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

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ABSTRACT

Atomic force microscopy (AFM) has been used to measure the adhesion force between a *flat* Si(0 0 1) wafer and a micrometer sized *flat* silicon AFM tip. Force–distance curves have been recorded at different setpoints in order to elucidate their individual effect on the derived adhesion force. No dependence of the derived adhesion force on the applied load has been detected, making sure that no plastic changes in the morphology of either tip and/or sample occur. Other setpoints as the residence time of the tip at the substrate, the relative humidity, the size of the tip and the retraction velocity of the tip have been varied systematically. We have found that the adhesion force depends strongly on the velocity of the z-piezo and the tip size while, at least within the 0.5–41 s time window, the residence time does not have any measurable effect on the adhesion force. The time scale of the retraction varies between 0.2 and 25 s. The increase of the adhesion force with increasing retraction speed is ascribed to the viscous force. Finally, the adhesion force increases with increasing relative humidity.

1. Introduction

The notion of adhesion between two surfaces, i.e. that a force is required to separate two materials, is already quite old [cf. 1]. Simple experiments on a macroscopic scale show that area, humidity and separation velocity (hydrodynamic adhesion) are important parameters. The adhesion depends heavily on the roughness of a surface and thus one can manipulate the adhesion by structuring the surface on the micro-, or even the nanoscale. In some cases, e.g. wafer handling in semiconductor industry, the adhesion force needs to be minimized as much as possible in order to allow swifter handling times. In other cases, e.g. a gecko, one prefers to maximize the adhesion force. In order to benefit from the adhesion properties of a substrate in a controlled way, one has to study the adhesion on a length scale down to microns or even to nanometers.

The adhesion force between two surfaces physically originates from Van der Waals forces, electrostatic forces, intermolecular forces, Casimir forces or meniscus forces depending on physical and/or chemical properties of these surfaces [2]. With the use of atomic force microscopy (AFM), it has become possible to measure these interaction forces with a resolution down to nN or even pN using different types of AFM probes [3]. Many studies of adhesion forces have been reported and the influence of several experimental parameters has been examined. Typical variables include the relative humidity, the roughness of the substrate, the applied load on the tip, the size of the tip, the contact time and the retraction speed of the tip. However, many studies [4–14], performed so far, only consider a subset of these parameters while ignoring other possibly relevant, ones. This renders a meaningful comparison between different results difficult.

In this study we systematically varied experimental variables, such as load, contact time, contact area, relative humidity and retraction speed in order to arrive at a complete picture of the parameters that affect the adhesion forces between flat surfaces. We did so for a well-defined system: a smooth surface probed with a flat tip. This approach has two clear benefits: (1) it allows one to compare adhesion force studies and (2) it gives a more complete insight in the physics of adhesion. To the best of our knowledge our present report is the first to break down the problem by investigating each relevant parameter individually. We concentrate on hydrophilic flat surfaces for both tip and sample.

2. Experimental

All the adhesion force measurements were performed with a Molecular Imaging Pico SPM in AFM mode using a flat n-type silicon tip (Nanosensors, PL2-NCLR-10). The circular surface of the flat topped, cone shaped silicon tip has a diameter of $\sim$1.8 μm (see

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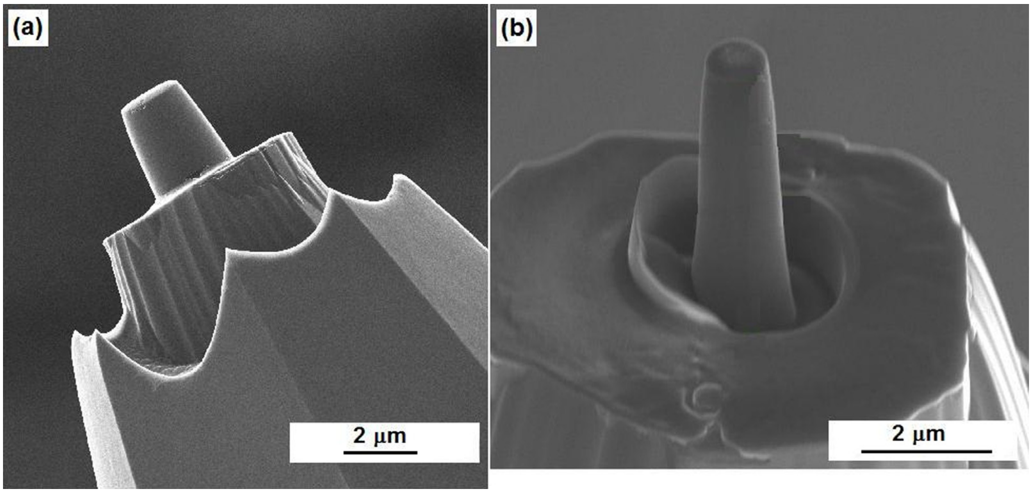

Fig. 1. The flat AFM probe: (a) He ion microscopy (HIM) image of a tip with a diameter of ~1.8 μm and (b) scanning electron microscopy (SEM) image of a FIB modified tip with a diameter of ~0.9 μm.

