Shear force microscopy with capacitance detection for near-field scanning optical microscopy

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Shear force microscopy is very useful for distance regulation in near-field scanning optical microscopy (NSOM). However, the optical method used to detect the shear force can cause problems when imaging photosensitive materials, i.e., the shear force detection beam can optically pump the sample. We present here a new approach to shear force detection based upon capacitance sensing. The design, operation, and performance of the capacitance detection are presented. Shear force topographic images of hard and soft surfaces are shown using tungsten and NSOM fiber tips. The closed loop vertical sensitivity achieved is 0.01 nm/$\sqrt{\text{Hz}}$. © *1995 American Institute of Physics.*

The atom force microscope (AFM)[1] is a powerful tool for investigating surfaces on a nanometer scale. The shear force microscope,[2,3] one of its descendants, has been shown to have some advantages over the AFM. In particular, one advantage is the elimination of the spontaneous jump-to-contact of the tip onto a surface. Another advantage is that it can provide a simple distance regulation method for the near-field scanning optical microscope (NSOM).[3–11] Shear force detection is presently accomplished by optical methods.[3–11] These optical approaches can cause troubles for NSOM spectroscopy due to the optical pumping of the surface of interest by the shear force detection beam and the mixing of this beam with the near-field optical signal. Capacitance sensing of probe motion can eliminate these optical problems. Previously, capacitance detection has been used to measure magnetic forces acting on a vertical cantilever.[12] In this work, the implementation and performance of a capacitance based shear force microscope is described. It is shown that the approach is simple, easily implemented, and has excellent sensitivity.

The schematic of the new shear force microscope is shown in Fig. 1. The tip is held perpendicular with respect to the sample plane. A dither piezo is used to vibrate the tip at a frequency slightly off its resonance. The tip–sample interaction via the shear force takes place within a few nanometers of the surface and produces a damping of tip motion. The amplitude of tip vibration is measured by a capacitance lead coming out from an ultrasensitive capacitance sensor.[13] The signal output from the capacitance sensor is sent to a lock-in amplifier, where it is filtered and rectified. The lock-in output is then compared to a set point in the feedback loop. The loop regulates tip–sample separation distance by maintaining a constant damping of tip motion as the tip is scanned across the sample.

The basic design of the shear force microscope head is depicted in Fig. 2. The tip is glued in a micropipette which is held on a quartered dither piezo tube. The dither piezo is attached to an upper tripod which is mounted on a lower tripod. The lower tripod is mounted on a base plate. The capacitance lead is glued on a ceramic plate which is fixed on the lower tripod. The sample is mounted on a scan piezo tube, which is attached to the base plate. The tip–capacitance lead distance and the capacitance lead position are adjusted easily using the upper tripod. The tip–sample distance is adjusted using the lower tripod. The stability of this mechanical system is adequate to lock the feedback loop overnight without any need for adjustment in an open air laboratory environment. It takes less than 5 min to replace a tip and reposition the shear force sensor.

The heart of capacitance detection is a capacitance sensor which operates at a frequency near 1 GHz and can measure capacitive changes between the tip and capacitance lead on the order of 3×10^{-21} F/$\sqrt{\text{Hz}}$.[13] The capacitance sensor lead is inductively grounded. The capacitance lead is made of a tungsten wire (0.002 in. in diameter). The length of the capacitance lead from the sensor to tip is approximately 6 1/2 in. long, using the second resonance of the capacitance sensor. The first resonance requires a 1 in. capacitance lead and is too short to use with our shear force microscope head. The capacitance sensor lead is shielded with metal plates to minimize stray capacitance effects. By adjusting the length of the capacitance lead and the shielding, the maximum sensitivity of the capacitance sensor is achieved. The end of the capacitance lead should be smooth and clean in order to sense the oscillation of the tip. The position of the capacitance lead should be very near the tip end, because the tip

