

# Energy Dissipated in Tapping Mode Atomic Force Microscopes due to Humidity

Xiaoting Zheng, Yan Sun, Zheng Wei ,

College of Mechanical and Electrical Engineering, Beijing University of Chemical Technology, Beijing 100029, China

Weizheng@mail.buct.edu.cn

**Abstract.** Phase imaging is an important imaging model of atomic force microscopes (AFMs). It provides many sample properties that the height image does not. The phase information reflects the energy dissipation of the probe and sample interactions. Therefore, the energy dissipation between the tip and sample in the tapping mode is of great significance for understanding the mechanism of phase imaging experimentally and theoretically. In this paper, we propose a new method for measuring energy dissipation in tapping mode AFMs. The formation and rupture processes of liquid bridges are studied by this method. Finally, the experimental results are compared with the theoretical model. The comparison shows that the method of experimental measurement and the formation mechanism of liquid bridges in the tapping mode are reliable.

## 1. Introduction

Atomic force microscopes (AFMs) originated from the scanning tunneling microscope (STM), which was invented by Nobel laureates Binnig and Rohrer. The AFM and STM are members of a family called scanning probe microscopes (SPMs). In contrast to an STM, which monitors the electron tunneling current between the tip and the conductive samples, an AFM essentially is essentially a force sensor that feels the interaction between the tip and the samples, which does not to be conductive [1,2]. Since its invention in the 1980s, the powerful capacities of AFMs give them a broad range of use in various areas such as materials science, life science and the semiconductor industry.

Based on vibration theory, the TM (Tapping Mode) AFM phase image mostly depends on energy dissipation between the tip and the sample [3]. Currently, measuring the energy dissipated in TM AFMs is based on the method proposed by Cleveland and coauthors [3,4]. We can measure the rupture energy of the liquid bridge by measuring the force curve, but this method is not suitable for the rupture of the liquid bridge in the tapping mode because the frequency of the force curve measurement is much less than the frequency of the tapping mode. Because of the limitation of the experimental method, the dynamics of the formation and rupture of the liquid bridge between the tip and sample remains an interesting field. There is no conclusive description of this dynamic process [5,6,7].

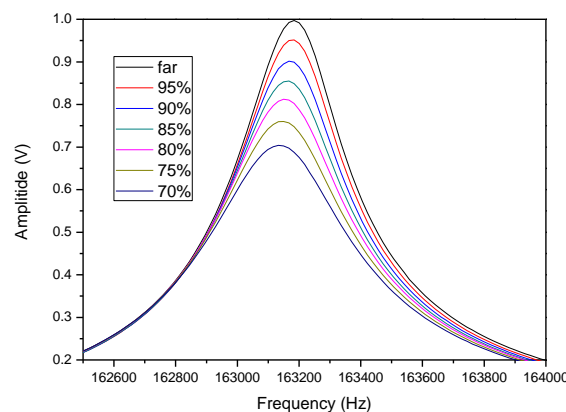
In this paper, we present an indirect method to measure the dissipation energy between the tip and sample in a TM AFM. Meanwhile, the relationship between the rupture energy of the liquid bridge and the relative humidity is theoretically analyzed for the tapping mode. By comparing the experimental data with the theoretical results, we demonstrate the feasibility of this method. We also verify the mechanism of the formation of the liquid bridge in this AFM operation mode.



## 2. Experimental results and discussion

### 2.1. Tuning the cantilever in air

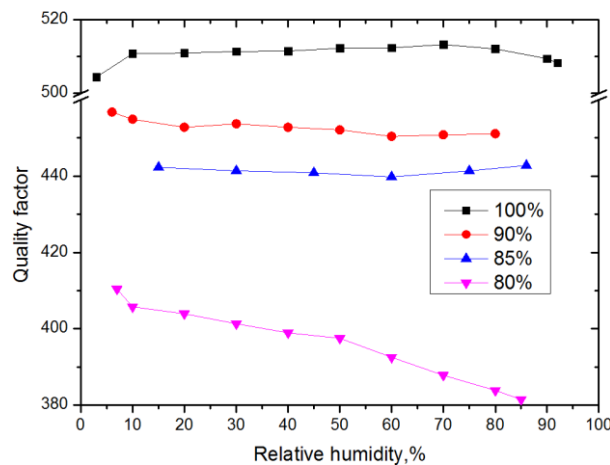
When the AFM tip is far away from the sample, we perform the auto tuning operation on the cantilever. The amplitude-frequency response curve (AFRC) is shown in Figure 1. The natural frequency and damping of the structure can be read from the AFRC. If we turn the front thumb wheel of the AFM scanning head counterclockwise to lower the cantilever towards the sample surface, the amplitude of the AFRC starts to decrease when the tip reaches the area of the tip-sample interaction. The amplitude of the AFRC continuously decreases until the tuning operation fails. In this experiment, we only change the distance between the tip and sample, leaving the excitation voltage applied to the piezoelectric tube unchanged.



