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著者	羽根　一博
journal or publication title	Review of scientific instruments
volume	67
number	2
page range	397-400
year	1996
URL	http://hdl.handle.net/10097/35579

doi: 10.1063/1.1146603

Micromachining with a force microscope tip assisted by electrostatic force

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(Received 18 September 1995; accepted for publication 10 November 1995)

We present a new technique for surface modification with a force microscope tip. By using electrostatic force between a cantilever and a sample surface, deformation on the surface can be performed with a very large load on the order of 10^{-6} N, even if the spring constant of the cantilever is small (on the order of 10^{-2} N/m). Because the tip does not shift laterally while the load is applied, pits are produced with precise positioning. Furthermore, very fast response of the modification on the order of 10^{-6} s was obtained. © *1996 American Institute of Physics.* [S0034-6748(96)03902-1]

I. INTRODUCTION

Recently, scanning probe microscopes have been used as powerful tools not only for observation of surfaces, but also for surface modification.[1–10] Surface modification techniques can be applied to material manipulation and fabrication on a nanometer scale. For example, nanoscale lithography[5,9] and high-density data storage[10] using a scanning probe microscope have been reported.

As for the surface modification with a scanning force microscope (SFM), mechanical modification with a probe tip[6–9] can be one of the most effective techniques for such future technologies as mentioned above because of its simplicity. In the previous reports, deflection of a cantilever holding the tip was increased to obtain a load enough for plastic deformation or wear on the sample surface. The cantilever deflection was increased by shifting the sample surface toward the cantilever. In these methods, however, modification characteristics such as precision and efficiency have not been sufficiently discussed. In other words, the processes were static rather than dynamic. In order to establish a practical modification technique, its performance should be studied and improved. For practical application, the conventional methods have the following disadvantages. First, the dynamic range of the available load is limited by the spring constant of the cantilever. In the case of a weak cantilever, the peak load is limited, while in the case of a strong cantilever, it is not easy to maintain a small load. In surface modification with a SFM, the applied load exerted by the tip should be variable for not only large but also small forces in order to observe the surface without damaging it. Second, because a cantilever is set with an angle with respect to a sample in order to ensure tip contact to the surface, a large cantilever deflection entails a large lateral shift of the contact region on the sample surface. Third, it is not easy to quickly change the loading force because it requires large displacements of the sample or the substrate to which the cantilever is fixed. As a solution to these problems, thermomechanical writing with a small load has already been demonstrated.[10] However, when a large load is needed, another motive force for modification should be chosen.

As seen in micromachine techniques, electrostatic force is widely used as an effective motive force for microstructures.[11–13] In an electrostatic force actuator, a voltage is applied between two adjacent electrodes in order to obtain the attractive force. Fortunately, a SFM cantilever and samples themselves can function as good electrodes for the electrostatic force actuator. It is not difficult to make a cantilever conductive. And it is feasible that a sample or a substrate on which a sample is mounted may be conductive. Taking this advantage, we have developed a new technique for surface modification with a SFM tip, without using a load arising from a large cantilever deflection. As far as we know, this is the first attempt to use electrostatic force for machining a material. In this method, commercially available microcantilevers can be used as is and there is little need to modify a conventional SFM system. Therefore, the functions of the system are not reduced at all. By using the electrostatic force, we have obtained very large loading force on the order of 10^{-6} N even with a weak cantilever. As a cantilever deflection was not needed, the contact area on the surface was not shifted and deformation was performed with accurate positioning. Furthermore, the loading force was quickly changed and large force can be obtained in a very short time.

II. PRINCIPLE

Figure 1 shows a schematic diagram of the experimental setup. As shown in Fig. 1, pulsed voltage is applied between the cantilever and the sample surface in order to obtain a load for indentation on the surface. For this purpose, both the cantilever and the sample surface have to be conductive. The cantilever is conductive on the back because it is gold coated for the purpose of the optical detection of the deflection. As for the sample, conductive material such as metal can be used as is. If the material to be modified is not conductive, the substrate on which it is mounted should be conductive. For nanoscale purposes, materials are often thin and the electrostatic force is not seriously reduced. Assuming a simple model of two plane electrodes parallel to each other, electrostatic force F between the electrodes is given by

$$F=\frac{1}{2}\varepsilon S\left(\frac{V}{d}\right)^2, \qquad (1)$$

where ε is the permittivity between the electrodes, S is the area of the electrodes, V is the applied voltage, and d is the gap between the electrodes. From Eq. (1), electrostatic force F can be roughly calculated to be 5 μN, where $\varepsilon=1\times10^{-11}$ F/m, $S=1\times10^{-8}$ m^2, $V=100$ V, and $d=1\times10^{-5}$ m. In our method, the tip of the cantilever is in contact with the surface

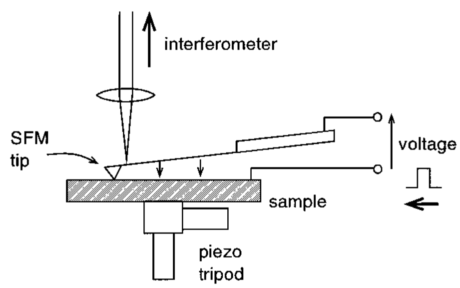

FIG. 1. Experimental setup.