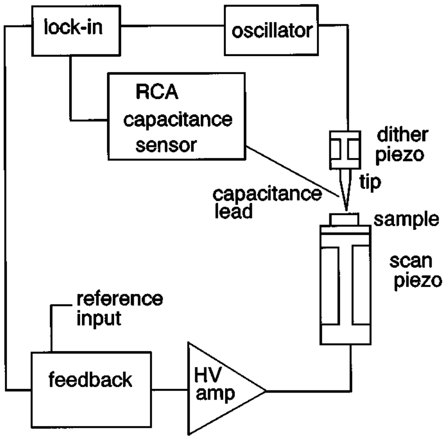

FIG. 1. Schematic of a new shear force microscope with capacitance detection.

motion is largest there. Electrochemical etching in NaOH of the capacitance lead gives a smooth and clean surface. The end of capacitance lead is bent to form a very small semicircle. Using the top part of the semicircle as the sensing area of the tip motion, we achieve not only smooth sensing but also easy adjustment between the capacitance lead and the tip. The distance between tip and capacitance lead and the sensing area of the capacitance lead are estimated to be 100 nm and 1 μm^2, respectively. The estimated capacitance change for 1 Å change of tip oscillation in this experiment is around 10^{-19} F. The detection sensitivity of tip oscillation is approximately 0.01 Å/$\sqrt{}$Hz.

We first attempted the capacitance detection of the shear force using tungsten tips made by etching tungsten wire. The resonance frequency, quality factor, and calculated spring constant of the probe used in this experiment are 16.62 kHz,

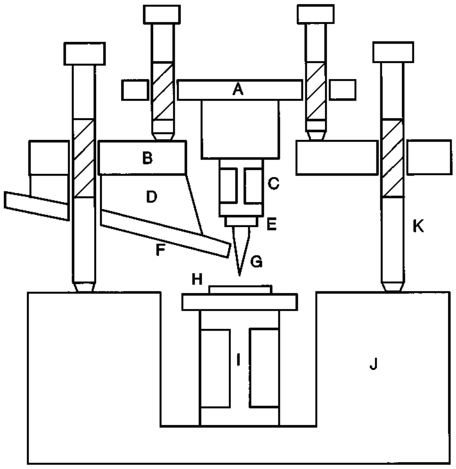

FIG. 2. A design of the shear force microscope head showing (A) the upper tripod, (B) the lower tripod, (C) the dither piezo, (D) the ceramic plate, (E) the micropipette, (F) the capacitance lead, (G) the tip, (H) the sample, (I) the scan piezo, (J) the base plate, and (K) the screw.

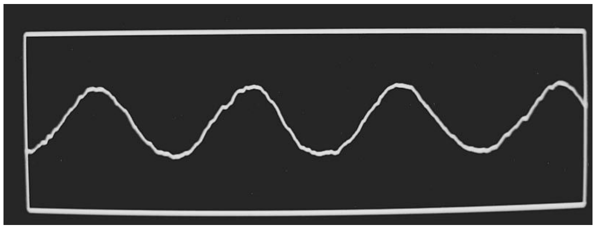

FIG. 3. Response of the system to 10 Å modulation of vertical sample position at 100 Hz. The loop bandwidth was approximately 200 Hz.

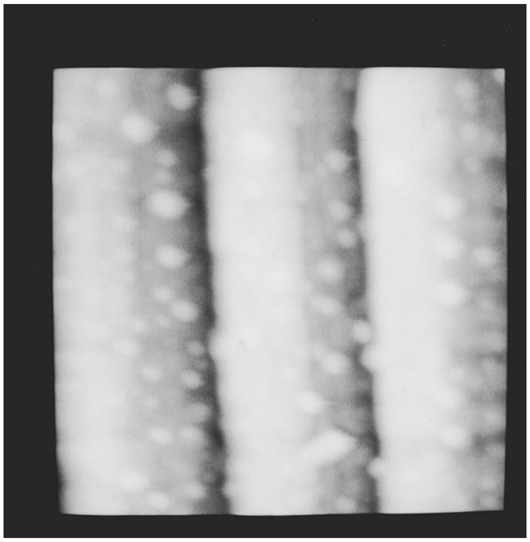

FIG. 4. Shear force detected topographic image (3 μm×3 μm) of gold grating taken by tungsten tip. The periodicity of the grating is 1 μm. The vertical height is 50 nm.