**Figure 1.** Tuning test for the AFRC for different distances between the AFM tip and sample at 45% relative humidity. The percentage in the inset key represents the relative positions of tip and sample, while the ratio of the maximum response amplitude to the free vibration amplitude at the resonance point is the percentage of the curve.

### 2.2. Quality factor of the cantilever at different positions and relative humidities

Wet nitrogen gas and dry nitrogen gas are mixed in a long tube, and the mixed gas is introduced into the humidity chamber. In order to achieve the desired humidity, two mass controllers are used to control the flow of the wet and dry gasses. At different locations and relative humidities, we obtain a series of AFRCs. Then, we obtain the corresponding quality factor,  $Q$ . The quality factor at different relative humidities and positions is shown in Figure 2. No matter what the relative humidity is, the quality factor decreases gradually when the tip approaches the sample. However, when the relative position is fixed, and the relative humidity is changed, the change in the quality factor is complicated. The quality factor changes little with humidity at positions of 100%, 90% and 85%, but after the position reaches 80%, the quality factor decreases with increases in relative humidity.



**Figure 2.** Quality factor variation of the cantilever at different positions and different relative humidities. The black curve represents the quality factor,  $Q$ , of the cantilever when the tip is far away from the sample.

### 3. Theory

#### 3.1. The oscillation system of TM AFM

As shown in Figure 2, when the tip approaches the sample so closely that the amplitude of the cantilever decreases to 80% of the free amplitude  $A$ , the quality factor decreases with the increase in relative humidity, unlike the upper three lines representing greater distances. Thus, the effect of the liquid bridge should be taken into consideration in this SDOF system. In the operation of the TM AFM, when the tip contacts the sample, a liquid bridge is formed. As the tip is drawn from the sample, the liquid bridge ruptures. Ignoring the mass of the liquid bridge, the elastic and damping effect of the liquid must be introduced into the SDOF system.

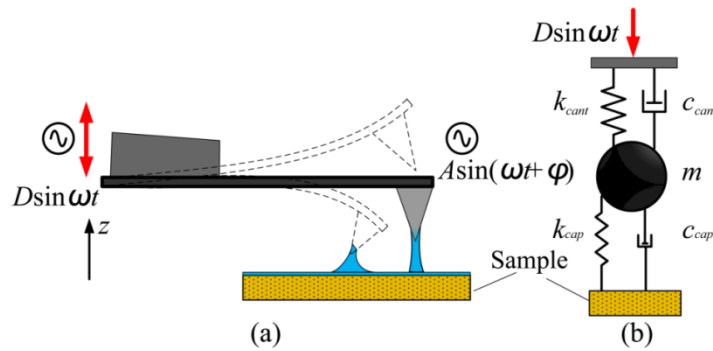
Here, we briefly analyze the vibration ensemble of the cantilever–tip–liquid bridge (CTLB) in the tapping mode. The vibration ensemble model is shown in Figure 3. Now, the dissipation energy of draw of the liquid bridge is taken into account as damping, so the effective quality factor of the SDOF system is

$$Q_{eff} = 2\pi \frac{E_0}{\Delta E_1 + \Delta E_2} \quad (1)$$

Where  $\Delta E_0$  is the energy of the SDOF system,  $\Delta E_1$  the internal dissipation energy in the cantilever,  $\Delta E_2$  is the dissipation energy due to the liquid bridge. Thus,

