Tapping mode capacitance microscopy

著者	羽根 一博
journal or publication title	Review of Scientific Instruments
volume	68
number	1
page range	120-123
year	1997
URL	http://hdl.handle.net/10097/46699

doi: 10.1063/1.1147749

Tapping mode capacitance microscopy

Kazuya Goto[a] and Kazuhiro Hane
Department of Mechatronics and Precision Engineering, Tohoku University, Sendai 980-77, Japan

(Received 11 September 1996; accepted for publication 17 October 1996)

We present a new technique for microscopic capacitance measurements. Capacitance microscopy is combined with tapping mode force microscopy. The tapping motion is successfully used for the capacitance modulation and also for the tip-sample distance regulation. Furthermore, capacitive and topographic images are simultaneously obtained. The technique was applied to observations of a gratinglike electrode and of a nitride-oxide-silicon structure for a nonvolatile memory. © *1997 American Institute of Physics.* [S0034-6748(97)04101-4]

I. INTRODUCTION

Scanning probe microscopy is a powerful technique for microscopic studies on electrical properties of samples.[1–10] In addition to scanning probe microscopy potentiometry,[1–4] scanning capacitance microscopy[1,2,6–10] is promising for observations and inspections of electronic devices. For the tip-sample capacitance detection, two types of methods are possible. In one type, the electrostatic force related to the tip-sample capacitance is sensed with force microscopy.[1,2,5] In the other type, the tip-sample capacitance is detected with external electric circuits.[6–10]

Recently, dielectric thin films with high permittivity and ferroelectric thin films are receiving increasing attention.[11–13] They play important roles as dielectric layers of capacitors in semiconductor memories. High permittivity films are advantageous for capacitors with small electrode areas. Ferroelectric thin films are used for nonvolatile memories. As the capacitor size is reduced, local properties of the films and dielectrics/semiconductor interfaces become important. The scanning capacitance microscopy can be efficiently used to study them.

In addition to sensitivity, the following aspects are important in scanning capacitance microscopy. First, higher lateral capacitive resolution is desired. Since the capacitance is related to the Coulomb interactions, the surfaces of the probe far from the sample are largely involved in the total tip-sample capacitance. This reduces the lateral capacitive resolution. As a solution to this, the tip-sample distance is modulated. With the modulation, small capacitance changes are obtained, which are determined by more concentrated regions of the tip and the sample.[7,10] In addition, the sensitivity is increased with lock-in detection. Second, it is required that the tip faithfully traces the surface because the capacitive signal varies with the tip-sample distance. In other words, the tip-sample distance regulation is indispensable. Previously, the distance regulation was performed with the aid of dynamic mode noncontact force microscopy.[1] In the noncontact force microscopy, the tip traces a surface of the constant attractive force gradient, which is assumed to reflect the surface topography. In the simultaneous capacitive measurement, however, the force gradient is affected by electrostatic force. If the force is large, it is difficult to maintain the tip-sample distance. This is significant for ferroelectrics. It is known that for ferroelectrics the tip-sample interactions are determined by electrostatic forces due to surface charges of spontaneous polarizations rather than by the tip-sample atomic forces.[14]

The requirements described above are satisfied by adopting the tapping motion to the capacitance detection. This is reasonable for dielectric thin films because current does not flow through the films. In tapping mode force microscopy,[15,16] the tip taps the surface while vibrating near it. The vibration amplitude is kept constant by compensating the surface height differences with a feedback control. The tip-sample distance and the tip vibration amplitude are regulated at the same time. The tip vibration is little affected by potentials near surfaces because a stiff lever is used and the amplitude is large. This is good for stable and accurate capacitance modulation.

In this article, we describe a new version of scanning capacitance microscopy, which works in tapping mode. The instrument is a combination of a conventional tapping mode force microscope and a capacitance microscope. Not only does the tip accurately trace the sample surface, but the capacitive image and the topographic image are also obtained independently and simultaneously. In addition, the tapping mode force microscopy gives as good a topographic resolution as the contact mode force microscopy and is immune from tip-sample friction problems. We believe this is one of the best suited measurement methods for dielectric properties of thin films.