Fig. 1(a)) and the cantilever has a spring constant of 48 N/m. No traces of plastic deformation were found after a tip was used in several adhesion measurements. In order to measure the effect of the tip size on the adhesion force, the size was decreased to ~0.9 μm (see Fig. 1(b)) using a Focused Ion Beam (FIB). The adhesion force experienced by the tip was extracted from the force–distance curve 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 cantilever has a trapezoidal shape and its spring constant is calculated according to a method put forward in ref. [15].

The sample is a p-type Si(0 0 1) wafer with a 2 nm native oxide layer with a root-mean-square roughness of less than 1 nm. The sample was cleaned in an ultrasonic bath of aceton for 15 min, and then boiled in isopropanol for 5 min at 85 °C, and dried using a dry N_2 flow.

The sample was placed on a sample holder that could be adjusted at different tilt angles. The optimal angle was determined by maximizing the adhesion force [16], which corresponds to a plan parallel configuration. All data reported below have been taken with this optimum alignment, i.e. the tip and the substrate were always perfectly parallel. The resulting alignment was established at a position close to the area of interest and is estimated to be accurate within 1°. Typical measurements are made at the same position for 20 subsequent times and no significant deviation was obtained, including the first data point. The Pico SPM allows to measure in a well-defined humidity atmosphere. The humidity was controlled by adjusting the flow ratio of dry and wet nitrogen streams. The relative humidity (RH) in the chamber was measured with two humidity sensors (SHT 75 Sensirion, Switzerland) located at two different positions in the chamber with a volume of 1 L. After changing the flow ratio, the RH was allowed to reach its equilibrium value by waiting for more than 1 h. The 'dry' condition was obtained after purging with dry nitrogen for at least 12 h. The force–distance curves were collected with an approach/retract cycle of the tip by modulating the cantilever at a frequency well below its resonance frequency. These curves were recorded with the Molecular Imaging (MI) picoview software as well as via a break-out box with a HP digital oscilloscope. The much more stable time basis of the oscilloscope was used to measure the response of the cantilever deflection including snap-in, and snap-off as shown in detail in Fig. 2. The deflection voltage and the drive voltage of the cantilever were recorded simultaneously. Several factors that were reported to have an impact on the adhesion force are elucidated with the force–time curve as depicted in Fig. 2. The residence time is defined as the contact time between sample and tip between the snap-in and snap-off points. 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. During the adhesion measurements, the tip is pushed to the substrate until a maximum normal load value is reached. This load is kept constant until the withdrawal of the tip. We varied the applied load in a window between 4 and 22 μN and analyzed the impact of this variation in load force on the adhesion force. The corresponding maximum pressure is about 30 MPa, i.e. more than two orders of magnitude below the plasticity limit at room temperature. The retraction velocity of the piezo was also varied systematically. It is pointed out here that we kept the displacement distance of the tip constant at about 1 μm and only varied the total time to record the force–distance trace in order to vary the piezo velocity. Note that only the velocity of the piezo can be measured as the actual tip velocity cannot be detected. Of course, when one parameter was varied systematically, all others were kept constant. For insight into the influence of the tip size we used flat tips with diameters of about 1.8 and 0.9 μm. The influence of humidity on the adhesion force was measured at the same piezo velocity and load.

Each reported adhesion force in this work was obtained by measuring 20 consecutive force–distance curves and taking the average of these 20 measurements. This provides a highly accurate value

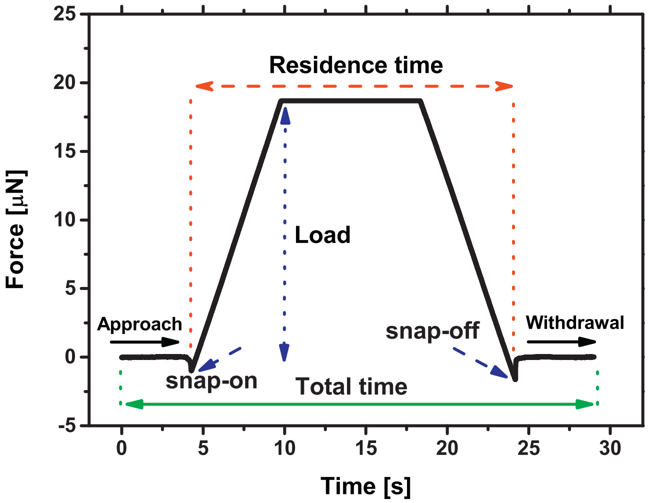

Fig. 2. Scheme of the force experienced by the tip as a function of time.

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