$$\frac{\Delta E_2}{E_0} = 2\pi \frac{Q_1 - Q_{eff}}{Q_1 Q_{eff}} \quad (2)$$

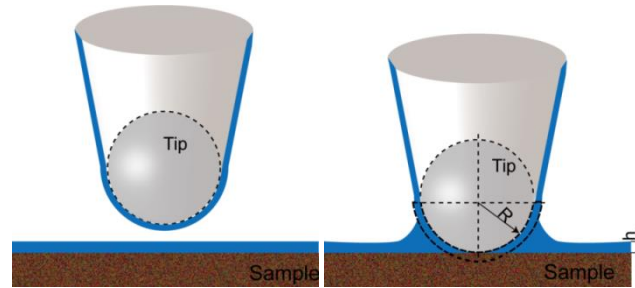
where  $Q_{eff}$  represents the experimental results shown in the 80% line of Figure 2.  $Q_1$  represents the experimental results shown in the upper three lines of Figure 2.



**Figure 3.** The cantilever oscillation ensemble and its simplified SDOF model.

### 3.2. The volume and shape of the liquid bridge in TM AFMs

Numerous experimental results prove that humidity has a strong effect on the vibration behavior of cantilevers. Thus, the liquid bridge must play its role in an oscillatory cycle. The squeezed model of liquid bridges is illustrated by Figure 4.



**Figure 4.** Squeeze model. Owing to the existing thin water film on the surfaces of tip and sample, when the AFM tip approaches to the sample, some water is squeezed out from the contact zone and subsequently forms a liquid bridge. The volume of the squeezed water is the overlapping region shown in the figure. The thickness of the thin water film is  $h$ . Generally, the tip radius  $R \gg h$ .

In equilibrium, the Young-Laplace pressure in the liquid bridge is equal to the disjoining pressure in the liquid film. In addition, the meniscus curvature ( $1/r_a + 1/r_m$ ) is associated with the relative vapour pressure (relative humidity for water) according to the well-known Kelvin equation. Based on the above physical facts, we obtain the thickness of a thin water film over the surface as [5,8]

$$h = \left( \frac{A_H v_m}{6\pi k_B T \ln H_r} \right)^{1/3} \quad (3)$$

### 3.3. Energy dissipation due to the liquid bridge in TM AFMs

In the extension of the liquid bridge, the capillary force, as a non-conservative force, will do the dissipation work. As shown in Figure 5, the capillary force is composed of an attractive force acting on the cross-section of the liquid bridge and a tension force acting on the contour of the cross-section [9].

$$F = F_{Y-L} + F_T \quad (4)$$

The attraction force caused by the Young-Laplace pressure on the cross-section is

$$F_{Y-L} = \pi r_a^2 \gamma \left( \frac{1}{r_a} + \frac{1}{r_m} \right). \quad (5)$$

So, the capillary force due to the liquid bridge is

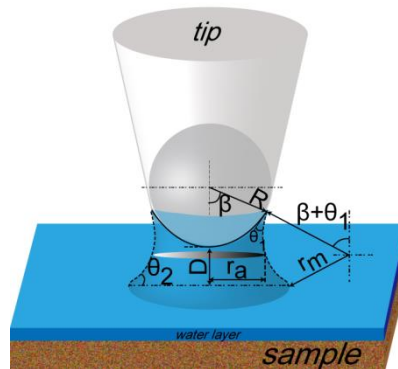
$$F = \pi r_a^2 \gamma \left( \frac{1}{r_a} + \frac{1}{r_m} \right) + 2\pi r_a \gamma. \quad (6)$$

Based on the force–distance curve of AFMs, it appears that the rupture distance is roughly proportional to the cube root of the volume of the liquid bridge [10,11],

$$D_r = \frac{1}{2} \left( 1 + \frac{\theta_1 + \theta_2}{4} \right) V^{1/3}. \quad (7)$$

Then, the dissipation energy for breaking the liquid bridge is

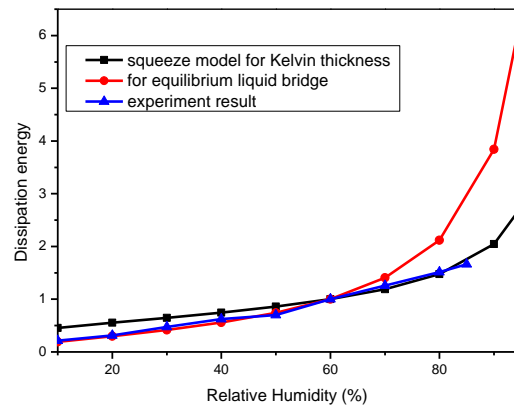
$$\Delta E = \int_0^{D_r} F dD. \quad (8)$$



**Figure 5.** The geometry of the liquid bridge between the AFM tip and samples.  $\theta_1$  and  $\theta_2$  are the contact angles of the tip and sample, respectively.

