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The influence of instrumental parameters on the adhesion force in a flat-on-rough contact geometry

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ABSTRACT

We have used atomic force microscopy (AFM) to measure the snap-off forces between a micron sized *flat* silicon AFM tip and a *rough* Si(0 0 1) surface. The current paper is a natural continuation of our previous paper (Çolak et al., 2014), dealing with snap-off forces between an *identical flat* tip and *flat* Si(0 0 1). Within the applied experimental parameter windows we observed no dependence of the snap-off forces on the applied normal loads (3–18 μN) and residence times (0.5–35 s) for the current *flat-on-rough* geometry as was the case for the former *flat-on-flat* geometry. The snap-off forces were found to increase with relative humidity in both geometries. As in the case of the *flat-on-flat* contact geometry, a strong dependence of the snap-off forces on the retraction speed of the tip was observed. Here, we find a strong *decrease* of the snap-off forces with increasing tip speed, especially at low velocities in the range 40–1000 nm/s for the *flat-on-rough* geometry. This is in contrast with the *flat-on-flat* geometry, where we found a strong *increase* of the snap-off force with increasing tip speed. These observations are explained in terms of a cross-over of the importance of capillary forces and viscous forces. We suggest that the relative importance of both forces can be checked via variation of the tip speed.

1. Introduction

The current paper is a logical continuation of Ref. [1], where we reported a thorough investigation of the influence of parameters as applied normal load, relative humidity, residence time, tip size and tip retraction speed on the snap-off forces between a *flat* AFM tip and a *flat* Si(0 0 1) surface. The relevant results are summarized briefly here: for the flat tip with radius 0.9 μm we found no dependence of the snap-off force (or adhesion force) on the applied external load (between about 4 and 20 μN) demonstrating the elastic nature of the tip surface interaction. No influence of the residence time (0.4–40 s) of the tip on the surface on the snap-off forces was observed either. We found that for the flat surface and tip diameter of 0.9 and 1.8 μm the snap-off forces scale with the contact area. A very strong dependence of the snap-off force on the tip retraction speed was observed. The forces increase markedly with the piezo velocity, which was ascribed to dominant contributions of viscous forces to the snap-off force. In all cases the obtained snap-off force was found to be highly reproducible, without differences between the result for initial contact between the virginal surface and the tip and subsequent contacts at the same position. Finally, the snap-off forces increase with relative humidity.

In this paper we focus on the interaction with identical *flat* tips and a *roughened* Si(0 0 1) surface with an rms roughness of 13.7 nm. The rms roughness of the used tip is estimated at about 1.5 nm. Note that all surfaces mentioned in both papers are hydrophilic. In order to prevent significant overlap between the current and the former paper we will refer to Ref. [1] in many instances. In the current paper we also made sure that we stayed within the elastic tip – surface interaction regime. We find that the snap-off force is virtually independent from the tip size. The most important result is that again the snap-off forces depend strongly on the tip retraction speed but, in contrast to the flat-on-flat geometry, the forces *decrease* with *increasing* speed.

2. Experimental details

The sample used in the current series of experiments is a rough Si(0 0 1) wafer that was roughened with an anisotropic wet chemical etchant. Before etching, the sample was cleaned in an ultrasonic bath of methanol for 15 min. Subsequently, the Si wafer was placed in a piranha solution with a 3:1 volume ratio mixture of H_2SO_4 and H_2O_2 for 30 min. After these two cleaning steps, the Si wafer was rinsed in deionized (DI) water, and dried using a dry N_2 flow. Finally,

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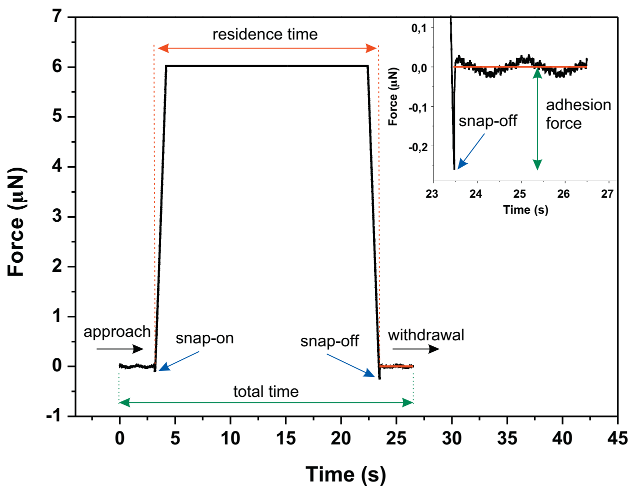