II. PRINCIPLE

Figure 1 shows a schematic diagram of the instrument. The probe is a conductive cantilever with the conductive tip. The cantilever is connected to the capacitance sensor,[6–9] which consists of the LCR resonator and the peak detector. Changes in the tip-sample capacitance are obtained as resonant frequency shifts in the resonator. The cantilever is vibrated for the capacitance modulation, which is aimed at the high lateral resolution and the high sensitivity.[7,10] The capacitive signal from the sensor is then lock-in amplified. The cantilever is oscillated at its resonant frequency with a piezoelectric actuator on its fixed end. The tip is approached to the sample surface and the tip taps the surface. The vibration amplitude is limited by the surface and varies linearly with

---

[a] Research Fellow of the Japan Society for the Promotion of Science.

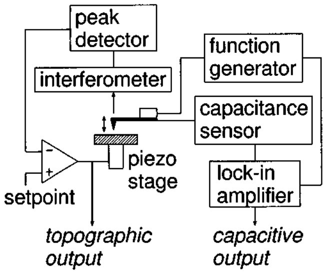

FIG. 1. Schematic diagram of the tapping mode capacitance microscopy system.

the average tip-sample distance. Because the lever is driven at its resonance, the vibration is purely sinusoidal. The tip vibration is detected with a laser interferometer. The amplitude is sensed with a peak detector (rectifier) and converted to a dc signal. While the sample is raster-scanned with a sample stage, the feedback control is performed to maintain the amplitude and at the same time the distance. In the feedback control, sample height differences are compensated by vertical sample displacements by a piezoelectric actuator in the sample stage. The vibration amplitude determines the amplitude of the capacitive modulation. In scanning the sample surface, because the tip faithfully follows the sample surface, stable regulation of the tip-sample distance and the modulation amplitude is possible at each point of the surface. Thus, the capacitance measurement is performed, in which the tip detects the capacitances defined at the surface. In a capacitor, generally, the distance between the two electrodes is equal to the film thickness. In other words, the capacitance is determined by the film thickness, or the surface topography. Therefore, the presented capacitance measurement, in which the film surface is traced, is more reasonable for dielectric thin films than the previous method in which a surface of the constant capacitive signal is traced.[7,10] The capacitive sensitivity and resolution in our method can be as high as that of the previously reported capacitance microscopy, because the principle is almost the same except that the tip taps the surface. With the tapping motion, however, the stability and the accuracy can be higher. Furthermore, the topographic signal is simultaneously obtained together with the capacitive signal. The topographic image is obtained as a result of the feedback control during the scan as in the conventional tapping mode force microscopy. The topographic resolution of the tapping mode force microscopy is as high as that of the contact mode force microscopy. Thus, neither the performance of the capacitance detection nor of the topographic detection is reduced in the simultaneous measurements.

III. EXPERIMENTAL SETUP

The instrument is our homemade scanning force microscope[17] modified for the tapping mode capacitance measurement. The cantilever with the tip was made of a tungsten wire. The free end of the lever was bent at a right angle to work as a tip. The tip was electrochemically etched in a 1 N KOH solution. The levers we used were 50 μm in diameter and typically 2 mm in length. The tip length was 1 mm or less. The resonant frequency of the levers was 2–4 kHz. The quality factor of the levers was more than 100. The spring constant was calculated to be about 50 N/m. A small aluminum foil for the interferometry was glued to the free end of the lever as a reflector. The cantilever is oscillated with a piezoelectric actuator 2 mm thick. The vibration amplitude in our experiment is less than 100 nm.

