Investigation on capillary force of nanoscale water cluster using a Hybrid AFM-MEMS System

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ABSTRACT

The forces that arise from the presence of liquids play an important role in many systems. To investigate capillary force we have developed a measurement platform that combines the force-gradient sensitivity of atomic force microscope (AFM) with the force measuring capability of a micro-electromechanical force sensor. The hybrid AFM–MEMS scheme provides both direct and indirect information of the discontinuous as well as continuous forces associated with the water water cluster. The experimental technique is described along with results showing the advantage of this approach and two simultaneously sensed signals are mutually complimentary for the guarantee of the capillary forces.

Keywords: MEMS, QTF-AFM, Capillary force, elasticity, viscosity

1 INTRODUCTION

The interaction force between surfaces has a major effect on the behavior of the materials, such as nanoparticles, proteins, various bio-molecules, and so on [1, 2]. The phenomenon of stiction influenced by interaction forces impacts the repetitive use of the devices based on the micro/nanoscale materials. Although there have been several studies to discover the origin of the interaction forces including Van der Waals, Casimir, Electrostatic, and capillary forces for the purpose of energy conservation with various systems, it is difficult to solve and still challenging issues to investigate the force effected molecular behaviors. In particular, the role of liquids at the interface is more important to understand the vital phenomena in the field of nanoscience [3]. Study of nanoscale surfaces using surface forces apparatus (SFA), surface force balance (SFB), atomic force microscopy (AFM), scanning tunneling microscopy (STM) typically requires either an UHV or liquid environment to overcome unwilling effects of capillary force between the tip and the sample, which is critical in mapping and imaging. However, the capillary force of naturally confined interfacial water in a nanometer gap in ambient conditions rather gives a critical clue to understand the bio-molecular behavior with the AFM system. There has been a great interest in the capillary condensed water cluster between two surfaces, which is a ubiquitous form of water in nature in ambient condition [4, 5]. Special focus has been made on the stable formation of the nanoscale water cluster as well as the measurement of its viscoelastic and hydrodynamic forces [6,7]. However, one cannot simply integrate the AFM results to obtain the corresponding capillary force, due to its discontinuous behavior associated with formation or rupture of the water cluster. Moreover, it is a great experimental challenge to measure directly the normal force of the meniscus. Therefore, for holistic understanding of the liquid mediated interactions, one requires an independent and direct measurement of the capillary force, which can also justifies the diverse dynamic AFM methods [8]. We demonstrated the strength of the hybrid MEMS-AFM system to exploit the nanoscale phenomena, and also provide additional information on the intrinsic physical quantities of the nanoscale meniscus.

2 EXPERIMENT DETAILS

Non-contact AFM has been well suited for studying the interaction forces with boundary conditions such as Van der Waals force, electrostatic force but not the capillary force

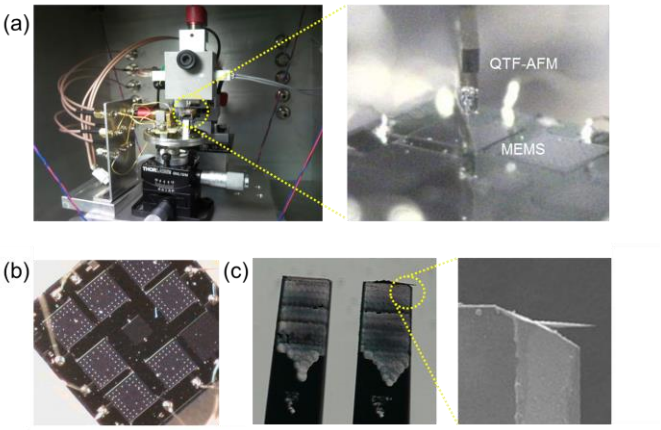

Figure 1: The hybrid MEMS-AFM system :(a) The image shows experimental setup and nanoscale water cluster condensed in between a quartz tip of small amplitude driven QTF-based AFM and a top of the MEMS plate. (b) The image of MEMS force sensor. (c) Optical microscope image and SEM images of Fused quartz tip attached to the bottom of the QTF and operated in tapping mode.