Fig. 1. The force experienced by the tip as a function of time for 165 nm/s piezo speed at 60% RH condition. The total time, and residence time were set at 26.5 and 20 s respectively. The inset shows the zoomed image of the snap-off part of the curve used for adhesion force calculation. The red line in the inset represents the fitted line to the centre of the peak-to-peak distance of the oscillation in the baseline. For this curve, the ratio of the peak-to-peak amplitude of the oscillation signal to the adhesion force is found around 0.2. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

the sample was chemically roughened in a solution of NH_4OH/H_2O (1/5) at 80 °C [2,3] for 5 min, again rinsed in DI water and dried using a flow of dry N_2. Subsequent to the etching process, the sample roughness was recorded, with a silicon tip (Nanosensors, PPP-NCL-20) in tapping mode AFM. The resulting rms value was determined at 13.7 nm.

The used tip has been described in detail in the experimental section of Ref. [1]. Again the plan-parallel tip – (macroscopic) surface alignment was made by maximizing the adhesion force. However, in the current *flat-on-rough* geometry the measured snap-off forces were rather insensitive to the exact alignment of the tip surface and the average macroscopic surface. The humidity control was realized in the way described in Ref. [1], including the definition of the 'dry' condition.

In order to measure the adhesion force, the cantilever is moved in the vertical direction towards the sample, and then withdrawn. During the approach/retract cycle of the cantilever, the cantilever deflects due to the forces acting between the tip and the substrate. We recorded the deflections of the cantilever with the Molecular Imaging (MI) software. In the study of the temporal behaviour it is important that the response is not limited by the AFM electronics. Therefore, we used a break-out box to monitor the normal displacement signal and piezo voltage with a HP digital oscilloscope. The high resolution time basis of the oscilloscope was used to measure the response of the cantilever deflection including snap-in, and snap-off. At the end, deflections of the cantilever are recorded as a function of time as shown in detail in Fig. 1. When recording such force–distance curves, at the noncontact region where the cantilever is far from the surface, the interaction forces are virtually zero. Hence, the baseline value of noncontact part of the curves should be flat. However, artefacts such as wave-like periodic oscillations can be observed at the zero deflection points due to the interference of the laser light reflecting on the surface with the reflection on the tip. When we observed this artefact, as shown in the inset of Fig. 1, a flat line attributed to the centre of the peak-to-peak distance of the oscillation signal as being the zero baseline (red line in the inset of Fig. 1). When a noisy signal is observed instead of a clear wave-like oscillation, then a flat line fitted through the average of the data points as the zero deflection points. Finally, the adhesion force experienced by the tip is extracted from the positions between the maximum deflection point of the cantilever just before it snaps-off and the zero deflection point at the freestanding position of the cantilever.

The residence time is defined by the time lapse between snap-in and snap-off, while the total time is defined as the time to record a complete force-distance curve, including the time that the tip does not make any contact with the substrate. We also refer to Ref. [1] for the definition of the tip speed.

We do stress once more that each of the data points in the figures below has been obtained from averaging the results of twenty consecutive force distance curves recorded with the AFM. We did not observe any significant difference between the first measurement (on the virgin surface) and subsequent ones. The reported error is the standard deviation obtained from this series of measurements.

3. Results and discussion

We have chosen to present here our full data set. However, we do so without an extensive discussion as long as the results are basically similar to those reported and described in Ref. [1]. An exception is made for the variation of the externally applied load, since not the applied load is decisive, but rather the resulting pressure and we are here dealing with relatively small contact areas. The second and last exception is made for the results for varying tip speeds. In this case very strong dependences on the tip speed are observed and these are not only quantitatively, but even qualitatively different from those previously obtained for the *flat-on-flat* geometry.