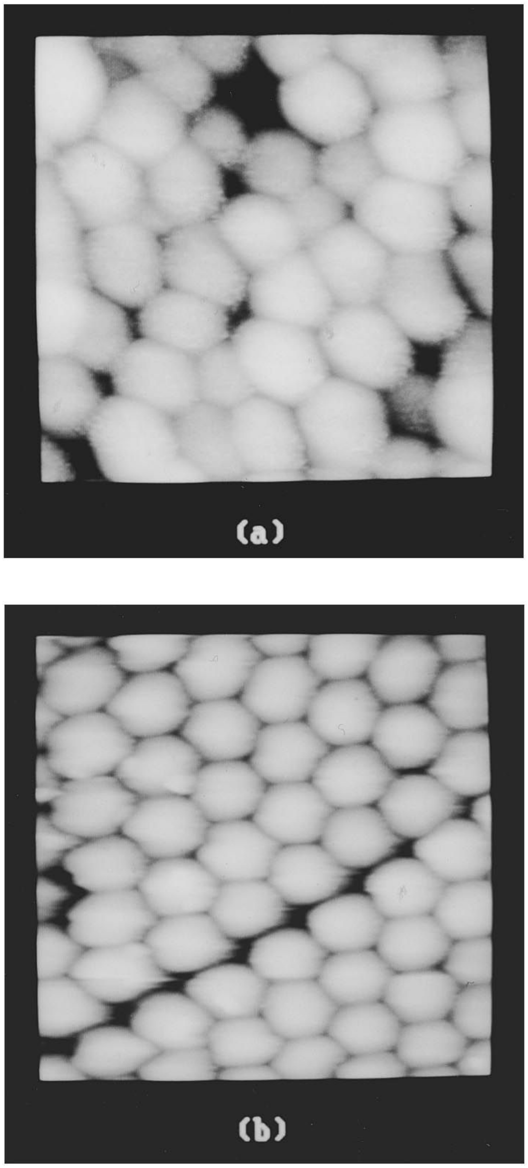

FIG. 5. (a) Shear force detected topographic image (500 nm×500 nm) of 69 nm polystyrene spheres on a glass substrate. (b) Shear force detected topographic image (2.8 μm×2.8 μm) of 400 nm polystyrene spheres on a glass substrate. Both are taken by tungsten tip.

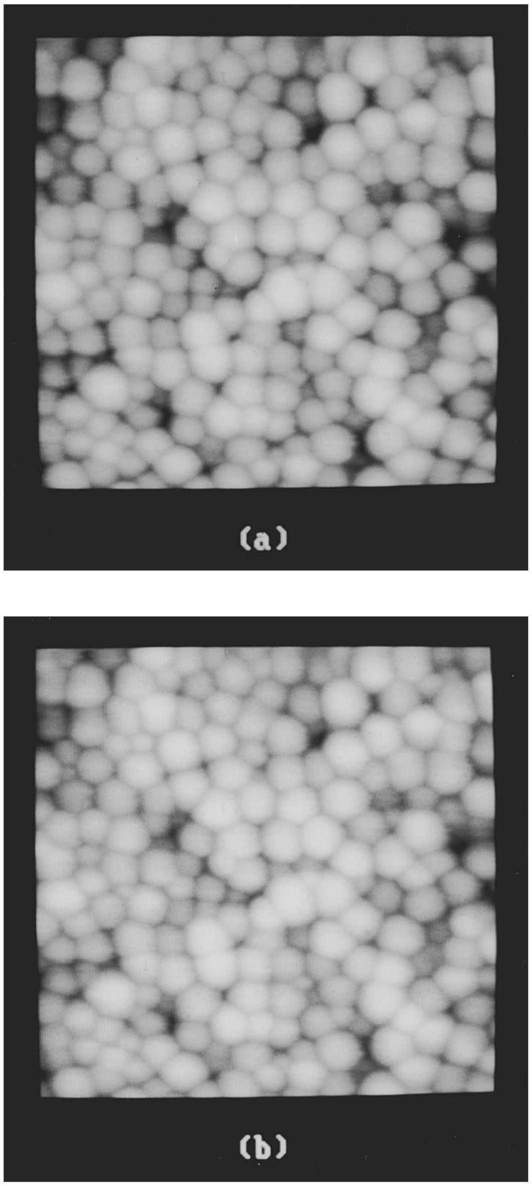

FIG. 6. Shear force detected topographic images (930 nm×930 nm) of 69 nm polystyrene spheres on a glass substrate. Both are images of the same area and taken with the same aluminum coated fiber tip.

