Electrostatic force microscopy using a quartz tuning fork

Yongho Seo and Wonho Jhe
Center for Near-field Atom-Photon Technology and School of Physics, Seoul National University, Seoul 151-742, Korea

Cheol Seong Hwang
School of Materials Science and Engineering, Seoul National University, Kwanak-ku, Seoul 151-742, Korea

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We demonstrate a nonoptical electrostatic force microscopy based on a quartz tuning fork with 50 nm spatial resolution and 1 pN force sensitivity. We use a tuning fork with a spring constant of 1300 N/m and a Q factor of 3000. A sharpened nickel tip is attached to a prong of the tuning fork as well as electrically connected to the electrode of the prong. By applying a dc bias to the tip, ferroelectric domain patterns are recorded and read out on piezoelectric thin film. © 2002 American Institute of Physics. [DOI: 10.1063/1.1485312]

Since local electrostatic surface charge or potential distributions were imaged by scanning force microscopy,1–3 there have been several experimental efforts to obtain nanoscale electrostatic images of the ferroelectric polarization,4,5 charges in metallic nanoparticles,6 and bound surface charges in semiconductors7 using electrostatic force microscopy (EFM). In conventional EFM, the Si cantilever is employed as a force sensor and optical deflection detection is employed to measure its motion. A microfabricated cantilever has such a small spring constant (0.1–10 N/m) that it has a high force sensitivity (∼ pN). Besides delicate optical alignment, however, the cantilever has some weak points: (i) high resolution EFM is difficult because the dithering amplitude is large (∼ 10 nm) and (ii) laser light should be employed, which is undesirable, particularly for EFM of semiconductors and metallic nanoparticles because the laser light modifies the charge distribution due to the photoionization effect.8

Recently, to overcome these problems, EFM using a piezoresistive cantilever has been developed.9,10 The piezoresistive-cantilever-based EFM uses piezoresistivity of boron-doped Si: when the cantilever is deflected, the resistance of the boron doped layers change. Because no light is needed for deflection measurement, it is possible to operate in dark conditions, avoiding unwanted photoionization effect. However, there are also some disadvantages: (i) the change in resistance ∆R/R is very small (∼ 1 ppm/pnm), which indicates that to measure its frequency shift in EFM, the dithering amplitude of the cantilever should be on the order of 100 nm and (ii) the dissipative power due to Joule heating is serious, particularly in low temperature experiments. For the resistance and applying voltage of ∼ 2 kΩ and ∼ 5 V, respectively, the resultant dissipated power amounts to 10 mW.

In this letter, we demonstrate nonoptical EFM that employs a quartz tuning fork using the piezoelectric effect. Since Guethner et al.11 introduced the quartz tuning fork as a force sensor in atomic force microscopy, there have been several attempts to employ the tuning fork in near-field scanning optical microscopy,12 atomic force microscopy,13–15 magnetic force microscopy,16,17 and acoustic near-field microscopy.18 There are important advantages in tuning-fork-based force microscopy over using the piezoresistive cantilever: a high Q-value (103–104), small oscillation amplitude (∼ 1 nm), and low dissipative power (∼ 1 nW).

A prong of the tuning fork is of 2.2 mm in length, 190 μm in thickness, and 100 μm in width. Note that this geometry of the tuning fork corresponds to a spring constant of k ≈ 1300 N/m. Ni wire with a 150 μm diameter is first etched electrochemically in H3PO4 acid and the wire diameter is reduced to 10 μm. Then the thin wire is inserted into a platinum wire loop containing H3PO4, so that the wire section goes through the electrolyte. The tuning fork is then moved so that the end of Ni wire is attached to the prong of the tuning fork with silver paste. As a result, the Ni wire is connected mechanically as well as electrically to the electrode of the prong. After curing, a dc voltage between the electrode (+) and the platinum wire loop (−) is applied, which makes the etched Ni wire sharp and separated into two parts. All the processes are done with a home-made micro-manipulator.

The length of the protruded tip is ∼ 100 μm and the tip diameter is ∼ 100 nm. Due to additional mass, the resonance frequency of 33 kHz was reduced slightly by 100 Hz. At resonance, the full width at half maximum is about 12 Hz and the corresponding Q value is about 3000, which is 30 times larger than that of a conventional microfabricated cantilever. The minimum detectable capacitance limited by the thermal noise is given by

\[ C_{\text{min}} = \frac{16k_B T B (eA)^2 k}{V^4 Q \omega} \] (1)

where \( k_B \) is the Boltzmann constant, \( T \) the temperature, \( e \) the permittivity, and \( A \) the area of the capacitor. For example, for the tuning-fork-based EFM with a bandwidth of \( B = 10 \text{ Hz} \) at room temperature, the minimum detectable capacitance is \( 2 \times 10^{-20} \text{ F} \).

A dc voltage \( V_{dc} \) and an ac voltage \( V_0 \cos \omega t \) are applied to the electrode that is connected to the tip. The dc voltage is for writing as well as reading out the ferroelectric domains,
whereas the ac voltage is oscillating the tuning fork at its resonance frequency. Consequently, on the other electrode of the tuning fork, current is induced due to piezoelectricity of the tuning fork, which is measured by an current–voltage converter. To measure the shift in frequency associated with the tip approach, phase detection mode is employed. A home-made phase detector consisting of a voltage comparator and an exclusive OR gate is used with a low pass filter (RC=10 ms). The signal to noise ratio is $\approx 10^4$ and the frequency resolution is about 2 mHz.