3.1. Impact of externally applied load on the flat AFM tip

The asperities on surfaces can be elastically, viscoelastically or plastically deformed during contact by surface forces [4]. An increase of the adhesion force [5,6] was observed when the solid was deformed plastically. In order to obtain with an AFM accurate values of the adhesion force for the intrinsic surface, it is necessary to establish a purely elastic contact between the substrate and the tip. Thus, there is a distinct need to study the behaviour of the adhesion force as a function of the externally applied load for, in the current case, the flat AFM tip and the rough Si(0 0 1) surface. Note that the pressures applied here for the *flat-on-rough* geometry are substantially higher than those in the *flat-on-flat* geometry figuring in Ref. [1].

Fig. 2 shows the adhesion force between the flat AFM tip with a diameter of ~0.9 μm and the rough Si(0 0 1) (with 13.7 nm rms) at various applied normal loads. The measurement was done for applied loads between 3 and 18 μN, with the piezo velocity and the residence time set at 2 μm/s and 5 s, respectively. We refer for a characteristic force–distance curve to Fig. 4 of Ref. [7]. As is immediately obvious from the figure there is no discernible influence of the applied load on the adhesion force. This applies for both relative humidity values of 0% and 40%. The error bar for the adhesion force obtained for each individual load level is quite large. As mentioned above, the size of the error bar is the standard deviation of 20 consecutively measured adhesion force values. Fig. 3(a) shows an example of such a data set of 20 measured adhesion forces for each of two applied loads of 4.5 μN, and 17.5 μN, respectively, obtained at 0% RH. In Fig. 3(b), a similar set of 20 measured adhesion forces is shown at 40% RH for two applied loads of 3.4 μN and 16 μN, respectively. As seen from Fig. 3(a) and (b), there is no tendency for a systematic trend between the individual measurements. This implies that the measurements have been performed well within the elastic regime. Note that each first data point applies for the virgin surface. The variation in the data set is random, i.e. no evidence

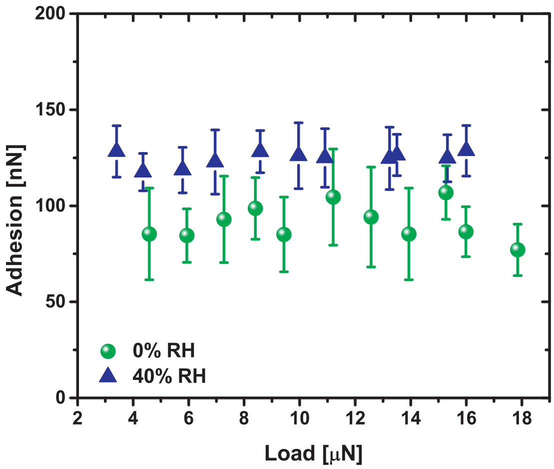

Fig. 2. The adhesion force as a function of an externally applied normal load for a rough Si(0 0 1) surface measured with a flat AFM tip with a diameter of 0.9 μm. The piezo speed was 2.0 μm/s and the residence time was set at 5 s for the two given values of the relative humidity.

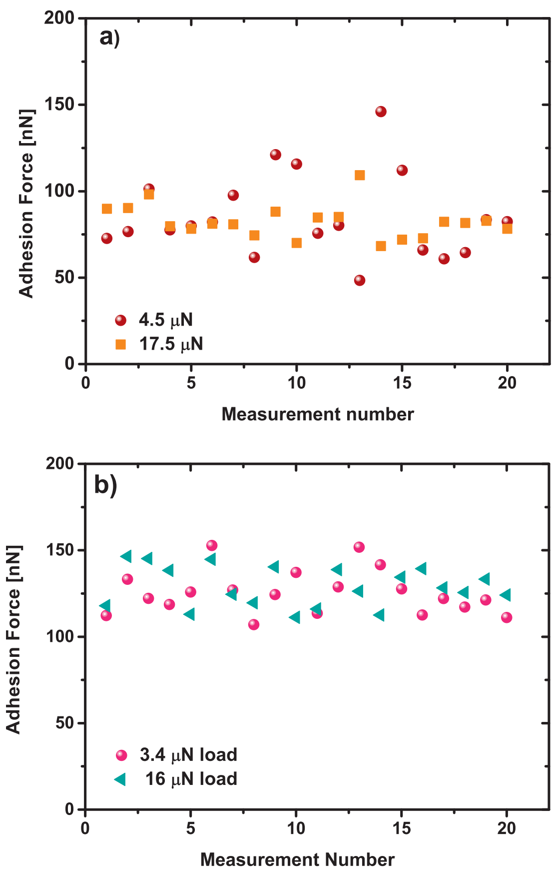

Fig. 3. The measured adhesion forces with 20 consecutive force–distance curves at (a) 0%RH for applied loads of 4.5 μN (brown circles), and 17.5 μN (orange squares) (b) 40%RH for applied loads of 3.4 μN (pink circles), and 16 μN (green triangles). The data numbered 1 have been obtained at the first contact with the virgin surface. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