which has no exact boundary conditions. Contact AFM can measure the capillary force except the dynamic properties like damping coefficient and energy dissipation. The enhanced hybrid system, however, can provide both the dynamic properties using the quartz tuning fork (QTF)-based AFM with improvement of previous non-contact AFM and the static properties by directly measuring the force using the MEMS sensor [9]. Especially, the MEMS force sensor provides the additional force information, verifies the QTF measurement, allows accessibility of multiple driving frequencies, and expresses the stress or strain of the confined nanoscale water column. Figure 1(a) shows the demonstrated hybrid MEMS-AFM system that consists of a MEMS force sensor (Fig. 1(b)) and a QTF-based AFM (Fig. 1(c)). Over the last several years, we have worked on pioneering the amplitude modulation (AM) QTF-AFM as a reliable method for studying viscoelasticity unlike traditional approaches, which use the compliant cantilever beam. The QTF provides many useful characteristics [10], such as reasonably high quality factor (~ 5000), simple electrical detection, high stiffness (10^3 ~ 10^4 N/m). Thus it is well suited for measurements of the nanometric water meniscus in ambient condition due to no susceptibility of the pull-in effects resulting from van der Waals interaction that generally occurs in conventional cantilever-based AFM system. We use Quartz tip fabricated by a commercial laser puller (P-2000, Sutter Instruments Co.) to produce tips that are more rigid yet still have radii below 100nm and the AFM tip oscillates perpendicular to the substrate. Assuming small oscillations a linear oscillator model which describes the motion of the QTF can be applied as, [11]

$$m\ddot{z} + b\dot{z} + kz = F\cos\omega t + F_{\mathrm{int}}, \qquad (1)$$

here m is the effective mass of the probe, b the damping coefficient, k the spring constant, ω the driving frequency, F the amplitude of the driving force, and F_{int} the interaction force which is the quantity of interest. An interacted elasticity (k_{int}) and viscosity (b_{int}) can be written as follows,

$$k_{\mathrm{int}} = \frac{F}{A(z)}\sin\theta(z) - (k_{\mathrm{int}} - m\omega^2), \qquad (2)$$

$$b_{\mathrm{int}} = \frac{F}{A(z)\omega}\cos\theta(z) - b, \qquad (3)$$

here A is an amplitude of the QTF output signal. While the QTF sensor provides the information of the viscoelastic properties of the confined nanoscale water meniscus, it is limited to provide the absolute force on position, which is possible to measure with MEMS force sensor [12, 13]. The force sensor used in our experiments is composed of an electrically isolated ground layer and another layer of poly-silicon (500 µm × 500 µm). The plate displacement (Δx) gives information of the interaction forces. The movable top plates are suspended with a distance of 2 µm above the ground layer by four poly-silicon springs connected to each electrode. The detection scheme is a differential capacitance measurement technique used to optimize the detection of the plate's displacement [8, 13]. When the nanometric water meniscus spontaneously forms, it applies an attractive force on the top plate of the MEMS device. This causes the plate to rise up and change the distance between the test capacitor. Equation (4) states that this change in the test capacitance leads to a change in displacement x, which can be measured and converted to the actual force applied by the water column onto the movable MEMS plates.

$$F_{\mathrm{electrostatic}} = \frac{\varepsilon_0 A V_{\mathrm{dc}}^2}{2(d-x)^2}, \qquad (4)$$

$$F_{\mathrm{MEMS}} = k_{\mathrm{MEMS}}\, x(V_{\mathrm{out}}), \qquad (5)$$

here V_{dc} is an applied DC voltage and x is the change in position measured from d. Since the electrostatic force is well known, and the response signal is approximately linear for small changes in x, the output signal (V_{out}) can be converted to MEMS detection force (F_{MEMS}), assuming the spring constant of the device is known.

