Development of a combined atomic force microscope with an AT-cut quartz resonator

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Abstract. To study the microscopic mechanism of energy dissipation due to sliding motion, we are developing a combined atomic force microscope (AFM) with a quartz crystal microbalance (QCM) resonator. In this system, the loading force is controlled by AFM, while the energy dissipation is measured by the change in Q value of the quartz crystal. At room temperature, we have checked the energy dissipation for several substrates as a function of loading force. The energy dissipation depends both on substrate material and on oscillation amplitude.

1. Introduction
Physical property of small contacting asperities is a subject of study attracting the interest of many researchers because of the importance to macroscopic and microscopic applications. Characterization of nanoscale contacting asperities have been performed by means of nanoindentation, atomic force microscopy (AFM). It is recognized that a probe tip-quartz crystal microbalance (QCM) has large potential as a sensitive probe of the physical properties [1, 2, 3]. In 1991, Sasaki et al. combined a scanning tunneling microscope (STM) with an AT-cut quartz resonator and reported a simultaneous image of STM surface topographies and a frequency shift of an AT-cut quartz resonator for gold thin films.

To study the microscopic mechanism of energy dissipation due to sliding motion, we have started to develop a combined atomic force microscope (AFM) with an AT-cut quartz crystal resonator which can operate even under low temperature condition. In this paper, we explain our combined AFM-QCM system, and also report preliminary results of the energy dissipation for several substrates as a function of loading force at room temperature.

2. The AFM-QCM system
Figure 1 shows a schematic diagram of our AFM-QCM combined system. The loading force is controlled by AFM, while the energy dissipation is measured by the change in Q value of the quartz crystal. The advantage of this system is to measure directly the energy dissipation due to sliding motion by changing the magnitude of displacement in the nanometer scale and the loading force. A detail description of this system will be given below.
2.1. AFM
For the AFM system, we use a Si self-detecting cantilever (NPX1CTP003, SII) of a spring constant of 4 N/m as a sensor of the loading force, which can operate easily even under low temperature condition [4]. The typical radius of the probe-tip is 20 nm. The loading force acting on the cantilever was measured by the change in piezoresistance of its joint. Its change $\Delta R/R$ is related to the loading force $N$ as [5]

$$\frac{\Delta R}{R} = \pi_L \frac{6LN}{(2a)^2 t^2},$$

where $\pi_L$ is the longitudinal piezoresistive coefficient for Si along the $\langle 001 \rangle$ direction, $LN$ is the torque, and $L$ is the length of the cantilever. Here, $2a$ and $t$ is the width and the thickness of the joint which is a part of the cantilever deformed by the loading force. In our case, $a \approx 2 \mu m$, $t \approx 4 \mu m$ and $L \approx 400 \mu m$. Then, the loading force of 1 nN corresponds to $\Delta R/R \sim 11$ ppm.

The change in resistance is measured using a DC bridge circuit. The reference resistance is also fabricated in the basement of cantilever. The resistance of the cantilever and reference is 600-700 $\Omega$, and the excitation voltage is 1.0 V. The out-of-balance signal is amplified by the preamplifier (LI-75A, NF Corporation). Then, we can easily measured the loading force less than 1 nN.

The position of the substrate is driven by a linear positioner (ANPz101, atocube systems) for the coarse adjustment and a piezo tube actuator for the fine adjustment. The sensitivity of the piezo tube actuator is 1.86 nm/V, and the applied voltage is swept from -100 V to 100 V.

2.2. QCM
The energy dissipation caused by the sliding motion between the probe-tip and the substrate attached on the crystal is related to $\Delta(1/Q)$ of the quartz crystal as

$$\frac{\dot{E}}{E} = \omega_0 \Delta \left( \frac{1}{Q} \right),$$

where $E$ is the oscillation energy of the crystal, and $\omega_0 = 2\pi f_0$ is the resonance angular frequency. In this system, the resonance frequency and amplitude are measured using a transmission circuit. The crystal is placed in series with a coaxial line connecting to a 50 $\Omega$ signal generator and an RF lock-in amplifier. The frequency of the signal generator is controlled in order to keep the in-phase signals zero, and is locked to the resonance frequency. The quadrature signal at this frequency is the resonance amplitude. Here, $\Delta(1/Q)$ is obtained as

$$\Delta \left( \frac{1}{Q} \right) = -\frac{1}{Q} \cdot \frac{\Delta A_q}{A_q},$$
where $A_q$ is the magnitude of the quadrature signal. The oscillation amplitude of the crystal is controlled by the alternating input voltage, and is estimated from the magnitude of the quadrature signal and the oscillating area of the crystal with the substrate. In the present experiments, the input voltage is in the range of 50 to 0.5 mV$_{pp}$, which corresponds typically to the oscillation amplitude of 4 to 0.06 nm [6].

