Waveguide ultrasonic force microscopy at 60 MHz

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We present measurements using ultrasonic force microscopy at ~60 MHz, operating in a “waveguide" mode in which the cantilever base is vibrated and flexural ultrasonic vibrations are launched down the cantilever without exciting any particular cantilever resonance. The nonlinearity of the tip-sample force-distance curve allows the conversion of a modulated ultrasonic frequency into a low frequency vibration of the cantilever, detected in a conventional atomic force microscope. Images of Ge quantum dots on a Si substrate show contrast related to elasticity and adhesion differences, and this is interpreted with the Johnson–Kendall–Roberts model of the force-distance curve. © 2000 American Institute of Physics.

Nanoscale imaging of elastic properties using scanning force microscopy has been studied intensively. Ultrasonic force microscopy (UFM) is a promising example because it can be used to map nondestructively the elastic properties of relatively stiff materials such as metals and semiconductors with standard atomic force microscope (AFM) cantilevers, by exploiting the nonlinear tip-sample force curve. The technique originally used for UFM involves vibrating the sample itself. Later it was proposed to vibrate the cantilever base, enabling the study of the effect of contact stiffness on the cantilever flexural vibrational harmonics up to 1 MHz. Then in preliminary investigations 2–100 MHz ultrasonic waves were launched down the cantilever as in a waveguide, without exciting any particular cantilever resonance (“waveguide-UFM" or WUFM). By exciting the cantilever base, the effects of ultrasonic attenuation in the sample are avoided and higher frequency operation is more easily attained. The local imaging of elastic, viscoelastic, and adhesive properties at very high frequencies is an uncharted area of research, and the dispersion of these quantities is of theoretical interest. In this letter, we present experiments and analysis of WUFM images for a sample of Ge quantum dots on a Si substrate obtained at ~60 MHz.

We use a modified commercial AFM. A U-shaped 100-μm-thick BeCu electrode is first bonded with conducting epoxy to a 36° y-cut LiNbO3 single crystal of thickness 50 μm. The opposite face of the crystal is then bonded to the cantilever chip with cyanoacrylate adhesive. The chip and crystal combination can resonate freely when the crystal is driven in its fundamental quasiextensional mode at ~64 MHz. A ceramic support is finally bonded to the BeCu electrode. The cantilever is a commercial V-shaped silicon single crystal of dimensions 180×25×0.8 μm3, with nominal stiffness 0.16 Nm⁻¹, fundamental resonance frequency 25 kHz, and tip radius ~10 nm. A saw-tooth modulated radio frequency voltage (at ~64 MHz) is fed to the transducer. The modulation frequency is chosen to be 3 kHz, well below the cantilever fundamental resonance and high enough to avoid WUFM signal distortion due to the reaction of the standard AFM feedback circuit to the ultrasonically induced displacement of the cantilever. Variations in the cantilever slope in the region of the tip—proportional to the cantilever deflection—are monitored by a standard optical lever with quadrant photodiodes. The output signal of the AFM is fed to a lock-in amplifier tuned to the modulation frequency. We checked that the ultrasonic vibration was successfully excited at the tip-sample contact by watching the decrease of the lateral force signal. Knowing the output voltage of the AFM per unit tip deflection, 5 mV/nm (to ~20% accuracy), and that a voltage of 2 mV root-mean-square (rms) at the lock-in amplifier input corresponds to 10 V at its output, quantitative interpretation of the WUFM images is possible.

