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Enhanced higher-harmonic imaging in tapping-mode atomic force microscopy

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Higher-harmonics generation in a tapping-mode atomic force microscope is a consequence of the nonlinear tip-sample interaction force. The higher harmonics contain important information about the materials' nanomechanical properties. These harmonics can be significantly enhanced by driving the cantilever close to a submultiple of its resonant frequency. We present the results of enhanced higher-harmonic imaging experiments on several samples. The results indicate that enhanced higher harmonics can be utilized effectively for both material characterization and surface roughness analysis with a high signal-to-noise ratio. © *2005 American Institute of Physics*.
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Tapping mode is the most widely used dynamic operation method of the atomic force microscope.[1,2] In this mode, the tip goes into contact with the sample surface periodically. Since the tip-sample interaction force is highly nonlinear, the frequency spectrum of the cantilever contains higher harmonics. Several research groups have recognized the importance of these harmonics.[3–9] As the cantilever is excited at its resonant frequency, the higher harmonics are faced with a low transfer gain unless a higher-order flexural eigenmode is at an integer multiple of the fundamental mode. Therefore, the detection of higher harmonics with a reasonable signal-to-noise ratio (SNR) is not always possible.[10,11]

The detection problem in the conventional mode of operation has been solved by Sahin *et al.*[12] by using a special cantilever called a "harmonic cantilever." A harmonic cantilever has a special geometry that contains an eigenmode at an integer multiple of the fundamental resonance frequency. Hence, that particular higher harmonic is enhanced by the Q factor of the eigenmode for easy detection. This technique is limited to using only one higher harmonic. Recently, we proposed a method in which an ordinary cantilever is excited at or close to a submultiple of its resonant frequency to enhance any one of the higher harmonics.[13] In this letter, we present the results of our initial enhanced higher-harmonic imaging experiments on several test structures.

A schematic description of our experimental setup is shown in Fig. 1. We used two lock-in amplifiers, two synchronized signal generators, and a controller to perform the experiments. The first signal generator excites the cantilever at close to w_1/n and provides a reference signal for the first lock-in amplifier (Model SR830, Stanford Research Systems, Sunnyvale, CA) which measures the fundamental oscillation amplitude. The output of the first lock-in amplifier is fed back to the controller (NanoMagnetics Instruments Ltd., UK), which adjusts the vertical position of the piezotube. The second signal generator is used to provide a reference signal at close to w_1 to the second lock-in amplifier (Model SR844, Stanford Research Systems, Sunnyvale, CA), which measures the nth-harmonic amplitude. The resonant frequency and quality factor of the cantilever (Model No. MPA-11100, NanoDevices, Santa Barbara, CA) are measured to be $w_1 = 2\pi \times 254.4$ krad/s and $Q = 420$, respectively. The spring constant is estimated to be $k \approx 28$ N/m.

In the experiments, we chose to utilize the third harmonic to characterize the samples. The excitation frequency is selected to be $w = 0.97 w_1/3$. We did not choose to drive the cantilever exactly at its submultiple of resonant frequency ($w_1/3$) since we obtained a chaotic response for very stiff samples in our numerical simulations.[13]

Our first sample is an etched GaAs substrate. The enhanced third-harmonic image along with topography is given in Fig. 2. We see that the third harmonic does not change with topography, except at the edges of the patterns where the fundamental oscillation amplitude changes. This is what we expect since there is no material variation over the sample surface. For this experiment, we chose the free-oscillation amplitude, $A_0 \approx 1.6$ nm, and the set point amplitude, $A_1 = 1.2\, A_0$. Note that A_1 is larger than A_0. Since the cantilever is excited below the resonance, its amplitude can increase if the fundamental component of the tip-sample force is in phase with the tip oscillation.[14,15] This situation can also be interpreted by using the linearized model of Martin *et al.*[2] as a decrease in the cantilever spring constant due to positive force gradient. The third-harmonic amplitude A_3 is approximately 0.12 nm, on the average. The total noise is measured to be 0.04 Å, which yields an SNR of 30 dB. The free-oscillation amplitude for this experiment is an order of magnitude smaller than the typically chosen values. Since A_3 is proportional to A_0, we can further increase the SNR by increasing A_0.