for building up of any "history" when repeatedly measuring at the same position has been obtained. The relative variation of the measured adhesion force is considerably higher than that obtained for probing a flat Si(0 0 1) surface with a flat tip [1]. The most important reason for the decreased relative accuracy of the measured adhesion force. Compared to the flat-on-flat geometry the absolute value

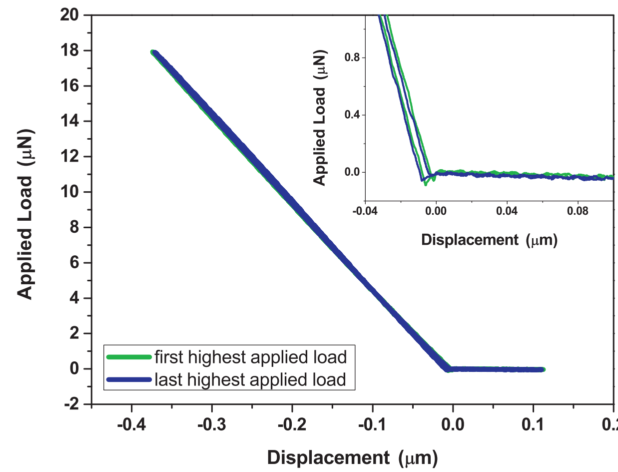

Fig. 4. Force–distance curves obtained for the highest applied load of 16 μN and a relative humidity of 40%. The curves have been measured at an identical position: green results for a virgin location and blue: at the end of a series. The inset shows their critical part with higher resolution. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

of the adhesion force is 15–20 times lower for the presently studied rough surface. This not only supports the much smaller effective contact area in the latter case, but also stresses that uncertainties in the tip position far out of contact, which is used as a reference point for zero force, are much more important in a relative sense: a ripple (as a result of interference between the laser beam reflected from the cantilever and random parts in the scan tube) in the reference level would cause relative uncertainties in the adhesion force which are multiplied 15–20 times when compared to the flat surface case. We believe that this latter factor accounts for the current relatively large error bars (see Fig. 4).

Summarizing, we do not observe any indication of a dependence of the measured adhesion force on the applied load. This important observation indicates that under the given conditions plastic deformation of neither the tip nor the contacting asperities at the sample takes place. Therefore, the measured adhesion force is believed to represent the real adhesion force between the rough surface and the used flat tip. Assuming that the adhesion force scales with the contact area [1] the integrated contact area in the present situation amounts to about 0.04 μm^2. This estimate allows us to calculate the applied pressure at 0.5 GPa, i.e. one order of magnitude below the plasticitiy limit [8]. Note that nanoscale material is even more persistent against plastic deformation [9]. Therefore, the lack of dependence on the load as shown in Fig. 1 is nicely within the expectations. Although we are still on the safe side in this experiment, the elasticity test is crucial. Ferreira et al. [10] found for SiC evidence for an irreversible change of the apex of their 15 nm tip when applying pressure above 0.2 GPa. In Fig. 1, the adhesion force increases with the increase of humidity as pointed out before (Ref. [7]). This is attributed to the larger diameter of the meniscus bridges formed between apex of the rough substrate and the tip at higher humidities.

Fig. 3 illustrates and demonstrates the absence of any "history" effect on the obtained snap off forces. See also Ref. [1]. These results further corroborate the absence of plastic deformations as discussed further above.