A small thin substrate ( ~ $1 \times 1$ mm$^2$ ) is attached on the electrode of an AT-cut quartz crystal with the resonance frequency of 3.2 MHz. After attaching the substrate, the $Q$ value remains on the order of $10^4$.

3. Energy Dissipation vs Loading Force
We have checked the energy dissipation for several substrates at room temperature as a function of loading force. It was found that the energy dissipation depends both on substrate materials and on oscillation amplitude.

First, we briefly explain the energy dissipation on the assumption of some conditions. In case when the sliding friction between the probe-tip and the substrate is described as the Amontons-Coulomb law $F = \mu'N$, where $N$ is the loading force, $\Delta(1/Q)$ is obtained as

$$\Delta \left( \frac{1}{Q} \right) = \frac{8}{\pi^2} \cdot \frac{\mu'N}{\omega_0 \rho v AS} ,$$

where $\rho$ and $v$ are the density and the sound velocity of the quartz crystal, and $A$ is the oscillation amplitude. Here, $S$ is the oscillating area of the crystal. As seen from this equation, $\Delta(1/Q)$
The loading force decreases almost linearly and the cantilever shows a single sharp jump-out. The attractive force at the jump-out depends on oscillation amplitude. When the amplitude becomes small, this force increases drastically. The middle panel is the change in the frequency $\Delta f/f$ as a function of piezo position. It was found that the frequency does not depend strongly on loading force, and shows a stepwise change at the contact between the probe-tip and the substrate surface. In addition, the frequency clearly shows the oscillation amplitude dependence. In the experimental range of amplitude, it decreases monotonously from 0.8 to 0.05 ppm when the amplitude increases from 0.06 to 4 nm. The bottom panel is the change in $Q$ value as a function of piezo position. $\Delta Q^{-1}$ shows an interesting behavior. On the whole, $\Delta Q^{-1}$ takes the maximum at around an oscillation amplitude of 0.4 nm. At this amplitude, $\Delta Q^{-1}$ shows a stepwise change at the contact and increases gradually with increasing loading force. When the amplitude decreases from this value, $\Delta Q^{-1}$ becomes small and does not depend on loading force. On the other hand, when the amplitude increases to 4 nm, $\Delta Q^{-1}$ becomes small again. This behavior is reproducible regardless of the position of the surface.

**Figure 3.** Piezo position vs (a) the loading force, (b) the resonance frequency, and (c) the $Q$ value when retracting the cantilever from mica surface.
As mentioned above, when the sliding friction obeys the Amontons-Coulomb law, $\Delta Q^{-1}$ is in inverse proportion to the amplitude. On the other hand, in the case of a viscous friction, $\Delta Q^{-1}$ does not depend on amplitude. In contrary to these expectations, $\Delta Q^{-1}$ increases with increasing the amplitude and takes the maximum at 0.4 nm. On the other hand, $\Delta f/f$ decreases monotonously with increasing the amplitude. This means that the sliding motion has a typical length in which the energy dissipation becomes large. At present, the mechanism for the typical length is an open question, but the typical length may be compared with the lattice distance.

Next, we make a short comment on mica surface. Concerning the loading force, the attractive force at the jump-out is small. Furthermore, $\Delta Q^{-1}$ is significant small. In a large oscillation amplitude, we observed that $\Delta f/f$ sometimes changes rapidly during piezo position scan.

4. Summary
We are developing AFM combined with an AT-cut quartz resonator. In this system, the loading force is controlled by AFM, while the energy dissipation is measured by the change in $Q$ value of the crystal. At room temperature, we measured the energy dissipation for several substrates as a function of loading force and oscillation amplitude. For HOPG surface, the energy dissipation does not depend strongly on loading force up to about 150 nN. Concerning the amplitude dependence, $\Delta Q^{-1}$ increases with increasing the amplitude and takes the maximum, while the $\Delta f/f$ decreases monotonously. This means that the sliding motion has a typical length in which the energy dissipation becomes large. At present, the mechanism for the typical length is an open question.

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