Eqs.6-8 must be solved simultaneously. Because of the complicated relationship between the geometric parameters, we apply numeric methods. The parameters are assigned as follows:  $\theta_1 = 30^\circ$ ,  $\theta_2 = 8^\circ$ ,  $R = 50\text{nm}$  and  $A_H = 8.7 \times 10^{-21}\text{J}$  [12]. Here, two growth models of liquid bridge are considered, one is the squeeze model, another is the equilibrium model[13].

After normalizing the dissipation energy due to the liquid bridge, the relationship between the energy dissipation and relative humidity is shown in Figure 6. Because the predicted thickness of Eq. (3) has a large deviation from experiments carried out in lower relative humidity, we chose the relative humidity at 60% as the standard for energy normalization. Figure 6 shows that the experimental results agree well with the theoretical results. It proves that the method of tuning the cantilever is feasible for investigating energy dissipation between the tip and sample and is also feasible for investigating the dynamic growth and rupture processes of the liquid bridge.



**Figure 6.** Energy dissipated due to the rupture of the liquid bridge vs. the relative humidity.

#### 4. Conclusion

In conclusion, we have shown a method for measuring the relative energy dissipated in TM AFMs. The quality factor,  $Q$ , will be given in various test conditions by the tuning operation. The energy dissipation in the TM AFM results from analysis of the oscillation system of the TM AFM. Of course, if the stiffness and amplitude of the cantilever are known, we can also measure the absolute dissipated energy. By this method, it has been proven that in the tapping mode, the liquid bridge is mainly formed by the extrusion effect.

#### Acknowledgments

This work was supported by the National Natural Science Foundation of China (NSFC, No. 11572031)

#### References

- [1] Binnig G, Rohrer H. Scanning tunneling microscopy: from birth to adolescence. *Rev Mod Phys.* 1987;59:615-625.
- [2] Binnig G, Rohrer H. In touch with atoms. *Rev Mod Phys.* 1999;72:s324-s330.
- [3] Cleveland JP, Anczykowski B, Schmid AE, et al. Energy dissipation in tapping-mode atomic force microscopy. *Appl Phys Lett.* 1998;72:2613-2615.
- [4] Anczykowski B, Gotsmann B, Fuchs H, et al. How to measure energy dissipation in dynamic mode atomic force microscopy. *Appl Surf Sci.* 1999;140:376-382.
- [5] Wei Z, Sun Y, Ding WX, et al. The formation of liquid bridge in different operating modes of AFM. *Sci China-Phys Mech Astron.* 2016;59:694611.
- [6] Szoszkiewicz R, Riedo E. Nanoscopic friction as a probe of local phase transitions. *Appl Phys Lett.* 2005;87:084502.
- [7] Sahagún E, García-Mochales P, Sacha GM, et al. Energy Dissipation due to Capillary Interactions: Hydrophobicity Maps in Force Microscopy. *Phys Rev Lett.* 2007;98:176106.
- [8] Asay DB, Kim SH. Evolution of the adsorbed water layer structure on silicon oxide at room temperature. *J Phys Chem B.* 2005;109:16760-16763.
- [9] Butt HJ, Kappl M. Normal capillary forces. *Adv Colloid Interfac.* 2009;146:48.
- [10] Lian G, Thornton C, Adams MJ, et al. A Theoretical Study of the Liquid Bridge Forces between Two Rigid Spherical Bodies. *J Colloid Interface Sci.* 1993;161:138-147.
- [11] Lucel S, Robert SA, Riedo E, et al. Volume of a Nanoscale Water Bridge. *Langmuir.* 2006;22:1093-1098.
- [12] Israelachvili JN. *Intermolecular and Surface Forces* 3rd. Pittsburgh: Academic Press; 1985.
- [13] Wei Z, Sun Y, et al. The formation of liquid bridge in different operating modes of AFM, *Sci. China-Phys. Mech. Astron.* 2016 ; 59, 694611.