By far the most compelling evidence for the absence of plastic deformations is given in Fig. 4. It shows two force–distance curves measured with the highest applied load, first on approach and departure from a virgin location and second at the same position after having measured a series of 23 force distance curves in between. Obviously the occurrence of plastic deformation causes hysteresis between the approach and retract curves [11]. The total

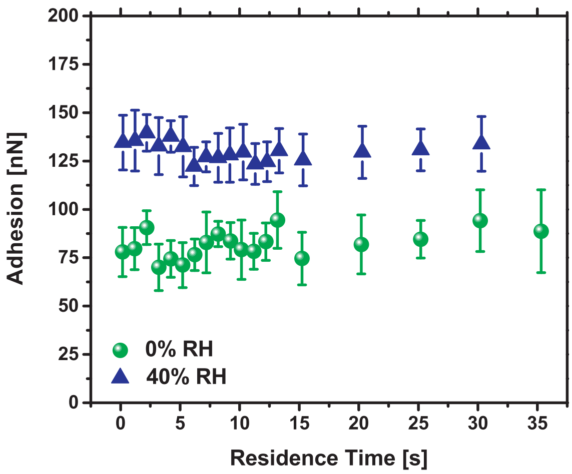

Fig. 5. The residence time dependent adhesion force of a rough Si(0 0 1). Piezo speed, and applied normal load were set at 2.0 μm/s and 6 μN, respectively. The tip diameter was 0.9 μm.

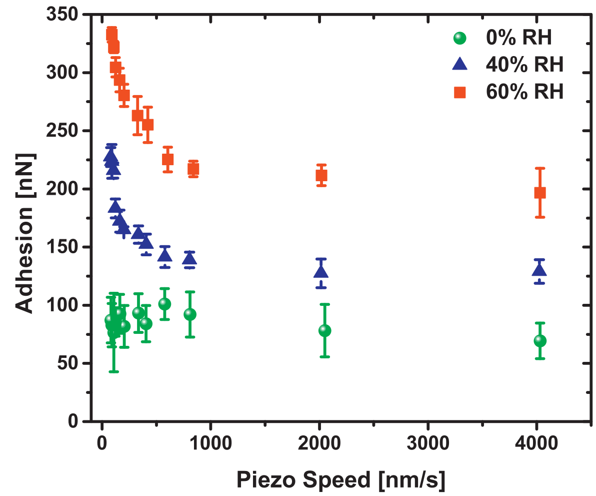

Fig. 6. Influence of the flat AFM tip's retraction speed on the adhesion force at different relative humidity while the tip diameter is 0.9 μm. The load was 5 μN and the residence time was set at 20 s.

absence of any history effect on the force distance curves demonstrates the complete lack of any plastic deformation.

3.2. Influence of residence time

At ambient conditions, when two hydrophilic bodies are brought in sufficiently close contact to each other, menisci as pendular rings are formed around the (near-) contacting asperities. As a result the adhesion forces increase and it has reported that the adhesion force between the surface and the probing AFM-tip depends (in some cases slightly) on the tip's residence time on the surface [12–15]. We found no evidence for this feature in the flat-on-flat geometry [1], but the current geometry may lead to a different result.

The influence of the residence time of a flat AFM tip (diameter of 0.9 μm) on the adhesion force of a rough Si(0 0 1) is shown in Fig. 5. The measurements were done for residence times varying from 0.5 to 35 s, while the applied load and the piezo velocity were kept constant at 6 μN, and 2.0 μm/s, respectively. Fig. 5 shows that, within the experimental errors, the adhesion force is independent of the actual value of the residence time. The values obtained at the virgin substrate are identical to those obtained subsequently at the same position. The relative humidity was again found to have a profound influence on the value of the adhesion force. For 0% RH, the adhesion force is around 80 nN, whereas a distinctly higher adhesion force around 125 nN is measured at 40% RH. The latter is a result of the increase of the contact area due to the condensation of more water between asperities of the rough substrate, and the AFM tip with increasing relative humidity.

The results are in line with those reported in Fig. 5 of Ref. [1] and the corresponding discussion also holds here.

3.3. Influence of the retraction velocity on the adhesion force

When two contacting surfaces are continuously pulled apart, the capillary bridge between them deforms gradually. While the height of menisci increases, their radius decreases during pull-off. After the initial motion, both the capillary force, and viscous forces operate inside the meniscus. During separation, the capillary forces decrease due to the decrease in the meniscus radius, whereas the viscous force, that is the dynamic contribution on the total adhesion force, increases with the separation distance and increasing retraction speed [16]. Either the capillary or the viscous force can be dominant, and this can be accessed by varying the retraction speed of the piezo. In the intermediate case both forces could be very similar. In order to address this issue, the adhesion forces of a rough Si(0 0 1) substrate were measured at different retraction speeds of the piezo.