The capacitance sensor with the LCR resonator (Victor) was originally designed as a capacitive pick-up for a video disk player. The LCR resonator is driven by the ultrahigh frequency (UHF) oscillator. This sensor is equivalent to the ones employed in previous reports.[6–9] It has been reported that the sensitivity of 10^{-19} F can be achieved. The cantilever is connected to the resonator with a lead ~20 mm long. The sample is electrically grounded to the casing of the resonator.

The interferometer for the vibration sensing is a homodyne interferometer.[17] Although the interference signal is a sinusoidal function of the displacement, the tip vibration with amplitudes of less than 100 nm can be sensitively detected. The peak detector for the tip vibration signal is a simple electric circuit of a diode with a resistor and a capacitor.

IV. EXPERIMENT

The tip-sample capacitance signal was measured as a function of the tip-sample distance. The tip-sample distance was changed with the sample stage while the lever is vibrated. The feedback loop was open for this experiment. The sample was a vapor-deposited aluminum film on a glass substrate. Because the aluminum surface is covered with its native oxide, current does not flow between the tip and the sample. The amplitude change of the tip was simultaneously measured.

For a basic performance test of the instrument, a grating-like electrode (2 μm line/space) was observed to obtain a capacitive image and the corresponding topographic image. The electrode was made of a chromium film deposited on a glass substrate. The thickness of the electrode was about 100 nm.

We also measured local capacitance changes due to depletion in a silicon substrate of the metal-nitride-oxide-silicon (MNOS) structure. The MNOS semiconductor memory is one of the most widely used nonvolatile memories.[18] The MNOS system has been successfully applied to the high-density memory using scanning probe microscopy.[9,19] The principle of the memory is as follows. Writing is performed by applying a voltage to the sample with a conductive probe. As a result, charges are trapped in the nitride, creating a depletion layer in the silicon substrate. The depleted region is read by capacitance microscopy. In our experiment, the nitride-oxide-silicon (NOS) sample was prepared as follows. An *n*-type silicon [100] wafer (0.1–0.2 Ω cm) was selected as a substrate. The oxidized surface is a result of a cleaning process of the wafer using HCl. The 50 nm thick nitride was deposited on the oxide film with the

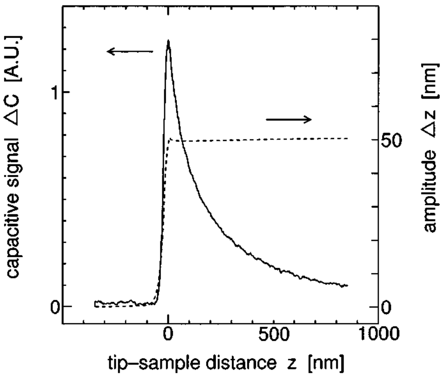

FIG. 2. The capacitive signal ΔC and the tip vibration amplitude Δz as functions of the tip-sample distance z. The tip-sample distance is set zero at the point where the tip starts to touch the surface.

chemical vapor deposition technique. Pulsed voltages of 50 V with 20 μs width were applied to the sample with the tip. The capacitance changes in the written bits were measured with our method and with the capacitance-voltage (C-V) method[8,9] reported previously.

V. RESULTS AND DISCUSSION

Figure 2 shows the capacitance signal ΔC obtained as a function of tip-sample distance z together with the amplitude of the tip vibration Δz. The initial value of the vibration amplitude was 50 nm. As shown in Fig. 2, the modulated capacitance signal increases with the decrease of tip-sample distance. After the tip touches the surface, the capacitance signal starts to decrease because the vibration amplitude is limited by the surface. The amplitude decrease is clearly shown in Fig. 2. The interference signal simultaneously monitored with an oscilloscope showed a clean sinusoidal vibration of the tip. At last, the cantilever stops vibrating and the tip sticks to the sample. The amplitude signal and the capacitance both become zero. By comparing the capacitance curve and the amplitude curve, it is found that the capacitive signal reaches the peak at the point when the tip begins to touch the surface.