A sample of Ge quantum dots on a (100) Si substrate was fabricated by the Stranski–Krastanow mode of molecular beam epitaxy. The dots are typically 200 nm in diameter and 15 nm in height. Figure 1 shows (a) the topography and (b) a simultaneously obtained WUFM image (found to be independent of scan direction) chosen for optimum contrast, obtained using a 1 Hz scan rate and a 5 nN average force. Single scans for topography and WUFM are also shown for corresponding lines on the two images. The WUFM lateral resolution, ~2 nm, is of the same order as the tip-sample contact radius. Also seen are bright haloes around the dots—related to dual contact with the tip—and an increased signal in the center of the dots—possibly related to effects of internal strain or to irregularities in adhesion and

![FIG. 1. Topography (left) and a 64 MHz WUFM image (right) of a sample of Ge quantum dots on a Si substrate. Line profiles corresponding to the arrows in the images are also shown underneath.](image-url)
the static (low frequency) cantilever stiffness, \( F(z) \) is the tip-sample force, and \( \phi \) is a constant phase shift (that does not affect the value of the integral). Equation (1) is essentially the same as that for SUFM.\(^1\)

If \( F(z) \) is obtained from standard Hertz contact mechanics, we find that the use of Eq. (1) leads to a WUFM Ge/Si image contrast of \( \sim 0.9–1 \), in disagreement with experiment. We therefore take into account both elastic and adhesive forces using the Johnson–Kendall–Roberts (JKR) model.\(^{13}\) For the tip and sample in question this model is a reasonable approximation.\(^{14}\) The contact between a sphere (of radius \( R \), Young’s modulus \( E_2 \), and Poisson’s ratio \( \nu_2 \)) and a flat surface (Young’s modulus \( E_1 \) and Poisson’s ratio \( \nu_1 \)), is characterized by the contact radius \( a \) and the indentation \( \delta \), given, in the JKR model, by

\[
a^3 = \frac{R}{K} \left[ P + 3 \pi WR + \sqrt{6 \pi WRP + (3 \pi WR)^2} \right],
\]

and

\[
\delta = \frac{a^2}{R} \left[ 1 - \frac{2}{3} \left( \frac{6 \pi W R^2}{K a} \right)^{2/3} \right],
\]

where \( P \) is the applied force, \( W \) is the work of adhesion, and \( K \) is the reduced Young’s modulus defined by \( 4L/(3K) = (1 - \nu_1^2)/E_1 + (1 - \nu_2^2)/E_2 \). Equations (1)–(3) were numerically solved for the average cantilever shift caused by the ultrasonic vibration.\(^{5,15}\) The complete modulation cycle, with maximum ultrasonic amplitude \( A = A_{\text{max}} \), is taken into account.

The inset B of Fig. 2 shows the results of the calculation of the rms cantilever shift at the modulation frequency as a function of \( A_{\text{max}} \) for (dotted lines) Ge and for (solid lines) Si (taking \( E_1 = 134 \text{ GPa}, \nu_1 = 0.21 \) for Ge; \( E_1 = 163 \text{ GPa}, \nu_1 = 0.22 \) for Si; \( k_c = 0.16 \text{ N m}^{-1} \), \( R = 10 \text{ nm} \), and with \( E_2 \) and \( \nu_2 \) equal to \( E_1 \) and \( \nu_1 \) for Si earlier). The moduli used here are orientationally averaged.\(^{16}\) The calculation was performed with the work of adhesion for the dots and substrate equal at \( W = 1.0, 2.0 \) and \( 3.0 \text{ J m}^{-2} \). These values are in a range typical of semiconductor contacts. (One should bear in mind that under ambient conditions \( W \) is a quantity that is very sensitive to oxide layer properties or adsorbed water layers.) The discontinuities in the slope of the curves correspond to the onset of an abrupt detachment of the tip from the sample during the modulation cycle.