The retraction velocity of the flat AFM probe has been varied between 80 and 4030 nm/s. For this measurement the load, and the residence time were set fixed at 5 μN and 20 s, respectively. The fixed residence time of 20 s in all measurements is sufficient to exclude possible consequences of a varying incomplete accommodation.

Fig. 6 shows the adhesion force as a function of the retraction velocity of the piezo for three values of the relative humidity: RH = 0, 0.40 and 0.60. We observe hardly any dependence of the adhesion force for zero humidity, RH = 0. This feature has been described already in Ref. [1]. However, for finite humidity, a RH of 40% and a RH of 60%, we observe a strong decay of the adhesion forces with increasing retraction speed. This is true especially for the tip speeds at the lower end of the applied speed window and appears to slow down for higher speeds. Komkov [17] has observed a similar behaviour for the dependence of the snap-off forces between a steel disc and a steel sphere (radius of 2.5 mm) on the separation velocity. He attributed the decrease of the snap-off force to a lowering of the capillary force with increasing velocity. In his studied velocity window (20–80 nm/s) also the most eyecatching changes occur at the lowest velocities.

We intermix the wordings piezo speed and retraction speed to some extent. Where we use piezo speed we actually refer to the commanded velocity. Since one is interested in the tip speed and its influence on the adhesion force rather than in that of the piezo speed we have checked possible ambiguities in tip speed. Obviously, we paid special attention to the time window around the all-important snap-off moment. The point of snap-off is chosen far from the moments the piezo velocity is changed, i.e. at the start and at the end of a force–displacement curve. The deflection signals were measured as a function of time, with a high time resolution, for extreme piezo speeds. After normalization for the adhesion forces the time response was found to be identical. Thus all indications from the force–displacement curves taken within this region indicate a continuous tip speed.

We also extensively investigated whether a change in humidity would affect the dynamic response of the tip. The high time resolution traces taken between 0 and 80% RH did show a large change in adhesion force, but no change in the dynamic response either just before or after the moment of snap-off. We conclude that for the

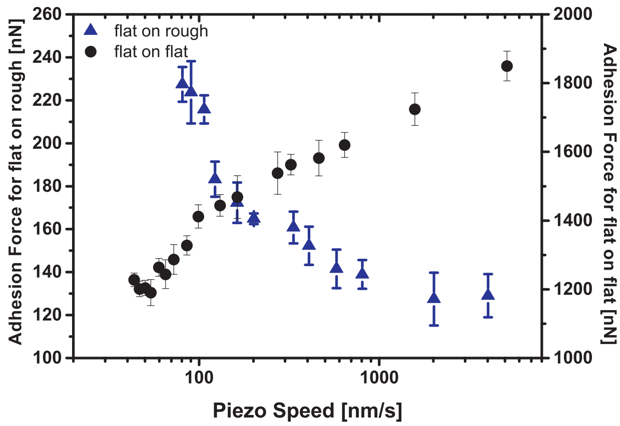

Fig. 7. Influence of the flat AFM tip's retraction speed on the adhesion force at 40% relative humidity for the flat tip – rough surface configuration (blue triangles) and the flat tip – flat surface one (black spheres). The load was 5 and 14 μN, respectively, and the residence time was fixed at 20 s, while the tip diameter is 0.9 μm. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

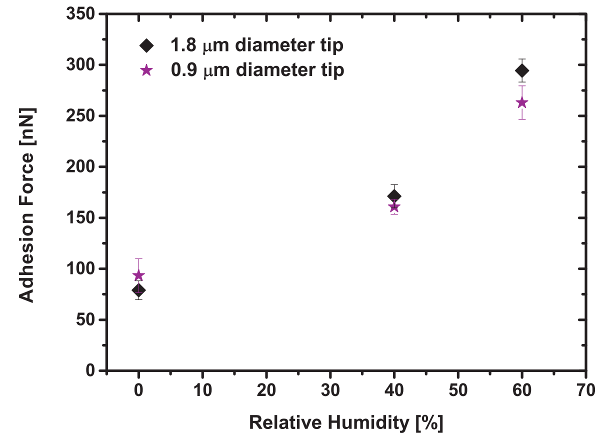

Fig. 8. Comparison of the adhesion forces of flat AFM tips with ∼0.9 μm (purple stars) and ∼1.8 μm (black diamonds) diameters, at various humidity conditions. The retraction speed is 0.3 μm/s for both tips and the applied loads are 5 μN and 14 μN, for small size tip (0.9 μm) and large size tip (1.8 μm), respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

explored conditions the piezo speed is a good measure of the tip speed.