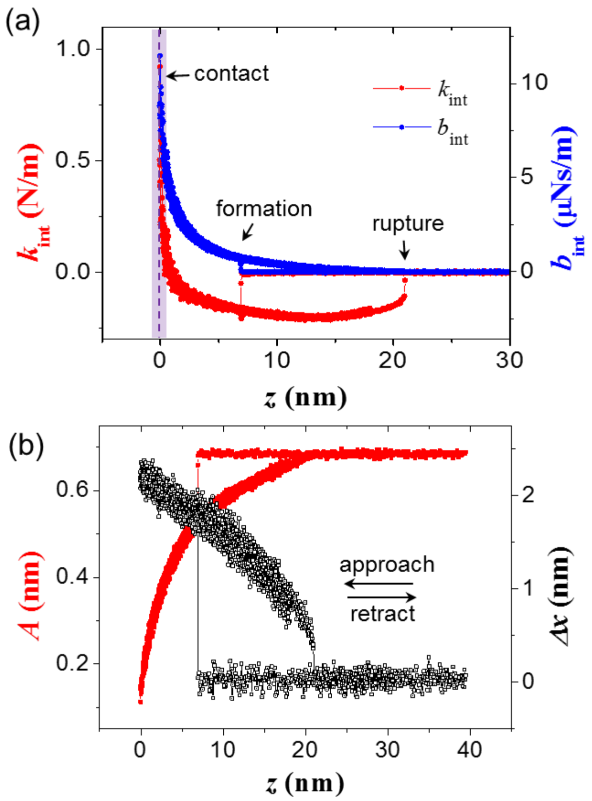

Figure 2: Experimental result from hybrid MEMS-AFM and determination of contact point: (a) The effective elasticity k_{int} and damping coefficient b_{int} of the nanoscale water cluster obtained by the QTF-based AFM and the contact point. (b) The plots are the responses of the AFM and MEMS including the formation of the during the approach/retraction cycle in typical experimental procedure

3 EXPERIMENT RESULTS

Figure 2 represents the experiment results of the approach and retraction curve which is generally used to determine the mechanical properties of nanoscale materials. The contact point is determined as the position where the effective elasticity k_{int} turns into positive showing an abrupt increase in its value as shown in Fig. 2(a). The simultaneous responses of the AFM and MEMS in the basic experimental procedure (Fig. 2(b)). Figure 3 shows the effective viscosity of nanoscale water cluster compared to its bulk value. The viscosity measured for the nanoscale water cluster is about 66 times to 27 times larger than the calculated viscosity based on the bulk model at formation and rupture. We found a large increase in the effective viscosity while no significant changes in viscosity of nanoconfined water in aqueous environment reported at a few nanometer [14]. Therefore we can presume it is affected by the surface tension effect of meniscus rather than an intrinsic viscosity change in water at nanoscale. Figure 4(a) shows the calculated forces curves of the QTF and the MEMS, which are matched each other and this indicates that the two approaches strongly concur with each other. The experimental procedure to measure the capillary force is presented in Fig. 4(b). The amplitude and phase values of QTF-AFM are converted into the force(F_{AFM}) by Eq. (2) and (6) and compared with MEMS results which is important part in this work to confirm the absolute values of the capillary force.

