Near-field microwave microscope with improved sensitivity and spatial resolution

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The near-field scanning microwave microscope has become a popular instrument to quantitatively image high-frequency properties of metals and dielectrics on length scales far shorter than the wavelength of the radiation. We have developed several new ways to operate this microscope to dramatically improve its spatial resolution and material property sensitivity. These include a novel distance-following method that takes advantage of the stability of a synthesized microwave source to improve the signal-to-noise ratio of our earlier frequency-following imaging technique. We also discuss novel height-modulated imaging techniques, culminating in a new tapping-mode method, which makes a 14 dB improvement in sensitivity, a 17.5 dB improvement in signal-to-noise ratio, and a factor of 2.3 improvement in spatial resolution compared to distance-following imaging. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1571954]

Near-field microwave microscopy with coaxial resonators has been used successfully to quantitatively image sheet resistance,[1] dielectric constant,[2–4] dielectric polarization,[5–7] topography,[8] magnetic permeability,[9] and Hall effect.[10] However, it has some limitations that are in part common to all the resonator-based near-field microwave microscopy techniques. In this Note, we describe a new technique for greatly improving the sensitivity and spatial resolution that can be implemented in a variety of existing microscope schemes for imaging dielectric as well as conducting samples.

The probe of our near-field microscope[8,11] is an open-ended coaxial transmission line resonator with capacitive coupling to the feed line (Fig. 1). In the presence of a sample at the open end of the resonator, the resonance frequency is shifted by an amount depending on the local properties of the sample beneath the probe and on the probe–sample separation. The probes can have a tip that extends the center conductor of the coaxial resonator to a sharp point (hereafter called "tipped probes"), thus greatly enhancing and concentrating the rf electric fields in the sample.[12] In the original frequency-following (FF) configuration,[11] the system is kept at resonance by adjusting the frequency of the microwave source with the help of frequency modulation of the source signal. A dc signal produced by the feedback circuit is used to control the central frequency of the source so that it is always equal to the resonance frequency of the resonator with the sample.[11] This dc signal, which is proportional to

the resonance frequency shift, is used for imaging. The measurements can be performed with the probe out of contact[1] or in a gentle (50 μN force) contact with a sample.[3] One important advantage of the FF imaging mode is its broad dynamic range.[13]

The main disadvantage of the out-of-contact imaging is the absence of any control on the probe-to-sample distance, making it difficult to image samples at small distances. A further disadvantage of the FF imaging mode is the use of a dc feedback signal into the frequency modulation input port of the microwave source. This configuration permits source frequency drift and any noise in the feedback electronics to directly affect the resulting images, and also limits the ultimate frequency shift resolution. We have developed a new tapping mode height-modulated distance-following imaging mode to overcome all of these problems.

We begin with a discussion of previously unpublished imaging techniques that form the basis for our new technique. The schematic of the distance-following (DF) microscope is displayed in Fig. 1. The adder shown in the shaded rectangle with the dashed boundaries is absent in this configuration, and the feedback loop is closed, bypassing the adder. The probe is out of contact with the sample. The microwave source operates in the fully synthesized ac/locked mode to take advantage of its $1:10^9$ frequency stability. The microwave signal is frequency modulated as with the FF mode, however the system is kept at the resonance by adjusting the sample–probe separation. The dc signal from the feedback circuit used for imaging is amplified and applied to the piezoelectric actuator holding the sample at a distance that keeps the resonant frequency of the microscope–sample system equal to the frequency of the stable synthesized

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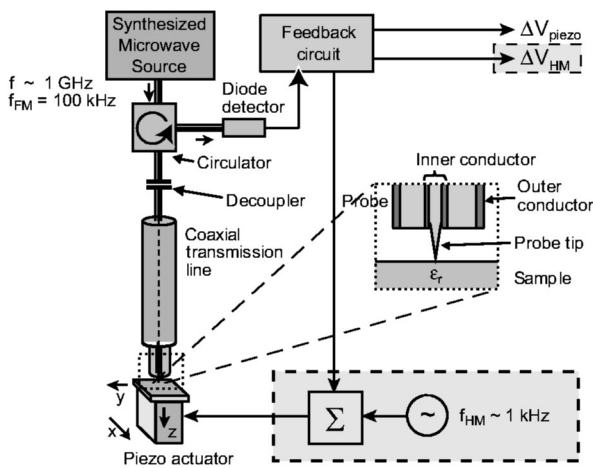
FIG. 1. Schematic of the near-field microwave microscope in the distance following mode. In the frequency following mode the feedback loop is closed through the microwave source to control its frequency. The voltage adder in the shaded rectangle is used for the height modulation and bypassed in the plain distance following mode.