Our second sample is a heterogeneous one, an etched photoresist (PR) on GaAs substrate. The result of enhanced third-harmonic imaging experiment with the same operating parameters is shown in Fig. 3. The third-harmonic amplitude is seen to be lower at the region of PR (squares) than at the region of GaAs. Since PR and GaAs have significantly different mechanical properties, the tip-sample interaction force is different for these materials for the same set point amplitude. Therefore, the harmonic content of the force is different and the third harmonic detects the material difference in the sample. The difference in the third-harmonic amplitude for these two materials is approximately 18 dB larger than the noise level.

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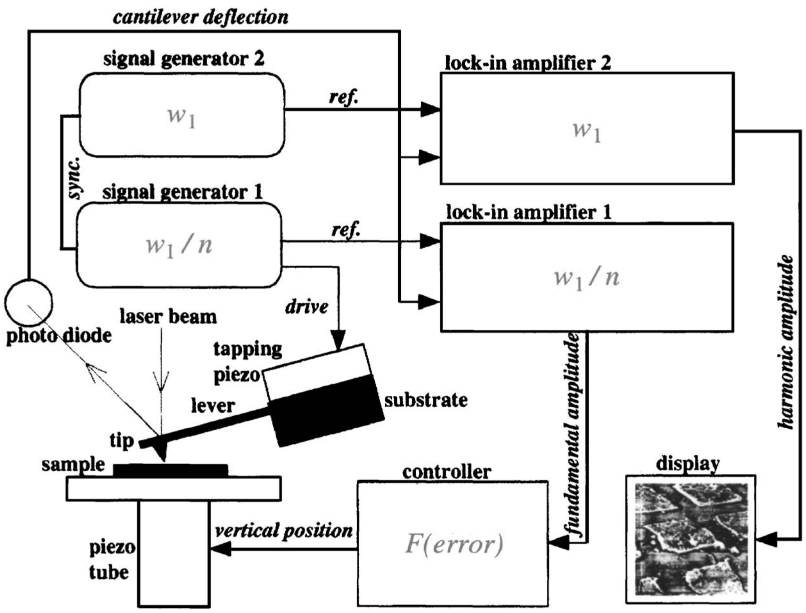

FIG. 1. Block diagram of the experimental setup.

Our last sample is an etched GaAs substrate, but its surface is not particularly clean. In the first experiment, we used a regular-patterned GaAs substrate, which has smooth steps to show that the harmonic amplitude is not influenced by the surface height. The aim of this experiment is to show how the enhanced harmonic responds to the surface roughness. For this experiment, the operating parameters are $A_0 \approx 2.1$ nm and $A_1 = 1.03\ A_0$. Figure 4 shows that the enhanced third harmonic recognizes even tiny surface features which are not easy to see in the topography image. Moreover, the average image intensity inside and outside of the rectangular areas are the same; meaning that the sample is homogeneous. Figure 5 shows the line [indicated in Fig. 4(a)] profiles of the topography and third-harmonic amplitude. We see that the average value of the third harmonic does not change. But, it shows an enhanced response for the small changes in the topography. This is a consequence of the fact that the tip-sample force depends not only on the sample properties but also on the tip-sample geometry. Hence, we can say that the enhanced higher harmonic is very sensitive to the surface