$$F_{\text{AFM}} = \int_{z_r}^{z} dz[-k_{\text{int}}] + F_0 , \tag{6}$$

here Z_r is rupture distance, F_0 is a constant of integration, and k is force gradient in small oscillation. Nevertheless, the force gradient with the amplitude phase values obtained by the AFM itself cannot be simply integrated to the force with a constant of integration unknown due to its discontinuous behavior associated with formation or rupture of the water cluster. While other interaction forces such as Van der Waals, electrostatic force have a boundary condition that is zero at infinity, the capillary force has no such a boundary condition with the discontinuity in amplitude and phase results. Simultaneous measurement presented here provides crucial information for non-contact AFM to obtain the capillary foce besides dynamic characteristics of nanoconfined water cluster. Henceforward, quantitative results in detail will be discussed so that the AFM study could take this advantage for obtaining the absolute capillary force in nanoscale. Having the ability to directly measure the elasticity, viscosity and force of the water column provides many useful features. The first thing is a confirmation of what the quantities is exactly measured by variation of the QTF-AFM. A better understanding of the nanoscale water cluster and the system capavilities is obtained by simultaneously and directly measuring the absolute force with the MEMS and dynamic properties such as elasticity ‘k_{int}’ and viscosity ‘b_{int}’ with the QTF-AFM. Furthermore, the hybrid MEMS-AFM provides conclusive evidence for the reliability of the previous quantitative analysis of the QTF-AFM based on the fact that the interaction force obtained by the QTF-AFM and the MEMS individually is the same.

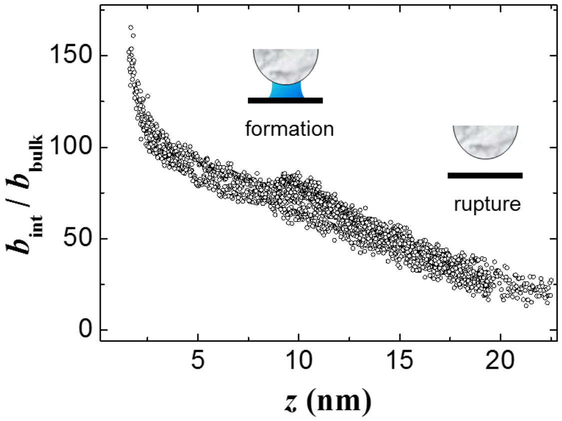

Figure 3: The effective viscosity calculated and compared with its bulk value in entire procedure.

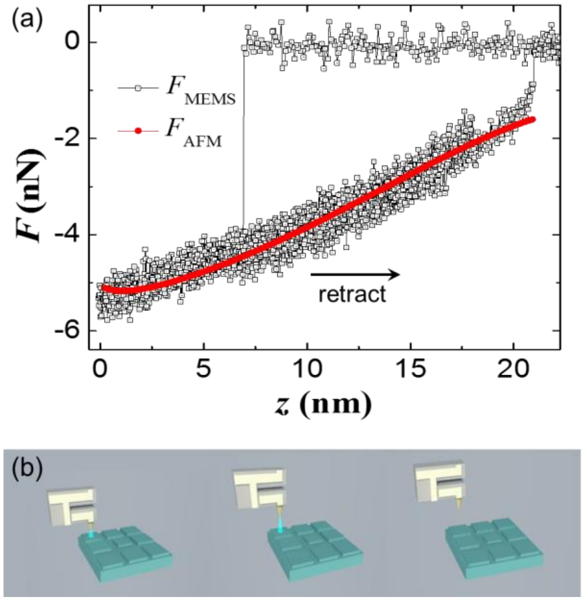

Figure 4: Capillary force obtained by the hybrid MEMS-AFM system. (a) The calculated capillary forces for the QTF-based AFM as well as the directly measured forces by the MEMS force sensor in the entire approach and retraction procedure. Here, the integration constant F_0 for F_{AFM} is set to -1.6 nN (red curve) showing the best fit with the F_{MEMS}. The result shows that the two approaches can work together significantly well. (b) The MEMS plate is retracted and the water meniscus is elongated until it finally breaks free and the capillary foce obtained during this approach/ retract procedure

4 CONCLUSION

We demonstrated the hybrid MEMS-AFM scheme to provide both direct and indirect information of the discontinuous as well as continuous forces associated with the water cluster in nanoscale. In particular, the capillary force obtained in entire length besides the dynamic properties in nanosale. The calculated interaction force for the QTF-based AFM and the diretly measured force by MEMS force sensor are in excellent agreement. Furthermore, the result can provide more information which is crucial for the QTF-based AFM to obtain absolute capillary force of confined water meniscus. We are progressing on the study of qualitative and quantitative analysis for the capillary force in-depth which intensifies the study on the characteristics and behaviors of the confined liquids in such a small systems.

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