The current results obtained with the flat tip on the rough surface are completely different from those obtained with the same tip and a flat surface [1]. For comparison, we have reproduced in Fig. 7 our measured data for the adhesion force for flat Si(0 0 1) as a function of the retraction speed of the piezo at 40% relative humidity. The corresponding current data from Fig. 6 have been replotted here. In the flat-on-flat and the flat-on-rough cases the tip velocity dependences have been measured at a constant applied loads of 5 and 14 μN, respectively, and a residence time of 20 s. Opposite to the behaviour for the rough surface the adhesion forces *increase* with *increasing* tip speed for the smooth surface at all humidity conditions. We attributed the latter behaviour to a dominant contribution of viscous forces which increase with increasing tip speed [16]. Obviously the viscous force contributions have to fade away at very low speeds. The essential difference between the observed response of the flat and rough surface has to be attributed to the very different response of the water menisci which are of very different size for the flat and rough surfaces. We will use the results published by Cai and Bhushan [16] in our attempt to rationalize our findings for the flat-on-flat and the flat-on-rough geometries.

Cai and Bhushan [16] reported that the adhesion force is the sum of a capillary force and a viscous force. For N menisci between tip and the substrate, they have derived for the adhesion force (see also the sketch in Fig. 7 of Ref. [1]):

$$F_a = \frac{\pi x_n^2 \gamma(\cos\,\theta_1 + \cos\,\theta_2)}{h} + 2\sqrt{N}\pi\gamma x_n \sin\,\theta_{1,2} + \frac{3\pi\eta x_n^4}{4Nt_s}\left(\frac{1}{h_s^2} - \frac{1}{h_0^2}\right) \quad (1)$$

where x_n is the meniscus radius, γ the surface tension of the liquid, h the meniscus height, θ is the contact angle between the liquid and the solid surface, t_s is the time to separate tip and the surface, η is the kinematic viscosity, h_s is the break point of the meniscus, h_0 is the initial meniscus height and subscripts 1 and 2 refer to the lower and upper surfaces, respectively. This equation is normalized to one of the N presumed identical contacts with a characteristic meniscus with contact area πx_n^2.

The first term is related to the Laplace pressure acting on the meniscus area, i.e. πx_n^2, and the second term corresponds to the surface tension integrated over the interface with the contact angle of the liquid on the surfaces (1 and 2) being pulled apart. The third term represents the dynamic force and occurs due to the viscosity of the liquid when separating the tip and substrate. Cai and Bhushan numerically studied the effect of contact angles during separation. Their main conclusion is that contact angles significantly affect the meniscus force, but they have hardly any effect on the viscous force. In general, large contact angles result in a smaller meniscus force.

For a smooth surface the number of menisci, N, is relatively small and therefore the viscous forces play a more prominent role during snap-off. During snap-off the meniscus force will decrease, while the viscous force will increase. The higher the retraction speed, the shorter t_s and thus the larger the viscous force will become. Assuming that the meniscus force is independent of the retraction speed, the increase of the adhesion force with increasing retraction speed is ascribed to the viscous force. The flattening out of the increase of the adhesion force is ascribed to a decrease of the diameter of the contact x_n, if the contact fluid is not able to follow the equilibrium situation. In other words the expanding "cylindrical" part in the centre of the contact will rapidly get a smaller diameter and assumes a larger length. This scenario enables one to understand the results for the flat-on-flat geometry.