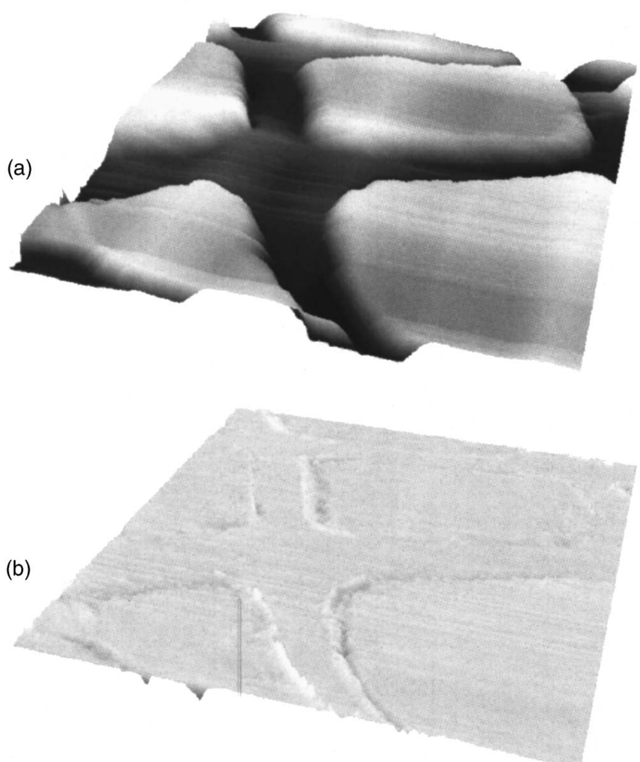

FIG. 2. 10×10 μm image of an etched GaAs substrate. (a) Topography and (b) third-harmonic amplitude. Grayscale is 340 nm in (a) and 0.54 nm in (b).

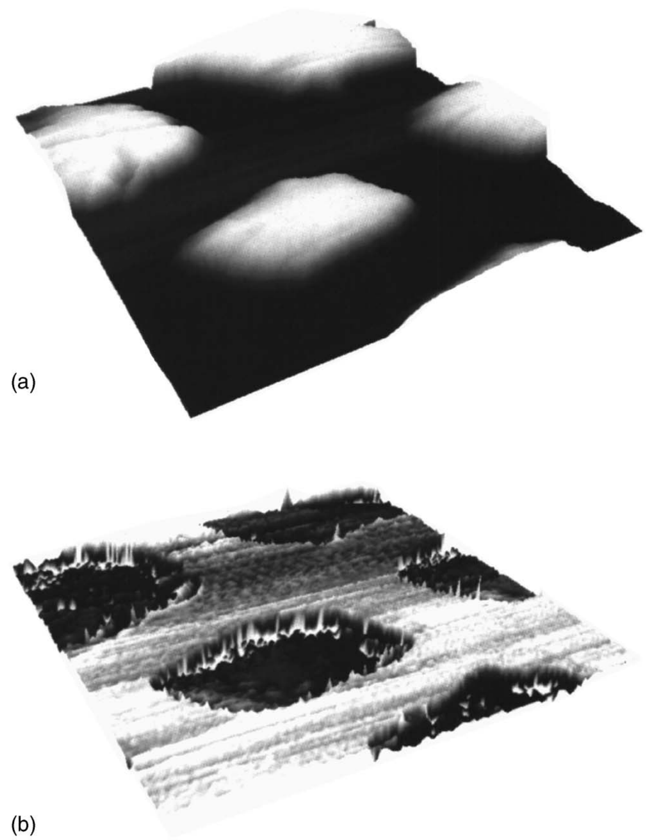

FIG. 3. 10×10 μm image of an etched photoresist (square regions) on GaAs substrate. (a) Topography and (b) third-harmonic amplitude. Grayscale is 810 nm in (a) and 0.24 nm in (b).