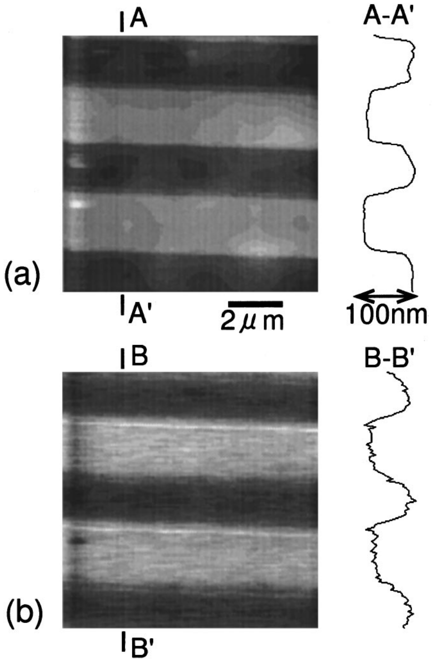

FIG. 3. A topographic image (a) and the corresponding capacitance image (b) of the gratinglike electrode.

Figure 3 shows the set of a topographic image (a) and the corresponding capacitive image (b) of the gratinglike electrode. The cross sections of the images are also shown. The scanned area is about 10 μm×10 μm. The tip vibration amplitude was set at 100 nm. There was no electrical contact between the tip and the sample during the measurement because of the oxide layer and contaminants. In Fig. 3(a), the light regions represent the conductive area, or the electrode while the dark regions represent the glass substrate. As shown in Fig. 3(b) the capacitive signal is large on the electrode. As a whole, Fig 3(a) is a good replica of the sample topography and this proves that the good tip-sample distance regulation was performed with the tapping motion. By comparing Figs. 3(a) and 3(b), it is noticed that the resolution of the topographic image is better than the capacitive image. This is because the topographic signal is determined by the tip-sample contact area, while the capacitive signal is a result of the long-distance nature of the Coulomb interactions. In addition, the cross section of the electrode in Fig. 3(b) is distorted, while the cross section in Fig. 3(a) is almost rectangular. Judging from this and the fact that the sample height difference is rather large, it can be said that the capacitance imaging was affected by the tip geometry. In principle, the

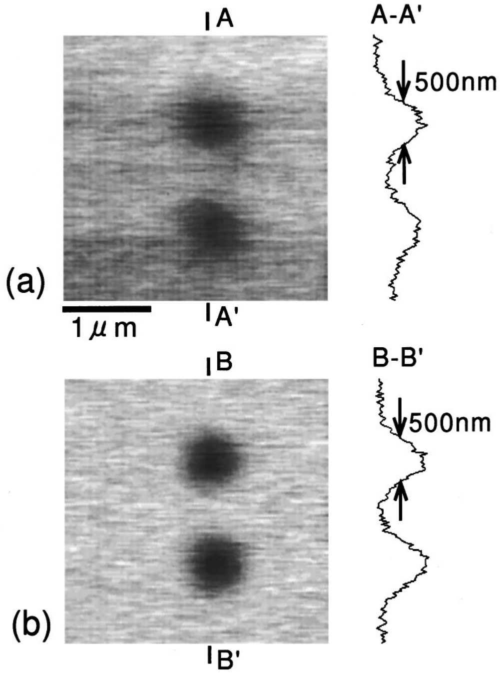

FIG. 4. Capacitance images of the NOS sample obtained by the proposed method (a) and by the C-V method (b).

capacitive resolution is determined by the tip size.[7,10] In our experiments, the resolution was estimated to be on the order of 100 nm. Another possible determinant of the lateral resolution is the tapping amplitude. A large tapping amplitude means the large average tip-sample distance. This factor might be dominant in a measurement with a very sharp tip.

