



Effect of nanobubbles on friction forces between hydrophobic surfaces in water

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ABSTRACT

The interaction between hydrophobic surfaces in aqueous solutions is particularly important because it is encountered in many industrial processes. Even though advances in surface science have been tremendous, the nature of the hydrophobic interaction remains one of the greatest challenges to the field. In this work an atomic force microscope (AFM) was used to measure the normal and lateral interactions between a silica bead and a smooth silica substrate hydrophobized by esterification with 1-octanol. The experiments were performed in water and in water after alcohol–water exchange, a method that has been shown to increase the occurrence and size of nanobubbles at the hydrophobic surface and in turn result in a longer range hydrophobic force due to capillary bridge formation. It was found that the alcohol–water exchange had a significant impact on the friction force due to the increased size of the capillary, which increased adhesion.

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1. Introduction

The interaction between hydrophobic surfaces in a liquid medium has been extensively studied due to its importance in fields such as froth flotation, water treatment, and colloidal suspensions. A number of studies, most notably using the atomic force microscope (AFM) and the surface force apparatus (SFA), have found a long-range attractive force between hydrophobic surfaces that cannot be explained by classical colloidal science theories [1–4]. The mechanism of the long-range attractive force between stable homogeneous hydrophobic surfaces is still under debate, but consensus is emerging on a nanobubble bridging mechanism, with the resulting capillary force bringing the surfaces into contact [3, 5–7]. Over the past 15 years much research has focused on the nanobubble bridging mechanism, with the vast majority focusing on the study of force curves [1,2,6–14] and nanobubble tapping mode AFM images [14–21] of a multitude of different surfaces in different fluid environments.

Despite the large number of experimental results that correlate the presence of nanobubbles with the hydrophobic force, there are inconsistent outcomes between different authors from experiments using the same conditions [3,20]. One of the most important discoveries in solving the inconsistencies involves recent investigations of surface pretreatment. Initial work showed that nanobubbles could only be produced on highly oriented pyrolytic graphite (HOPG) after ethanol–water exchange [21–23]. The ethanol–water

exchange process involves flushing liquids through the AFM fluid cell in the following consecutive steps: water–ethanol–water. Additionally, the importance of surface pretreatment has also been clarified by results showing that the population and size of nanobubbles on the hydrophobic surface could be manipulated in a number of ways, including temperature differences between the liquid and surface, and cleaning of the hydrophobic surface with alcohol [24].

Results published by our group have confirmed the effect of surface pretreatment on nanobubbles and how this is reflected in the magnitude of the long-range hydrophobic force [14]. It was found that the use of higher chained alcohols in the alcohol–water exchange process increased the amount of gas present on the hydrophobic surface in the form of nanobubbles, which in turn resulted in an increase in the range of the hydrophobic force. Scanning of the hydrophobic colloidal probe over the surface was found to increase the range of the long-range hydrophobic force due to coalescence of nanobubbles, which formed a larger capillary bridge. Additionally, the long-range hydrophobic force due to nanobubble interaction was investigated successfully using a simple capillary bridge model.

To date, no research has been completed on the effect of nanobubbles on the friction force between hydrophobic surfaces in aqueous conditions. The only significant contribution to the topic of nanobubble-influenced friction is focused on the interaction between hydrophobic and hydrophilic silica [25]. The study of friction forces has a long history, with the first recorded studies undertaken by Leonardo da Vinci and later confirmed by Amontons and Coulomb [26]. In recent history, lateral force microscopy (LFM) of the atomic force apparatus has been proven to be one of the most powerful tools for investigations of friction [27–30].

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Understanding of the friction between hydrophobic materials in aqueous conditions is important in a number of applications including micro- and nanoelectromechanical machines (MEMS and NEMS), shear in particle suspensions, microfluidics, and froth flotation. It is the purpose of this work to determine how friction forces between hydrophobic surfaces in water are influenced by the presence of the long-range hydrophobic force due to a nanoscopic gas capillary bridge.

The system under study here is analogous to research on friction between partially wetted surfaces in air containing nanoscopic liquid bridges, the size of which is controlled by humidity. Study of liquid bridges indicate that friction between partially wetted surfaces in air increases with humidity due to stronger adhesion from a larger bridge [31,32] or the formation of more capillary bridges [33–35], and increasing shear of the larger or more numerous menisci [36–38].

2. Experimental

2.1. Materials

Silicon wafer surfaces terminated with silicon oxide (Silicon Valley Microelectronics, USA) were used to produce flat hydrophobic surfaces by esterification [39–42] with 1-octanol (Merck, Australia). Silica substrates were first cleaned ultrasonically (10 min each in acetone, ethanol, and water), then soaked for 15 min in a 5H₂O:1NH₄OH:1H₂O₂ solution (RCA-SC1) at 75 °C and washed with copious amounts of water, and then dried under a nitrogen stream [43,44]. Water used was freshly purified using a setup consisting of a reverse osmosis RIO's unit and an Ultrapure Academic Milli-Q system (Millipore, USA). NH₄OH was purchased from Lab-Serv (AR grade) and H₂O₂ from Merck (GR grade). The cleaned silica substrate was then immersed in boiling octanol for 4 h under reflux, rendering it hydrophobic. Prior to experimentation the surface was cleaned with acetone (AR grade) and then left in water overnight to hydroxylate [45]. This resulted in a surface with advancing and receding water contact angles of 85° and 60°, respectively, as measured by the sessile drop method using a PAT-1 tensiometer system (SIinterface Technologies, Germany).