Figure 2 shows the corresponding curves for the image contrast, that is, the ratio (dot/substrate) between the rms cantilever shifts. The difference between these shifts is also a very useful parameter in UFM, but for our results chosen under special conditions in which the contrast is particularly large, we prefer to use the ratio here. The results of these calculations can be divided into three regimes. For small ultrasonic amplitudes, where \( A_{\text{max}} \) is below \( 0.3, 0.5, \) and \( 0.7 \text{ nm} \) for \( W = 1.0, 2.0, \) and \( 3.0 \text{ J m}^{-2} \), respectively, the image contrast is approximately constant at \( \sim 0.9 \). For intermediate amplitudes, the contrast drops to give a minimum value of \( \sim 0.7 \). The two discontinuities visible in each curve arise from the tip detachment effect. At higher amplitudes above the region of these discontinuities the contrast increases. Operation in this last regime has been extensively used in SUFM.
We, however, obtained a Ge/Si image contrast of $\sim 0.25$ in experiment, which is better than any point expected from Fig. 2. This can be accounted for on the basis of the present model if differences in the work of adhesion between Si and Ge are introduced. For example, Fig. 3 shows the Ge/Si image contrast as a function of $A_{\text{max}}$ when the values of the work of adhesion of the Ge dots (in combination with the Si tip) are taken as $W_{\text{dot}} = 1.0$, 2.0, and 3.0 J m$^{-2}$, whereas that for the substrate is kept at $W_{\text{sub}} = 1$ J m$^{-2}$. For $W_{\text{dot}} = 3.0$ J m$^{-2}$, the best contrast $\sim 0.2$ is obtained in the intermediate ultrasonic amplitude regime $A_{\text{max}} \sim 0.4–0.7$ nm, a regime lying between the two discontinuities in which it the tip detaches from the substrate during each modulation cycle but not from the dots. This is evident in the calculated traces in the inset A of Fig. 2 for these values of $W$ and with $A_{\text{max}} = 0.6$ nm. One encouraging aspect of this choice of $W_{\text{dot}}$ and $W_{\text{sub}}$ is that the absolute value of the predicted signal is in reasonable agreement with experiment: the calculated rms cantilever shift is $\sim 0.03$ nm rms for Ge and $\sim 0.13$ nm rms for Si (when $A_{\text{max}} = 0.6$ nm). Further modeling showed that other pairs of values of $W$ could also lead to the same contrast (although for smaller $W$ this lead to even larger ratios $W_{\text{dot}}/W_{\text{sub}}$). Such ambiguity could be lifted by, for example, operating the experiment at two different average forces.\textsuperscript{17}

For the approximate values of elastic constants chosen, differences in work of adhesion rather than in elastic constants are the dominant factor in determining the contrast. (For $W \sim 1–3$ J m$^{-2}$ and $A_{\text{max}} \sim 0.4–0.7$ nm, $\text{dln}C/\text{dln}W \approx 2.5$ is approximately twice the value of $\text{dln}C/\text{dln}E_1$, where $C$ is the contrast.) In separate friction scans taken with the same tip, we observed a $\sim 20\%$ enhancement in the Ge signal compared to that of Si. This increase is consistent with a greater adhesion for Ge, but the $W_{\text{dot}}/W_{\text{sub}}$ ratio of $\sim 3$ that fits our results seems unusually large. An alternative explanation could be a force curve different from that of the standard JKR model, for example, if the tip end were flattened like a punch, the WUFM signal would be very different for two materials immediately after the threshold (for large signal onset) for the first (stiffer) one. Another example is the effect of viscoelasticity at the ultrasonic or modulation frequencies. Indeed, comparing these results with those at lower frequencies,\textsuperscript{4} notably absent in the present experiment is the upward jump in the waveform for Si owing to tip detachment. The WUFM waveform is dependent on the cantilever damping, the sample (or contaminant layer) elasticity and anelasticity, and the “adhesion dynamics”\textsuperscript{5} of the system.\textsuperscript{5}

This waveform is therefore difficult to model quantitatively, but such simulations and further experiments should open up the possibility for the local mapping of the dispersion in elastic constants, internal friction or adhesion on nanometer length scales in the frequency range from 100 MHz to above 1 GHz.

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\textsuperscript{9}Park Scientific Instruments, Sunnyvale, CA.; SPM model CP-M.

\textsuperscript{10}Ultralever UL-06A, Park Scientific Instruments. The plane tangential to the bottom of the tip has (100) orientation.


\textsuperscript{15}The equation for the cantilever deflection $z_0$ before the application of ultrasound is also used: $k_z z_0 = F(z_0) + k_z z_0$. We make the approximation that the feedback circuit maintains the sample at a position independent of the ultrasonic amplitude.