In Figs. 4(a) and 4(b), results of scanning the NOS sample are shown. They are capacitive images obtained with our method and with the *C*-*V* method, respectively. Two written bits are recognized in each image. In the tapping mode measurement, the tapping amplitude was 100 nm. The capacitance signal decreased with the tip above the depleted regions. In the *C*-*V* measurement, the dc bias was 5 V and the voltage modulation of 1 $V_{p\text{-}p}$ was performed. The capacitance signal increased with the tip above the depleted regions. The gray scale in Fig.4(b) is reversed for ease of comparison with Fig. 4(a). The simultaneously obtained topographic image showed that the surface of the nitride film was fairly smooth and that the voltage application had little changed the surface topography. As for the capacitive resolution, as shown in the cross section in Figs. 4(a) and 4(b), the half widths of the depleted regions are measured to be almost the same in both methods. Figure 4 verifies that the performance of our method is as good as that of the conventional *C*-*V* method in detecting the depleted regions. Furthermore, it is possible to image real depletion areas in our method because the bias voltage is not necessary, while in the *C*-*V* method bias voltages are needed to obtain *dC*/*dV* signals, which change the degree of depletion.

ACKNOWLEDGMENTS

This work was supported in part by a Grant-in-Aid from the Ministry of Education, Science, and Culture of Japan, by Support Center for Advanced Telecommunications Technology Research, and by Hoso Bunka Foundation.

[1] Y. Martin, D. W. Abraham, and H. K. Wickramasinghe, Appl. Phys. Lett. **52**, 1103 (1988).
[2] H. Yokoyama and T. Inoue, Thin Solid Films **242**, 33 (1994).
[3] J. M. R. Weaver and D. W. Abraham, J. Vac. Sci. Technol. B **9**, 1559 (1991).
[4] S. Watanabe, K. Hane, T. Ohye, M. Ito, and T. Goto, J. Vac. Sci. Technol. B **11**, 1774 (1993).
[5] D. W. Abraham, C. Williams, J. Slinkman, and H. K. Wickramasinghe, J. Vac. Sci. Technol. B **9**, 703 (1991).
[6] J. R. Matey and J. Blanc, J. Appl. Phys. **57**, 1437 (1985).
[7] C. C. Williams, W. P. Hough, and S. A. Rishton, Appl. Phys. Lett. **55**, 203 (1989).
[8] C. C. Williams, J. Slinkman, W. P. Hough, and H. K. Wickramasinghe, Appl. Phys. Lett. **55**, 1662 (1989).
[9] R. C. Barrett and C. F. Quate, J. Appl. Phys. **70**, 2725 (1991).
[10] Š. Lányi, J. Török, and P. Řehůřek, Rev. Sci. Instrum. **65**, 2258 (1994).
[11] L. H. Parker and A. F. Tasch, IEEE Circuits Devices Mag. **CDM-6**, 17 (1990).
[12] S. Y. Wu, IEEE Trans. Electron Devices **ED-21**, 499 (1974).
[13] T. A. Rost, H. Lin, and T. A. Rabson, Appl. Phys. Lett. **59**, 3654 (1991).
[14] R. Lüthi, H. Haefke, K.-P. Meyer, E. Meyer, L. Howald, and H.-J. Güntherodt, J. Appl. Phys. **74**, 7461 (1993).
[15] Q. Zhong, D. Inniss, K. Kjoller, and V. B. Eklings, Surf. Sci. Lett. **290**, L688 (1993).
[16] C. A. J. Putman, K. O. Van der Werf, B. G. De Grooth, N. F. Van Hulst, and J. Greve, Appl. Phys. Lett. **64**, 2454 (1994).
[17] K. Goto, M. Sasaki, S. Okuma, and K. Hane, Rev. Sci. Instrum. **66**, 3182 (1995).
[18] Y. Yatsuda, S. Nabetani, K. Uchida, S. Minami, M. Terasawa, T. Hagiwara, H. Katto, and T. Yasui, IEEE Trans. Electron Devices **ED-32**, 224 (1985).
[19] S. Iwamura, Y. Nishida, and K. Hashimoto, IEEE Trans. Electron Devices **ED-28**, 854 (1981).