throughout the modification. Since the cantilever itself is dielectric, current does not flow through the tip. Under this condition, the cantilever is now interpreted as a beam clamped at one end and freely supported at the other end (tip). Then the load on the surface is the reaction of the upward supporting force exerted by the surface. Considering the static equilibrium of the beam with the uniformly distributed load (electrostatic force) F on the whole region of the beam, the load to the sample surface is derived to be $3F/8$. When the pressure caused with the tip is in excess of the yield stress of the material, the plastic deformation occurs and the permanent strain (pit) is given to the material surface.

In our method, tip–sample contact region shift does not occur in principle while the tip is pressing the surface. Strictly speaking, however, a slight shift is caused for the following reason. With the attractive electrostatic force on the cantilever beam, the cantilever deflection is largest about the center of the beam. As a result, the beam angle at the free end (tip) is changed, especially when the cantilever is weak. This angle change induces the contact region shifts. For better positioning performance, it should be alleviated. Fortunately, the tip shift is controlled by the pulse width of the voltage applied. That is, the pulse width determines the modes of vibration. Because the electrostatic force acts on the whole region of the cantilever, a number of modes of vibration at resonant frequencies can be excited. The tip shift depends on which of them have been generated. In particular, the amplitude of the first mode is much larger than that of the higher modes. Therefore, by exciting the first mode less, the tip shift is decreased. That is, the higher modes are employed for deformation. This is achieved by setting the pulse width shorter than half the vibration period of the first mode. While the tip shift is reduced, however, the load to the sample surface is also reduced for it. Therefore, the voltage to be applied should be raised to compensate for it.

III. EXPERIMENTAL SETUP

In our experiment, we used a homemade SFM system.[14] Since the optical interferometer is used in it, absolute value of a cantilever deflection is measured and the accurate surface topography can be obtained. For good electric isolation, a ceramic plate was inserted between the body and the cantilever. All the experiments with the SFM were performed in air.

As a cantilever for the SFM, a commercially available microfabricated cantilever (Park Scientific Instruments) was used. It was made of Si_3N_4 and its back was gold coated. It was V shaped and 200 μm long. The spring constant and the resonant frequency were 0.064 N/m and 17 kHz, respectively. The tip height was 4 μm. The angle between the cantilever and the sample surface was set to be 6°. That is, the average distance between the two electrodes was about 10 μm.

As a sample to be modified, for ease of deformation and stability in air, we selected an indium thin film (about 200 nm thick) evaporated on a glass substrate. The yield stress of bulk indium is known to be very small (2.6×10^6 Pa).

IV. EXPERIMENT

First, a load applied on the sample surface when the electrostatic force is acting on the cantilever was measured. Because it was not easy to directly measure the actual load with the tip on the surface, we obtained the value of the load using the principle of superposition. As mentioned above, the cantilever shown in Fig. 1 is treated as a beam clamped at one end and freely supported at the other end. Here, suppose that a distributed load (electrostatic force) is applied on it. The beam under this condition is divided into two beams. One is a cantilever deflected by Δz at the free end, with the same distributed load on it. The other is a cantilever deflected by $-\Delta z$ at the free end, with a concentrated load on the free end. According to the principle of superposition, the load to be obtained is regarded as equal to $k\Delta z$ for small Δz, where k is the spring constant of the cantilever. In the experiment, the cantilever deflection Δz was measured as a function of the applied voltage V, with the tip 100 nm above the surface. The deflection was measured with the optical interferometer in the SFM. The input signal was ac modulated in order to avoid the influence of dielectric absorption.

Next, the resonant frequency of the cantilever was measured under the condition that the tip is in contact with the sample surface. The cantilever was vibrated by applying small electrostatic force with sinusoidal modulation. Since in this configuration the boundary condition is not the same as that with the tip free, the resonant frequency can be changed.

Next, a load was applied to the indium thin film with electrostatic force and pits were produced. The average load was maintained with feedback circuits while the short pulse of the voltage is applied. After the indentation, the sample was scanned with the same tip for the observation of the topography change. The load was on the order of 10 nN during the observation.

In order to investigate the indentation characteristics, pit depth was measured as a function of the applied voltage. As a result, threshold voltage for the indentation was estimated.

V. RESULTS AND DISCUSSION

Figure 2 shows the measured load L to the surface as a function of the applied voltage V. The load is a product of the measured cantilever deflection multiplied by its spring constant. From Fig. 2, an equation of $L = 4.7\times10^{-11}\ V^2$ is

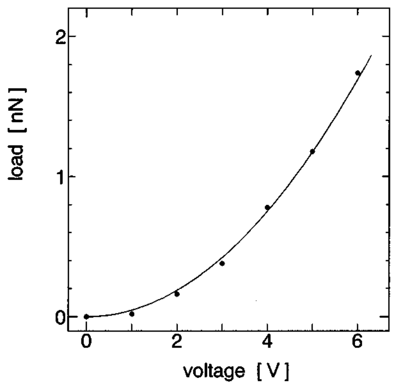

FIG. 2. Measured load as a function of the applied voltage.

