Scanning capacitance microscopy on a 25 nm scale

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A near-field capacitance microscope has been demonstrated on a 25 nm scale. A resonant circuit provides the means for sensing the capacitance variations between a sub-100-nm tip and surface with a sensitivity of 1×10^{-19} F in a 1 kHz bandwidth. Feedback control is used to scan the tip at constant gap across a sample, providing a means of noncontact surface profiling. Images of conducting and nonconducting structures are presented.

Many new microscopies have been developed based upon local interactions between an ultrasharp tip and sample. These "near-field" interactions include electron tunneling,[1] atomic force,[2] magnetic force,[3] and thermal,[4] optical,[5,6] and electrostatic[7] coupling. The lateral resolution of these new microscopies is determined by the physical size of the tip and the variation of the interaction strength with gap. Scanning capacitance microscopy[7–9] provides a mean for surface characterization through the measurement of local capacitance between tip and sample. It is useful for profiling both conductors and insulators. Perhaps more importantly, it provides a means for imaging variations in dielectric properties in or through insulating layers. This capability is projected to be useful in semiconductor devices. To date, scanning capacitance imaging has been demonstrated by Matey[7] with a lateral resolution of 0.1 μm by 2.5 μm, in a system where the tip is scanned in the tracks of a pregrooved disk. More recently, Bugg[8] has demonstrated 2 μm lateral resolution in an unguided scanning system. Capacitance force microscopy has also been demonstrated using the atomic force microscope[9] with resolution approaching the 100 nm scale. To address many potential applications, a resolution well below 100 nm is desired. In this letter we show that the present sensitivity limit for capacitance imaging is below 10 nm, and present results which demonstrate capacitive imaging on a scale of 25 nm.

The heart of the capacitance microscope is based upon the RCA Video Disc capacitance sensor. The sensor was developed by RCA to capacitively read back a video signal stamped on a disk.[7] The basic elements of the sensor can be seen in Fig. 1. An UHF (ultrahigh frequency) oscillator (915 MHz) is coupled to detection circuitry through a resonant circuit. The resonant circuit is composed of a strip line resonator and a fly lead. In this work, the standard RCA fly lead has been replaced by a 0.010 in. tungsten wire approximately 2 cm long, the end of which has been electrochemically etched to a tip. One end of the tungsten wire is attached to the strip line, while the tip end of the wire is brought close to the sample surface. The sample is electrically grounded to the casing of the resonator circuitry, so that some of the UHF field is dropped across the gap between tip and sample.

The resonant frequency of the circuit is largely determined by the total stray capacitance (0.1 pF) of the strip line and fly lead, but is modulated by a contribution introduced by the tip/sample capacitance. This tip/sample capacitance shifts the resonant frequency of the sensor, modifying the transmitted UHF signal from oscillator to detector. For small changes in capacitance, the output voltage changes linearly with the change in capacitance between tip and sample. It is important to minimize the stray capacitance between the fly lead and the local environment. If care is not taken, the resonance frequency will shift outside the tuning range of the resonant circuit.

The noise in the capacitance circuit is dominated by oscillator instability,[10] and the noise spectrum is highly biased toward the low frequencies. It is therefore advantageous to modulate the capacitance between tip and sample at a high frequency and detect the capacitive variations where excess noise is not significant. This is achieved by placing a small vibration on the tip or sample.[4,8] This capacitive gradient signal changes rapidly as the tip approaches the sample, providing a feedback control signal. For a simple parallel plate, the capacitive gradient goes as the inverse square of the gap spacing $dC/dz=\epsilon_0\epsilon\text{Area}/d^2$. A schematic of the feedback control loop is shown in Fig. 2. The ac signal out of the capacitance sensor is sent to a lock-in amplifier where it is rectified and filtered. In this work, the tip/sample modulation frequency was 27 kHz, although frequencies as high as 100 kHz have been used. The rectified signal is then sent into a conventional scanning tunneling microscope (STM) feedback loop. A piezoelectric tube scanner is used to provide up to 5 μm of scan in the x and y dimensions, and 2 μm in the vertical dimension. The control signal for the vertical tip position is stored with the scan coordinates in a lab computer, and displayed as either a grey scale or line scan image. The entire system under feedback loop control has been operated at a 0.5 kHz bandwidth.

A simple first-order calculation shows that capacitive imaging on a 10 nm scale requires a sensitivity given by $C=\epsilon_0\epsilon\text{Area}/d=5\times10^{-19}$ F, for a 10 nm × 10 nm square area, with 2 nm gap and a dielectric constant of 1. To measure the sensitivity of the capacitance probe, the sensor output was measured while an electrical modulation of a varactor diode capacitor in the resonant circuit was performed.