Silica spheres with a diameter of 18 μm (Fuso Chemicals Co. Ltd., Japan) were used as spherical surfaces. The silica particles were cleaned in RCA-SC1 solution, washed with copious amounts of water, and dried in a desiccator. The silica particles were then esterified in 1-octanol for 4 h, washed with acetone, and dried in a laminar flow cabinet. The particles were glued onto AFM microfabricated cantilevers (Mikro Masch NSC12/tipless/No Al, 7.5 N/m) using a small amount of epoxy resin (Selleys Araldite Super Strength, Australia), following a procedure available in the literature [46]. Due to the similarities in the sphere and flat plate preparation, it is assumed that the spheres have hydrophobicity similar to that of the flat substrate.

2.2. Methods

All experiments were performed using the colloidal probe technique [46,47] with a Veeco (Santa Barbara, CA) AFM (Nanoscope IV) equipped with a liquid cell. The normal spring constant of the Mikro Masch NSC12/tipless/No Al cantilevers (nominal spring constant $K_{\text{nom}} = 7.5$ N/m) was calibrated using the unloaded resonant frequency method (K_{calc}) [48]. Additionally, the effect the inclination of the cantilever (−15°) has on the spring constant was considered using the method developed by Attard et al. [49] ($K_N = 1.07K_{\text{calc}}$). Normal force measurements were done using the force calibration mode of the Nanoscope Version 5.13 software.

The frictional force measurements were performed using the “friction force” mode of the AFM. In this mode, the colloidal probe

is pressed against the substrate at a constant applied load while the substrate slides perpendicularly underneath the cantilever. The applied load was controlled by adjusting the cantilever deflection set point, but this was found to be erroneous in some situations and the load needed to be recalibrated, as explained later.

The magnitude of the lateral frictional force, F_L , was determined from half of the difference in the lateral force detector signal in one complete scan, V_L , as expressed by the equation [50]

$$F_L = \frac{V_L S_L K_L}{2H}, \quad (1)$$

where S_L is the lateral detector sensitivity, K_L is the lateral spring constant, and H is the distance from the bottom of the sphere to the midpoint of the cantilever.

The detector sensitivity in a lateral direction was determined using the method of Meurk and co-workers [51], and the lateral spring constant, K_L , was determined from the rectangular lever dimensions using the equation [50]

$$K_L = \frac{2K_N L^2}{3(1 + \nu)}, \quad (2)$$

where ν is the Poisson ratio = 0.27 [52], K_N is the normal spring constant, and L is the cantilever length.

All measurements were first performed in DI water (22 °C). The AFM cell was then washed with ethanol or 1-propanol (AR grade at 22 °C) followed by thorough rinsing with water (22 °C). After the alcohol–water exchange, measurements were again performed. In this way, differences in normal and frictional force before and after alcohol–water exchange could be observed.

In each experiment the particle was first scanned over the surface (10 × 10 μm) at very low loads (<20 nN) to coalesce nanobubbles and form a gas capillary at the contact region [14]. After scanning, a number of normal force interactions were performed at a single point at an approach and retract velocity of 2 μm/s and a maximum applied load of 1200 nN. Friction measurements at different applied loads and scanning velocities were then undertaken at a single 1-μm scan line (slow scan axis disabled). A 1-μm scan was found to be sufficient to achieve constant sliding mode friction. It must be noted that the maximum load in the friction and normal force measurements was the same in all experiments. Additionally, to make sure that the capillary did not change between normal and friction measurements, it was important that the normal force measurements were taken at the same position as the friction measurements.

3. Results and discussion

As shown in a previously reported article, alcohol–water exchange dramatically increases the volume of the gas capillary between hydrophobic surfaces due to the coalescence of larger nanobubbles, resulting in a longer-ranged force [14]. By the application of a constant-volume capillary force model to the approach force curves (not shown) the volume and contact radius of the capillary at contact was calculated [14]. As shown in Table 1, the capillary volume and contact radius substantially increase after alcohol–water exchange, with 1-propanol resulting in the larger increase.

The increase in the size of the gas capillary results in stronger adhesion, as shown by the retraction photodiode voltage versus piezo-crystal movement data in Fig. 1. As found by other authors, the capillary bridge resulted in a nonlinear constant compliance region for the retraction force curve [25,53]. As shown in Fig. 1, there is a constant compliance region at positive loads but a deviation from linearity at high negative loads. The nonlinear region of the constant compliance region was considerably reduced if a very stiff cantilever (>14 N/m) was used, and was influenced by

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