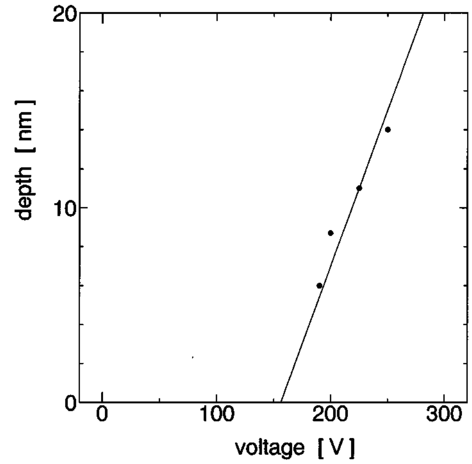

FIG. 4. Pit depth as a function of the applied voltage.

obtained, where $k = 0.064$ N/m. According to this equation, the loading force is calculated to be 0.47 μN, when $V = 100$ V.

The first resonant frequency of the cantilever used was 17 kHz. With the tip on the surface, however, it was observed to be 60 kHz. From this result, half the period of the resonance is 8.3 μs (1/60 000 s) and the pulse width should be set shorter than this.

Figure 3 shows a SFM image of a pit on the indium thin film. The scanned area is 200×200 nm. Voltage of 200 V was applied to the electrodes with the tip in contact with the surface. According to the above given relation between the voltage and the load, this corresponded to the load of 1.9 μN. The pulse width of applied voltage was 1 μs. As mentioned above, because the first mode of the cantilever resonance was suppressed, the net load was smaller than this. As shown in Fig. 3, the pit is about 50 nm across and 9 nm deep although the tip convolution might have affected the image. The pit is considered to reflect the tip shape. From the cross section $A-A'$ in Fig. 3, the tip radius is estimated to be about 40 nm. In passing, the pit looks long in the direction perpendicular to the cross section $A-A'$, because the edge is dull in that direction. The surface appears to have no lips around the pit. This suggests that the pit was formed as a result of the material having been totally pushed inside. As for the lateral shift of the tip contact region on the surface, little is observed in our method. If any, it is estimated to be on the order of several nanometers. In the conventional methods, where the cantilever deflection is increased to obtain a large load, the cantilever has to be deflected by 29 μm to obtain the load of 1.9 μN when the spring constant of the cantilever is 0.064 N/m. Then the lateral shift of the contact region on the surface is estimated to be 3 μm with the angle between the cantilever and the sample 6°. This is extremely large compared to the pit size obtained in our method.

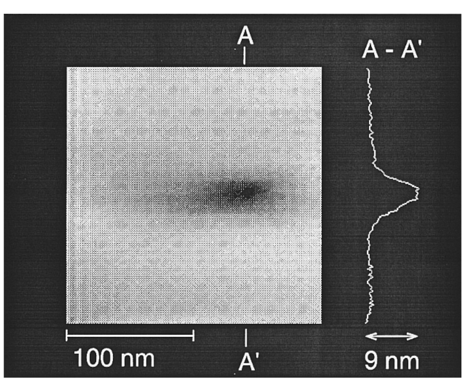

FIG. 3. SFM image of a pit.

As shown in Fig. 4, the pit depth is plotted against the applied voltage. As expected, the pit became deeper with the increase of the applied voltage. From Fig. 4, the threshold voltage is estimated to be 160 V. Then the threshold load is calculated to be 1.2 μN. It strongly depends on the tip sharpness, since the tip radius determines the contact area and therefore, the pressure to the surface. It also depends on defects near the surface. As is well known, plastic deformation is governed by defects in materials. In nanoscale regions, where the defects should be microscopically treated, plastic deformation properties are quite different from bulk properties.[15] In fact, in our experiment, some part of the sample surface exhibited little plastic deformation.

In this technique, the indentation speed can be set much higher than the first resonant frequency of the cantilever. However, the vibration of the first resonance is observed to remain a little after the voltage was applied. The remnant vibrations should be eliminated, because they might cause a problem in repetitive indentation. Ultimately, the motive force for the load should act right on the tip. Under this condition, the lower resonances would not be excited. And the indentation performance is determined not by the lever properties but totally by the tip–sample contact properties. In this sense, as long as the force changing rate permits, magnetic force[16] might be also the effective motive force for the surface deformation with a magnet on the tip. As for the ac characteristics in our system, the electrostatic force perfectly follows the input signal. The capacitance between the cantilever and the sample surface is on the order of 10^{-14} F and the time constant of the system is very small. It should be also noted that the indentation speed depends on the pit depth and the deformation resistance of the material. In other words, a certain amount of time is needed for material flows. This can be a significant limit of the indentation speed in performing considerably large deformation.

ACKNOWLEDGMENTS

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

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