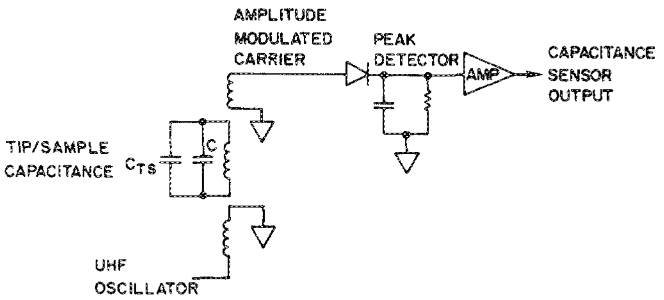

FIG. 1. Schematic diagram illustrating the RCA capacitive sensor.

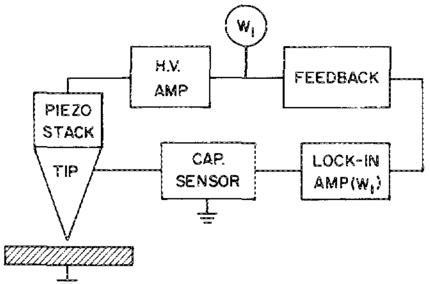

FIG. 2. Illustration of the capacitance servo loop.

This measurement was related to the tip/sample capacitance sensitivity using the analysis presented by Palmer.[10] The results indicated that the output sensitivity of the capacitive sensor was 70 mV/fF (10^{-15}) at the tip. The rms noise level in a 1 kHz bandwidth was measured at a frequency of 10 kHz to be 7 μV. These measurements demonstrate a capacitive sensitivity in a 1 kHz bandwidth of

$$C_{\min} = 1\times 10^{-19}\ \text{F}.$$

This sensitivity is adequate to image 10 nm square areas with a signal to noise ratio of 14 dB. Measurements at higher frequencies[10] have shown that noise levels can be an order of magnitude smaller, in a 30 kHz bandwidth. Under these conditions, the capacitive sensitivity is approximately

$$C_{\min} = 2\times 10^{-22}\ \text{F}$$

in a 1 Hz bandwidth. Capacitive variations of this size at 10 V correspond to charge variations $\Delta Q = \Delta V \Delta C$ of approximately 10^{-2} e. It is believed that improvements can be made to the capacitive sensor to further increase its sensitivity.

To demonstrate the high-resolution capabilities of capacitance microscopy, electrochemically etched tungsten tips were prepared. The 0.010 in. tungsten wires were placed in a 2 M NaOH solution with a positive dc bias voltage applied to the tip. Upon application of the voltage, etching of the tungsten proceeds at the air/NaOH interface, producing an hourglass shape. As the etch continues, the lower portion of the tip drops off, leaving a sharpened tip with a small cone angle. A circuit senses the change in current, and removes the dc voltage. The tip is then removed and rinsed with deionized water. Etching under these conditions produced tips with diameters as small as 25 nm as measured by a scanning electron microscope.

Two samples were imaged with the prepared tips. The first sample consisted of a two-dimensional array of circular holes with 50 nm diameter on 100 nm centers, fabricated in 100-nm-thick polymethylmethacrylate (PMMA) by a high-resolution vector scan lithography system.[11] The resist was over coated with a 20 nm gold coating. Figures 3(a) and 3(b) contain the line scan and grey scale capacitance images obtained by raster scanning the tip over the periodic structure, while under feedback control. Figure 3(c) displays a STM image of the same sample in a different area. In the STM image, the circular holes on 100 nm centers are seen covered by the granular gold film. The typical grain size of the gold film appears to be approximately 10 nm. A comparison of the three images reveals that in addition to the circular holes in the coated PMMA, some of the larger grains or grain clusters have been resolved by the capacitance microscope. The smallest cluster clearly imaged by capacitance has a FWHM of 25 nm. The minimum separation between resolved structure appears to be approximately 30 nm. The general character of the capacitance image is similar to the STM image, but at a lower resolution. STM imaging of the same area shown in Figs. 3(a) and 3(b) was attempted, but good tunneling was found to be impossible. This poor tun-

