is consistent with the measured frequency fluctuation (see text).

To evaluate the effect of such local surface charge fluctuations, we estimate the influence of a single elementary charge on the tip resonant frequency. The schematic used for this calculation is shown in Figure 5. A single charge on the surface will create an image charge on the tip. We consider that charges cannot freely move on the surface. As the tip radius is spatially limited, the image charge cannot always be above the surface charge and eventually will have a lateral offset. Let r be the distance between the fluctuating charge and its induced surface charge. As discussed above, this distance will typically be a fraction of the tip lateral displacement (i.e., a fraction of 60 nm for spectra reported in Figure 4). The spring constant of the system is extracted from the second derivative of the total energy, which is the sum of elastic and electrostatic energies

$$E_p = \frac{m\omega_0^2(x - x_{eq})^2}{2} - \frac{e^2}{4\pi\varepsilon_0(r + x)}$$

$$k = \frac{d^2E_p}{dx^2}\Big|_{x=0} = m\omega_0^2 - \frac{e^2}{2\pi\varepsilon_0 r^3}$$

The relative frequency change induced by this spring constant change can then be computed $\Delta(f)/f = -(e^2)/(4\pi\varepsilon_0 r^3 m\omega_0^2)$. Using $30 < r < 40$ nm yields relative change $-0.08\% < \Delta(f)/f < -0.18\%$ that translates in our case into resonance frequency jumps between 350 and 150 Hz. This compares well with the observed range of frequency changes. Note also that as this charge will exert a lateral force, frequency is expected to be downshifted in agreement with our observations.

To go further, it is useful to estimate how many electrons can fluctuate in and out of our system at room temperature. Simply considering the capacitance of our system $C = (\varepsilon_0 S)/d$, we find that with our geometry ($d = 10$ nm and $S \approx 4 \times 10^{-14}$ m^2), fluctuations of 10 electrons are expected, which would translate into kilohertz range frequency fluctuations.

To examine this the voltage difference was then increased starting from a configuration (Figure 4c) where tip and surface have identical electric potential (surface is grounded and −0.4 V is applied to the tip, workfunction difference compensated). Figure 4b,a shows resonance spectra recorded with controlled voltages difference between tip and surface of 0.2 (surface grounded and −0.2 V applied to the tip) and 0.4 V (surface is grounded and 0 V is applied to the tip). The latter case corresponds to workfunction difference.

Increasing the voltage difference increases the static charge in the capacitor but the amplitude of the charge fluctuation should still be comparable. However, recall that our tip is oscillating near the surface and even if this oscillation is perfectly parallel to the surface, the added static charges will interact with and try to move the charges related to the fluctuations. In other words, an increase of voltage difference will not increase the fluctuation amplitude (controlled by the amount of fluctuating charges), but will increase the fluctuation rate (the jump attempts frequency). This matches fairly well with the observations on Figure 4; an increase of the number of small ill-resolved peaks (increase of frequency jump rate W) that lead to a broad resonance peak without larger frequency jumps Δ. Note that the asymmetry toward frequency downshift is still present as expected. Note also that failure to resolve these frequency fluctuations will lead to a broad resonance. This fact, associated with the usual practice of equating a decrease in the *Q*-factor with an increase in the dissipation, leads to introduction of dissipative shear-force. To illustrate this point, we fit our data with a single Lorentzian and extract what we called an apparent *Q*-factor. This apparent *Q*-factor decreases as we increase the voltage difference and thus the fluctuation rate. Note that for Figure 4a, which corresponds to the absence of contact voltage compensation, the apparent *Q*-factor is only 300. This shows that even a purely conservative force, like electron-induced frequency shift presented in this paper, can lead to an increase of resonance peak-width if the related fluctuations are temporally not resolved.

It is interesting now to review other setups in the framework of Dykman's theory. Of course, one has to reconsider W, Δ, and Γ for each experiment. Γ can usually be extracted from the articles (cantilever *Q*-factor and resonant frequency). Δ also depends on the cantilever, however, within our model it scales like $1/(m\omega_0)$ and even if the amount of fluctuating charges and topological detail may change this number, we can make some rough estimation. Estimating W is impossible as this parameter depends on temperature, surface (contamination, annealing, etc.), and probe characteristics (tip area and shape). The total system capacitance should also be known to evaluate the maximum allowed charge fluctuation and then the possible range of the frequency changes. Recall that the multipeak feature will be observed if $W/\Gamma < 1$ and if the frequency jump Δ is larger that resonance width.

Tuning forks often used for shear force measurement[20] have lower resonance frequencies (32 kHz) and higher *Q* (~10^4 in vacuum) than our setup. However, their huge effective mass (10^{-6} kg) places Δ in the microhertz range for single charge fluctuations (compared to 3 Hz for the resonance width). As we do not know the tip−surface capacitance of these systems, it is not possible to give further quantitative estimations, but it would clearly be difficult to observe resonance splitting with these large effective mass. An interesting and pertinent paper concerns results reported by Stipe et al.[13] on a similar system. Their cantilever physical properties ($Q \sim 17\,000$, $\omega \sim 4000$ Hz, and $m = 5 \times 10^{-13}$ kg) yields $\Delta \sim 90$ Hz, which is much larger than the resonance width (about 0.2 Hz). Although most of their reported results are made 2 nm from the surface were other effects can occur, they do report dissipation 20 nm from surface. They note that their resonance peak is no longer Lorentzian with asymmetry toward lower frequency, but they consider it comes from an oscillator anharmonicity. The authors also report that an increase in temperature decreases the resonator *Q*-factor, which is consistent with thermal activation of charge fluctuations. They also observe a decrease of *Q*-factor while increasing voltage difference that is consistent with our result. The absence of multipeak in their spectra means that within our analysis their W has to be larger than ~3 Hz.

It is noteworthy to point out that contrary to what is usually sought for in NEMS, a high *Q*-factor (small Γ) is not necessarily an advantage. The combination of low effective mass, high resonant frequency, and moderate *Q*-factor give nanowires some interesting properties for fluctuation measurements like demonstrated here or for ultimate transient force sensors.[21]

Note that using a dielectric surface would not change our conclusion. Work on charge fluctuations in dielectrics[22,23] have also shown that even conservative electrostatic coupling will introduce peak broadening that could be interpreted as a *Q*-factor reduction. Also for highly resistive tips or surfaces, electrostatic dissipation might play a role in resonance damping.

In summary, we have found that in the 10−60 nm range there is no such a thing as a shear-force in vacuum in the sense of the nonconservative friction force often referred to in the literature. Interactions between the probe tip and the surface are dominated by capacitive coupling. For a metallic cantilever and surface, ohmic dissipation is negligible and frequency fluctuations are likely to be the origin of the cantilever resonance frequency broadening. The failure to resolve these fluctuations and the usual practice of equating a decrease in the

Q-factor with an increase in the dissipation have let the community to propose dissipative forces that actually does not exist.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Additional information and figures. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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