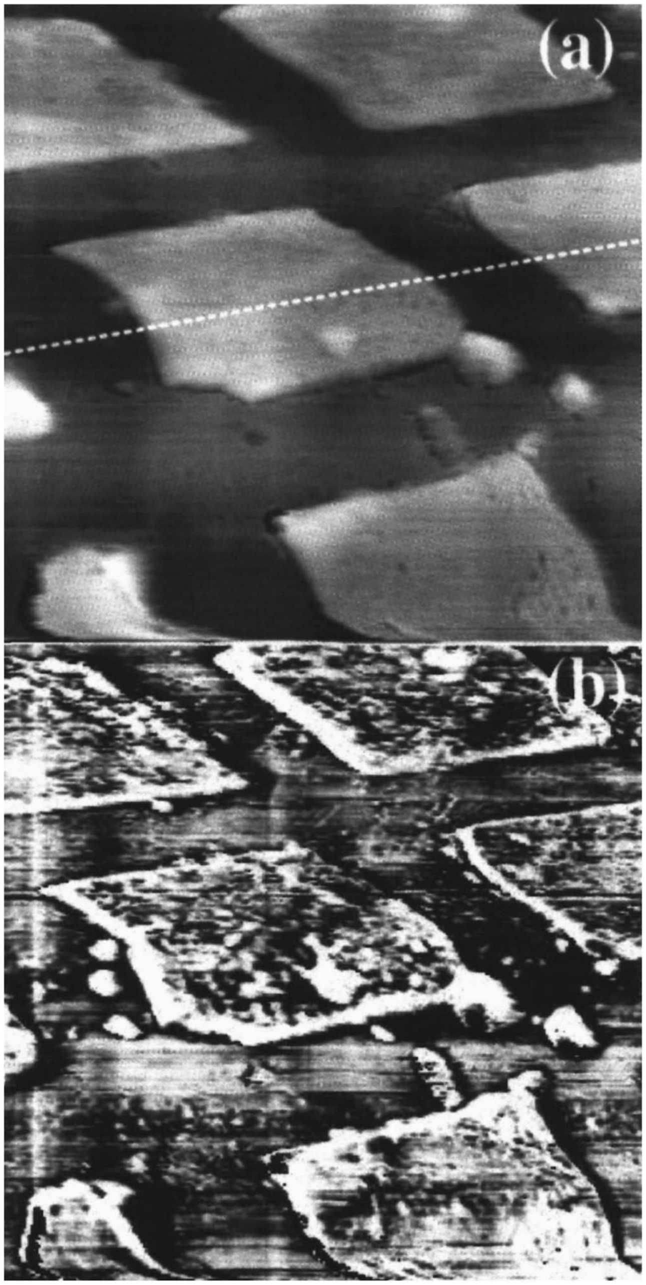

FIG. 4. (a) Topography and (b) third-harmonic amplitude. Scan size is 15 $\times 15\ \mu$m. Grayscale is 320 nm in (a) and 0.91 nm in (b).

roughness. We also note that the signal level is more than 30 dB larger than the noise level.

In summary, the initial experiments of enhanced higher-harmonic imaging pointed out that the amplitude of enhanced third harmonic changes if there is a material difference on the sample surface. If the material uniformity does not change through the surface, then the amplitude of enhanced third harmonic is found to be constant unless the tip-sample contact geometry changes. The proposed method is simple and can be easily adapted to commercial tapping-mode setups by an additional lock-in amplifier. Hence, one can utilize the enhanced harmonic imaging technique to map mechanically heterogeneous regions or to analyze the surface roughness at the nanoscale effectively.

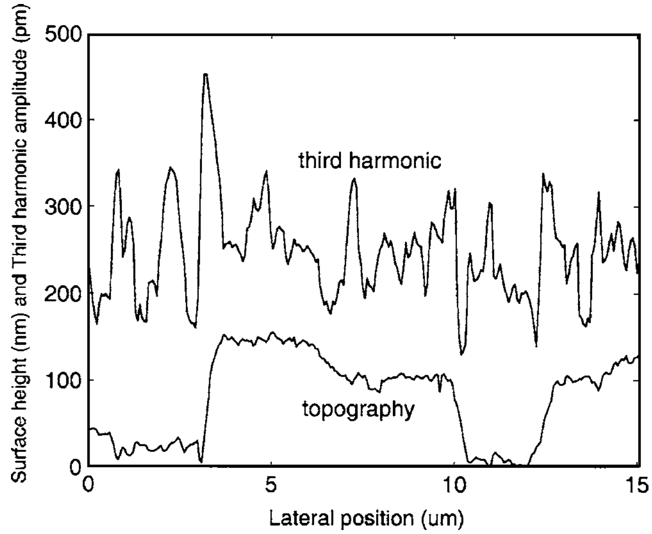

FIG. 5. Third-harmonic amplitude and surface topography variations across the line indicated in Fig. 4(a).

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