With the dc bias voltage $V_{dc}$ applied to the tip, the resonance frequency shifts are measured as a function of the distance, as shown in Fig. 1. The sample surface is a clean, grounded Al layer. Note that when the Al layer is biased with the tip grounded, we also obtain the same results. The dithering amplitude was 17 nm the drive voltage of 50 mV. When no bias voltage is applied, there is a shallow dip that indicates van der Waals attractive interaction.$^{3,19}$ As the tip becomes closer to the sample, the frequency increases rapidly. This suggests that repulsive interaction due to contact occurs. When $V_{dc}$ was applied, deeper dips appeared which indicate attractive electrostatic forces given by,$^1$

$$F_e = \frac{1}{2} \frac{\partial C}{\partial z} V_{dc}^2,$$

(2)

where $V_{dc}=1$ V, $C = \varepsilon_0 A / \varepsilon z$, $A \approx 10^{-14} \text{ m}^2$, and $z=5 \text{ nm}$. The force gradient expected, $\partial F_e / \partial z$, is $2 \times 10^{-2} \text{ N/m}$.

On the other hand, when a small force gradient is applied to the tip, the shift in frequency $\Delta f$ is obtained as

$$\Delta f = \frac{1}{2} \frac{f}{k} \frac{\partial F_e}{\partial z},$$

(3)

where $f$ is the resonance frequency ($=33 \text{ kHz}$). With frequency resolution of 2 mHz, the measurable force gradient is $10^{-4} \text{ N/m}$. Taking into consideration that the dithering amplitude of the tuning fork can be reduced to 10 nm, the minimum detectable force difference becomes 1 pN. Note that the electrostatic force between the tip and the sample is typically $\approx 1 \text{ nN}$. The shift in frequency at the dips in Fig. 1, $\Delta f = 0.1 \text{ Hz}$, corresponds to the force gradient $F_e' \approx 1 \times 10^{-2} \text{ N/m}$ calculated from Eq. (3), which is of the same order as that expected from Eq. (2).

In addition to capacitive force, there can be Coulombic force when the sample has spontaneous polarization or is charged by an external potential. Assuming that the surface polarization field of the sample $E_s$ is uniform and that the characteristic at the tip $q_t$ does not modify $E_s$, the Coulombic force can be expressed as $E_s q_t$, where $E_s = \sigma / 2 \varepsilon_0$ and $q_t = C V_{dc}$. Therefore the shift in frequency due the ferroelectric polarization is

$$\Delta f = \frac{1}{4} \frac{f}{\varepsilon_0} \frac{\partial C}{\partial z}.$$

(4)

Figure 2(a) shows an EFM image of a poled PbZr$_{0.5}$Ti$_{0.5}$O$_3$ (PZT) thin film of 250 nm thickness deposited on a Pt layer by the sol–gel process (the Pt layer is grounded during measurements). For a poling process, the bias applied at $V_{dc} = -10$ V is scanned on a $7 \times 7 \mu \text{m}^2$ area of the PZT sample in contact mode. Then a larger area of $15 \times 15 \mu \text{m}^2$ is scanned at a constant gap separation of 50 nm between the tip and the sample and at $V_{dc} = 2$ V. Its maximum contrast corresponds to a frequency shift of $\Delta f = 30 \text{ mHz}$. From Eq. (4), where $z = 300 \text{ nm}$ because $z$ is the distance from the tip end to the grounded Pt layer, the polarization is estimated to be $\sigma = 2 \mu \text{C/cm}^2$, which is a reasonable when one considers that the saturated polarization of the sample measured with a large parallel capacitor was $\approx 10 \mu \text{C/cm}^2$.

Narrow lines were written and read out as shown in Fig. 2(b). The image is $0.9 \times 0.9 \mu \text{m}^2$, the width of the left line is about 150 nm, and its expected spatial resolution is better than 50 nm. This spatical resolution, which is limited by the tip diameter, can be improved further by replacing the tip with a sharper one. In the writing process, the scanning speed does not influence the remanent polarization and thus the patterns written remained for longer than 5 h.

Figure 3 shows EFM images showing various patterns. The scanning areas are 4×4 and 7×7 $\mu \text{m}^2$, respectively.

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**FIG. 1.** Approach curves obtained at bias voltages of 0, 1, and 2 V between the tip and the sample.

**FIG. 2.** EFM images of a PZT film after (a) poling and (b) line drawing. The scanning areas are 15×15 and 0.9×0.9 $\mu \text{m}^2$, respectively.

**FIG. 3.** Some patterns recorded on the PZT sample with scanning areas of 4×4 and 7×7 $\mu \text{m}^2$, respectively.
The check pattern in Fig. 3(a) was written by applying $V_{dc} = -10 \text{ V}$ (dark) and $+10 \text{ V}$ (bright) alternately, although the bright dots are not very clear. We attribute the imperfection to the initial charge distribution or the imprinted polarization of the PZT film.\textsuperscript{21,22} Some characters were also recorded with $V_{dc} = -10 \text{ V}$ and read out with $V_{dc} = 2 \text{ V}$ [Fig. 3(b)]. Note that with 300 nm width, all letters are fully resolved.

In summary, we have demonstrated tuning-fork-based EFM. With 50 nm spatial resolution and 1 pN force sensitivity achieved, such simple EFM has important advantages: (i) it is convenient and inexpensive to manufacture, (ii) it is possible to operate the EFM in a dark environment so that optical deflection detection is not needed, (iii) its low power dissipation allows operation at low stable temperatures.

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