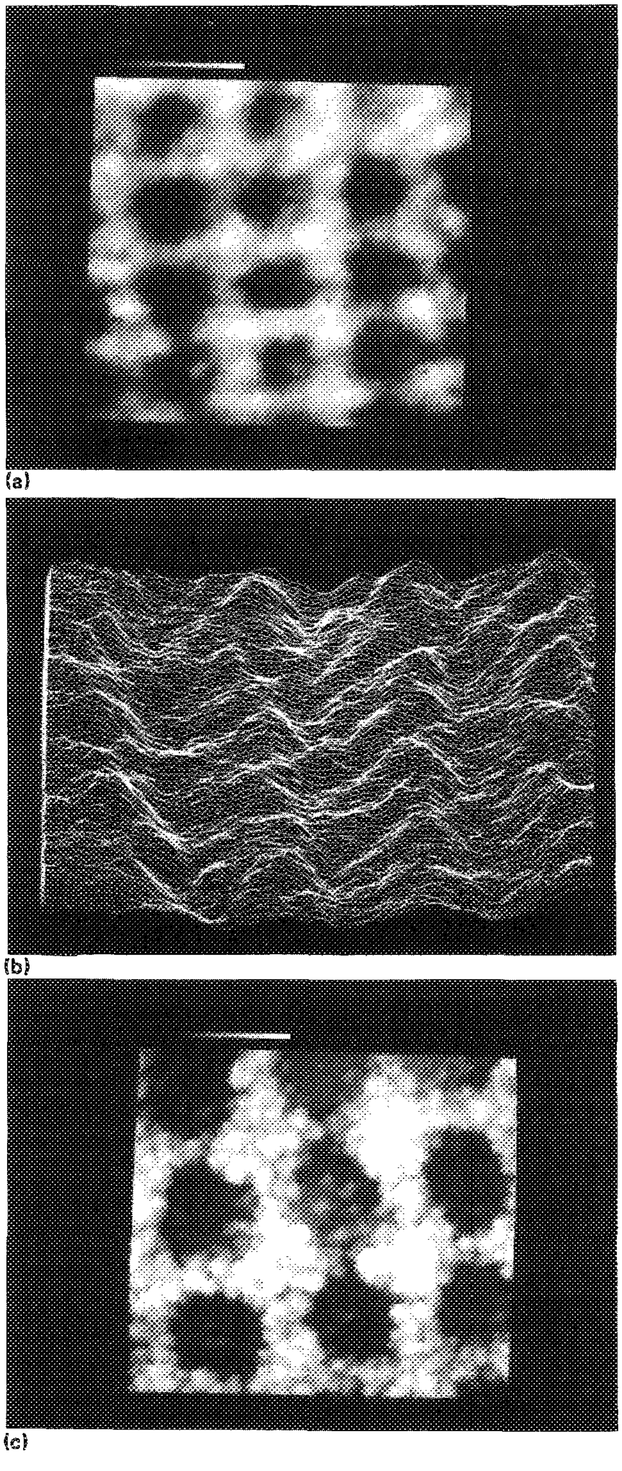

FIG. 3. (a) (b) Gray scale and line scan capacitance images of an array of 50 nm circular holes on 100 nm centers. The structure is in a 100-nm-thick PMMA film, overcoated with 20 nm of gold. The field of view of a 370 nm square. (c) Gray scale STM image of the same sample in a different region. The field of view is a 270 nm square.

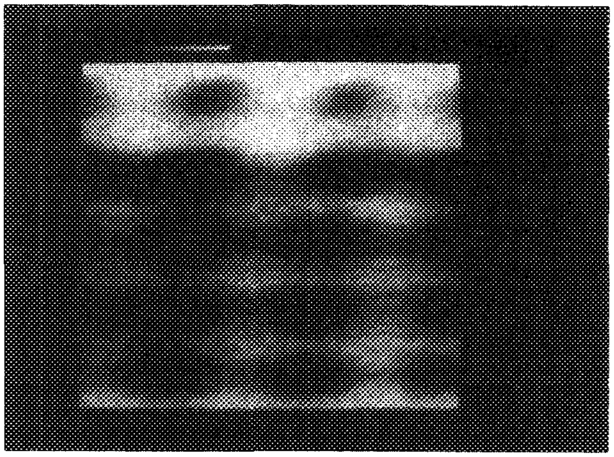

FIG. 4. Capacitive image of a 30-nm-thick PMMA film on silicon with an array of 50 nm holes on 100 nm centers. The sample is not coated with gold.

neling condition has been seen on many samples that have been out in air for many days. This illustrates the virtue of capacitive imaging, in that imaging through insulating layers is possible. Because the tip shape did not permit the tip to reach the bottom of the circular holes, the vertical motion during the scans was limited to approximately 10 nm. This is much less than the actual depth of the hole, which reduces the contrast seen in the capacitance image.

Figure 4 contains the image of an array of 50 nm holes of 100 nm centers in a 30-nm-thick PMMA film. The substrate was silicon, with no metalic over coat. In this image, the array is clearly seen, at what appears to be somewhat lower resolution. The signal to noise ratio in these measurements was lower than in the case of the gold-coated sample. This makes sense in that the capacitance is largest when the distance between tip and ground plane is smallest. Without metalization, the fields are dropped across the 30 nm film, whereas with the gold overcoat, the capacitor plates may be separated by only 5 nm, increasing the capacitance by almost an order of magnitude.

We have demonstrated near-field capacitance imaging on a 25 nm scale. These results, however, do not represent the limit for capacitive imaging. With an optimization of capacitance sensors, and the use of tips with ultrasmall tip curvatures, sub-10-nm imaging should be achievable. Even at present capability, the promise for imaging dielectric properties on such a scale will prove interesting and useful.

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