FIG. 3. Schematic frequency shift vs distance curves for two different materials illustrating the origin of the height modulation signals used in the HMDF imaging mode and in the HMDF mode with tapping. Inset shows a measured height-modulation signal amplitude vs frequency shift of the microwave source for the probe above a $SrTiO_4$ crystal. Zero-frequency shift corresponds to the tip far away (5 mm) from the sample. The nominal height-modulation amplitude was 1.5 nm.

source. The sample–probe separation can be adjusted by changing the frequency of the microwave generator, and can be kept well below 1 μm.[14]

As an illustration of the effect of the source frequency instability in FF imaging, a featureless silicon wafer was scanned in three different ways using a probe with a 480-μm-diam blunt open end, as shown in Fig. 2. (The microwave frequency for all experiments described in this note is about 1.08 GHz.) The noise level is significantly reduced in DF imaging, Fig. 2(c), compared to the two FF scans shown in Figs. 2(a) and 2(b). However, such images are influenced by both topography and electrical properties of the samples, and the DF technique is most useful if either topography or electrical properties are known *a priori*, or if other quantities (such as local loss) are to be measured. The DF approach suffers also from other instabilities, including temperature-dependent changes of geometry of the scanning stage or resonator. To overcome this problem, we added a modulated probe–sample separation.[15]

The probe–sample separation was modulated with a fixed amplitude by adding an ac voltage to the voltage from the feedback circuit to the piezoactuator as shown in Fig. 1. The frequency of the height modulation f_{HM} lay between 0.5
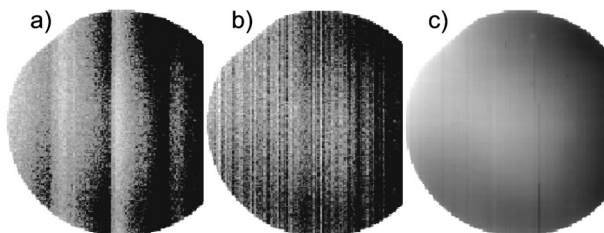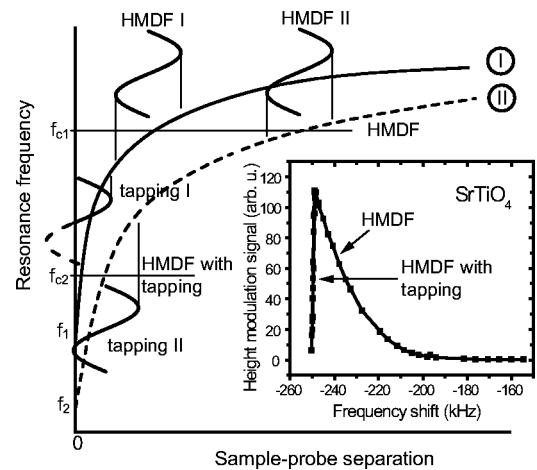

FIG. 2. Images of a featureless 2-in.-diam Si wafer taken by a probe with a 480-μm-diam blunt open end: (a) in the frequency following mode without any correction; (b) in the frequency following mode after background subtraction by calibrating each scan line with respect to a single measurement point away from the sample; and (c) in the distance following mode. Each scan was done under identical conditions (probe–sample separation, scan rate, etc.).

kHz and 1.5 kHz. The time constant $\tau \sim 1/f_{HM}$ and sensitivity of the DF feedback loop are chosen so that it follows low frequency (a few Hz) variations of the probe–sample separation caused by vibrations and topography of the sample during the scan. At the same time, the response time of the feedback loop is limited, which results in the feedback loop error signal modulated at f_{HM}. The error signal is monitored at the frequency of the height modulation using a lock-in amplifier for imaging. The height modulation signal is proportional, to first approximation, to the slope of the frequency shift versus distance curve at a given sample–probe separation, as illustrated in Fig. 3. These slopes, at a given resonance frequency, are very sensitive to local materials properties and can approach values of 1 kHz/nm near the surface [for scanning tunneling microscope (STM) -tipped probes].[16] Therefore, if the lateral dimensions of topographic features are much larger or much smaller than the probe diameter, the height modulation images are images of the local electrical properties that are free from topography interference. In addition, the height modulation approach is less susceptible to vibrational noise. We call this method height modulation distance following (HMDF) imaging.