100, and 4 N/m, respectively. The estimated peak amplitude of the tip vibration was approximately 1 nm, while the sample was not engaged. The position of capacitance lead was approximately 50 μm from the tip end. The sensitivity of the system to vertical displacement was tested by vertically modulating the sample at an amplitude of 1 nm with sine wave of 100 Hz, while maintaining the system under feedback control. The resulting response of the system, taken at a true loop bandwidth of 200 Hz, is shown in Fig. 3. Sample height variations of about 0.1 nm are detectable at this bandwidth.

By this method, the imaging of hard surfaces is very straight forward. The image of Fig. 4 shows a gold grating imaged by the tungsten tip. The periodicity of the grating is 1 μm. Small structure on the grating is also observable. We found that the imaging of soft surfaces requires a dither amplitude of approximately 1 nm, a low spring constant tip, and the capacitance lead close to the tip end. Figures 5(a) and 5(b) display the resulting images of polystyrene spheres with diameters of 69 and 400 nm, respectively, by the tungsten tip. After changing the tip, we repeated these images. We found that the system is very reliable, repeatable, and nondestructive.

We have also imaged soft surfaces using an aluminum coated NSOM fiber tip.[5] The resonance frequency, quality factor, and calculated force constant of the aluminum coated fiber tip used in this experiment are 44 kHz, 103, and 2 N/m, respectively. Figure 6 shows the two separate images of the polystyrene spheres of 69 nm diameter by the aluminum coated fiber tip. These two images prove that good repeatability on soft surfaces is achieved by this method. Although the fiber tip diameter was around 300 nm after aluminum coating, the image of the 69 nm diameter spheres are still clear due to small asperities on the end of the coated fiber. The overnight stability of the feedback loop and minimal preparation time show that the drift is low and the system is easy to use in this microscope.

In summary, we have presented a simple and sensitive shear force microscope based upon capacitance detection. This new shear force microscope is capable of imaging both hard and soft surfaces repeatedly and nondestructively. We believe that it will be particularly useful in imaging photosensitive materials by NSOM. It may also be generally useful for imaging soft surfaces at high spatial resolution.

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[1] G. Binnig, C. F. Quate, and Ch. Gerber, Phys. Rev. Lett. **56**, 930 (1986).
[2] P. C. Yang, Y. Chen, and M. Vaez-Iravani, J. Appl. Phys. **71**, 2499 (1992).
[3] E. Betzig, P. L. Finn, and J. S. Weiner, Appl. Phys. Lett. **60**, 2484 (1992).
[4] R. Toledo-Crow, P. C. Yang, Y. Chen, and M. Vaez-Iravani, Appl. Phys. Lett. **60**, 2957 (1992).
[5] E. Betzig and J. K. Trautman, Science **257**, 189 (1992).
[6] E. Betzig, J. K. Trautman, R. Wolfe, E. M. Gyorgy, P. L. Finn, M. H. Kryder, and C.-H. Chang, Appl. Phys. Lett. **61**, 142 (1992).
[7] M. Vaez-Iravani and R. Toledo-Crow, Appl. Phys. Lett. **63**, 138 (1993).
[8] E. Betzig, S. G. Grubb, R. J. Chichester, D. J. DiGiovanni, and J. S. Weiner, Appl. Phys. Lett. **63**, 3550 (1993).
[9] E. Betzig and R. J. Chichester, Science **262**, 1422 (1993).
[10] W. P. Ambrose, P. M. Goodwin, J. C. Martin, and R. A. Keller, Phys. Rev. Lett. **72**, 160 (1994).
[11] R. D. Grober, T. D. Harris, J. K. Trautman, E. Betzig, W. Wegscheider, L. Pfeiffer, and K. West, Appl. Phys. Lett. **64**, 1421 (1994).
[12] A. DiCarlo, M. R. Scheinfein, and R. V. Chamberlin, Appl. Phys. Lett. **61**, 2108 (1992).
[13] J. R. Matey and J. Blanc, J. Appl. Phys. **47**, 1437 (1985). .