For the flat-on-rough geometry the number of assumed identical surface asperities in contact with the flat tip increases. In the simplified case of going from one contact for the flat-on-flat geometry to three contacts for the flat on rough geometry (see also further below) one is able to estimate the change in snap-off force for (extremely) low piezo speeds. For a dominant first term in Eq. (1) the snap off force scales with x_n^2, with x_n differing roughly by a factor of three and one expects a decrease of the snap-off force by an order of magnitude in gross agreement with the measurements: ∼1100 nN and ∼100 nN for the flat-on-flat and the flat-on-rough geometries, respectively. Another glance at Eq. (1) teaches one that the relative importance of the three contributing terms changes. The viscosity contribution in the third term becomes less important, while the interface contribution to the meniscus forces (second term) becomes more important. The sum would still be a less steep but persistent increase of the adhesion force with increasing retraction speed due to a decreasing separation time t_s in the denominator of the viscous term in Eq. (1). This result is at variance with the observations and this discrepancy can naturally be removed by a dominance of the first term in combination with an increasing meniscus height, h, in the denominator of the first term in Eq. (1), with increasing separation rate. Indeed this effect has been calculated by Cai and Bhushan [16] who found a profound increase of *h* with increasing separation rate. The latter effect is then ultimately responsible for the observed decrease of the adhesion force with increasing speed for the flat tip – rough surface system. Our finding that capillary forces dominate the interactions between flat and rough surface are in line with the conclusion reached by Komkov [17].

Quite frequently a distinction is made between “true” or “proper” adhesion forces, defined as Van der Waals forces, and kinetic contributions such as capillary forces and viscous forces. We note that, according to Fig. 7, the kinetic effects are of the order of at least 50%, and thus cannot be seen as just a disturbance of dominating Van der Waals forces. Information on these “proper” adhesion forces might be expected from the zero humidity data. However, zero humidity conditions do not imply that water is absent at the interface. It was reported that even in this case, a water layer confined between the surface and the probing tip actually forms a layered ice film, affecting strongly the interaction between tip and surface [18]. Therefore, access to the pure Van der Waals forces is virtually impossible.

3.4. Influence of the size of the flat tip

Fig. 8 shows the adhesion force of Si(0 0 1) measured with two different flat AFM tips with a diameter of 0.9 μm (purple stars) and 1.8 μm (black diamonds), respectively. For the small size tip (0.9 μm), the measurements were performed at a fixed tip speed of 0.3 μm/s, at a constant applied normal load of 5 μN. For the large size tip (1.8 μm), the measurements were performed at a fixed tip speed of 0.3 μm/s and at a constant applied normal load of 14 μN. As seen from the graph, the value of the adhesion force increases again with increasing RH for both tips. This increase of the adhesion force is a direct consequence of the increase of the water layer thickness with increasing relative humidity. As a result the diameter of the meniscus upon breaking will be higher and both the capillary forces as well as the viscous forces will increase [16]. The data show no significant dependence on the tip size. For this rough surface this can be rationalized if the number of contacts is relatively independent of the tip size and the asperities in contact with the tip are similar. An increase of size of the tip will (theoretically) lead to still three, perhaps different asperities on the surface in contact with the tip. In contrast, the results obtained for the flat-on-(extremely) flat geometry the adhesion forces increase quadratically with the tip diameter [1]. Therefore, we have probed two extreme cases: the flat-on-flat geometry with the quadratic dependence and the flat-on-extremely rough situation with no dependence on tip size at all. It is, therefore, no surprise that the reported results in literature show intermediate dependences, especially when taking into account that the probing AFM-tip has typically a spherical shape [e.g., 12,19,20].

4. Conclusions

We have measured the adhesion force as a function of several experimental parameters, including applied load, residence time, piezo velocity and tip size. We have performed measurements for a flat tip – rough sample geometry, with hydrophilic tip and sample. We have found that the adhesion force of rough Si(0 0 1) (rms roughness is 13.7 nm) does not depend on the applied load on the tip, certifying that our measurements are performed in the elastic regime. Under these circumstances, the obtained adhesion forces do not depend on the residence time of the tip on the substrate and neither on the flat tip size. In the experimental parameter space, for an applied load of 5 μN, and residence time of 20 s, a *decrease* of the adhesion force was measured for rough Si(0 0 1) with an increase of the piezo velocity. This is in clear contrast to the flat surface, where we observed the opposite behaviour, i.e. an *increase* of the adhesion force with increasing piezo velocity. The difference is attributed to a predominant dependence on viscous force for the flat surface and a predominant influence of capillary forces for the rough surface. We propose that the tip velocity dependence can be used to identify the nature of the dominant adhesion forces.

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