Working with tipped probes, we can further decrease the average tip–sample separation to a value that is less than the amplitude of the height modulation while again imaging the height modulation signal. In the case corresponding to the resonance frequency f_{c2} in Fig. 3, the tip will be in contact with the sample during a part of the modulation period. The resonance frequency modulation is limited from below and the height-modulation feedback error signal will be smaller for material I than for material II in Fig. 3. This novel imaging mode is analogous to the "tapping mode" used in atomic force microscopy (AFM). In AFM, the "amount of tapping" is kept constant so that topography can be measured by keeping the probe at a constant height. Here, the (unknown) probe height is determined by the sample's physical properties, and the amount of tapping that occurs serves as a mea-
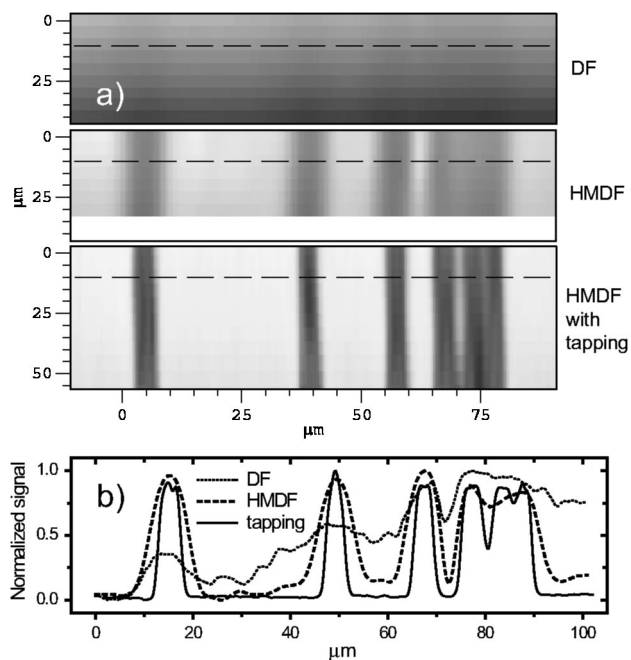
FIG. 4. (a) Comparison of images obtained in DF, HMDF, and HMDF with tapping modes from a locally B-doped Si sample. First two images were taken simultaneously. (b) Line cuts along the dashed lines in the images above.

sure of this height and thus as a measure of the local physical properties. The curve in the inset of Fig. 3 is the measured height-modulation signal above the surface of a SrTiO$_4$ crystal versus the source frequency shift, which in turn is related to the central tip–sample separation. The distance at which the tip begins to touch the sample corresponds to the maximum on the curve.

To test and compare the DF, HMDF, and tapping mode HMDF methods, a locally B-doped Si wafer was imaged. Boron was implanted at 20 keV with a dose of 10^{14} ions/cm^2, through a mask, followed by activation annealing. The doped regions are stripes of equal 2 μm width with varying spacing between them. The spacing is decreased in geometrical progression—32, 16, 8, 4, and 2 μm. Through independent STM measurements, we have determined that the sample has negligibly small topographic features. Probes for these experiments were tipped with commercial tungsten STM tips protruding 0.4 mm from the blunt open end of the resonator. The tip apex radii were about 1.0 μm.

Figure 4(a) displays DF imaging of the locally B-doped Si wafer together with the simultaneously acquired HMDF image. The height-modulation amplitude was nominally 20 nm at $f_{HM}=850$ Hz, and the average tip–sample separation was about 0.5 μm. The overall tilt of the sample (a kind of topography) precludes us from seeing the stripe-shaped doped regions in the raw DF image, although they are clearly seen in the HMDF image (no background was subtracted in either case). The images and line scans of the locally doped Si sample in Fig. 4 demonstrate a significant increase of the contrast and spatial resolution in the HMDF tapping imaging mode. The resolution in this case—slightly less than 2 μm—is dictated by the probe size. After analysis, the HMDF

mode with tapping showed about 6 dB enhancement of sensitivity and signal-to-noise ratio with twofold enhancement of spatial resolution relative to HMDF without tapping for our system. The corresponding numbers by comparison of the HMDF with tapping and DF modes are 14 dB increase of sensitivity, 17.5 dB increase of signal-to-noise ratio, and 2.3 times improvement of spatial resolution.

To summarize, it has become apparent that one of the main advantages of the FF approach is its large dynamic range (in terms of sample properties). This makes frequency following the ideal instrument operation mode for imaging an unknown sample, although it has lower sensitivity, due to drift and noise inherent to this technique. The distance following approach takes advantage of the superb stability of a synthesized microwave source improving noise characteristics of the instrument. This technique, however, is also not free from the influence of topography, and also suffers from thermal drifts in the system parts. Height-modulation distance following helps to overcome these problems. Bringing the probe in contact with the sample through a combination of height modulation and distance following at a central distance less than the height-modulation amplitude drastically improves the sensitivity and signal-to-noise ratio of images giving an enhanced spatial resolution. The advantages of this technique can be fully realized with implementation of tips able to sustain contact with the sample without damage. All of the imaging techniques described can be used in various existing microwave microscopes.

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