Acoustic microscopy by atomic force microscopy

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We have constructed an atomic force microscope enabling one to image the topography of a sample, and to monitor simultaneously ultrasonic surface vibrations in the MHz range. For detection of the distribution of the ultrasonic vibration amplitude, a part of the position-sensing light beam reflected from the cantilever is directed to an external knife-edge detector. Acoustic images taken on the surface of a wafer show a lateral resolution of about 100 nm at an ultrasonic frequency of 20 MHz.

High-resolution acoustic imaging is a powerful tool for materials investigation.[1,2] By using such techniques, elastic properties and defects in materials can be determined with a resolution given by the wavelength. Various optical schemes have been developed in order to detect the displacements of surface, longitudinal, and shear acoustic waves,[3] either for local analysis or combined with a scanning technique such as scanning laser acoustic microscopy.[4] But due to Abbe's principle a lateral resolution not better than about a wavelength is obtained in techniques where focused beams are used. This limit can only be surmounted by using near-field techniques. Recently, it has been successfully demonstrated that the high lateral resolution of near-field techniques can be exploited for the detection of acoustic waves by using a scanning tunneling microscope (STM),[5–9] and even images have been obtained,[9,10] but up to now detection of ultrasound by atomic force microscopy (AFM) has not been reported. Vibrating the sample at frequencies below the resonance frequency of the cantilever is used for elasticity mapping with AFM,[11] and is a standard technique in noncontact scanning force microscopy.[12] With the technique used in our experiment, however, vibrations at ultrasonic frequencies (5–20 MHz) are detected by an AFM well above the cantilever resonance frequency of several kHz.

A sample to be examined is attached by a coupling medium onto a delay line of an ultrasonic transducer (20 MHz) which is fixed on top of the AFM scanner (Fig. 1). Longitudinal ultrasonic waves are generated which are reflected at the sample surface causing vertical displacements. The AFM (Nanoscope III, manufactured by Digital Instruments, Santa Barbara, CA) works in contact mode with a beam deflection position sensor. The cantilever is a standard Si_3N_4 microfabricated triangular cantilever with pyramidal tip of approximately a 50-nm radius. We modified the instrument; by an optical beam splitter, half of the intensity of the light beam reflected from the cantilever is coupled to an external knife-edge detector using a fast photodetector (Si-*pin* diode, rise time 1 ns), while the undeviated part is used for topography imaging. Knife-edge detection is well known for optical detection of surface acoustic waves.[13] Other types of sensors which allow an absolute calibration of the cantilever vibration amplitude, like interferometers or capacitive detectors could also be used.

The A scan after 60-dB amplification (Fig. 2) shows first a small signal (1) due to direct electrical pick-up of the exciting spike (400 V, 15-ns rise time). The first ultrasonic signal (2) follows after a delay given by the delay line, the sample, and the tip of the cantilever. It is followed by a series of echoes caused by multiple reflections and mode conversion. Signal-to-noise ratio is 22 dB in a detection bandwidth of 27 MHz. To evaluate the amplitude of the first ultrasonic signal, it is fed into a boxcar integrator (Fig. 3) yielding a voltage proportional to the amplitude of the selected pulse. A gate from a pulse generator was used and was delayed such that it coincides in time and duration with this first ultrasonic signal [(2), Fig. 2]. The integrated signal is stored in the second input channel of the microscope electronics. It would also be possible to digitize the ultrasonic signal and display the peak value or any other suitable signal.

The experimental procedure is as follows: in absence of tip–sample interaction, the laser diode beam is centered onto the cantilever. The position sensor and the knife-edge detector are adjusted to their most sensitive working point. The sample is approached to the tip and the feedback loop is activated. Then an image is taken. The feedback loop maintains a constant low interaction force during the scan, also keeping the working point of the external knife-edge detector constant. A special vibration isolation is not necessary because mechanical vibrations are well below the 3-dB bandwidth of the knife-edge detector (1–28 MHz).

Figure 4(a) shows an unfiltered image of the ultrasonic

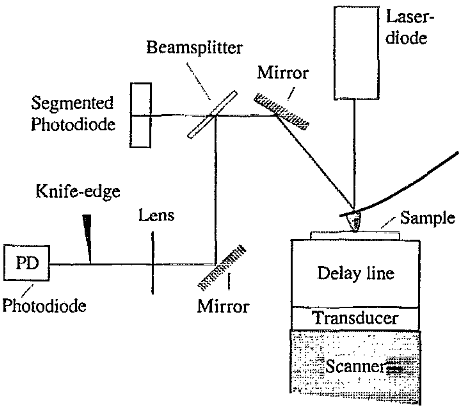

FIG. 1. Modified AFM for detection of ultrasonic vibrations. By the beam splitter half of the intensity of the light beam reflected from the cantilever is directed via a mirror to an external knife-edge detector with a photodiode (PD) which is used for detection of the ultrasonic signal.

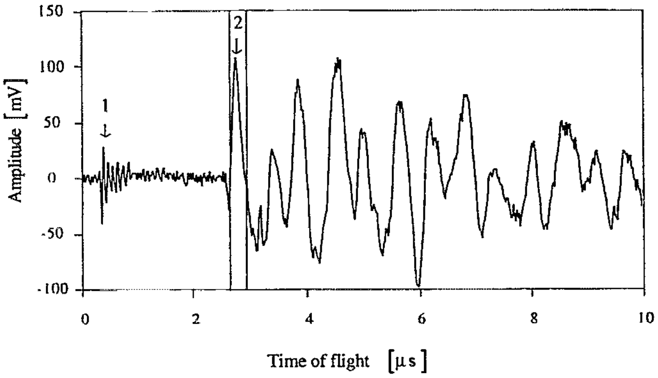

FIG. 2. Signal obtained by the cantilever knife-edge detector. After a delay time an ultrasonic signal and various echoes are received. Signal-to-noise ratio is 22 dB. The first signal, received (2) was used for image buildup (indicated by the thin solid lines. Electrical pick-up signals from the transmitter are also present. (1).

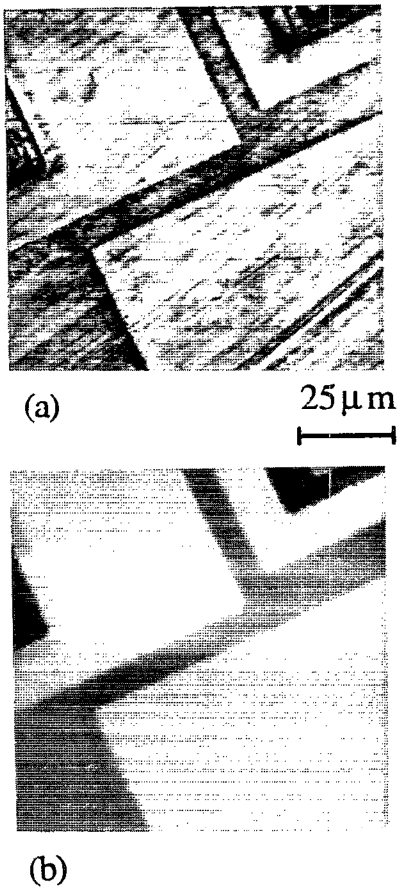

FIG. 4. Images taken of a Si wafer. Signals used for imaging are (a) integrated ultrasonic amplitude, the gray scale covers 0.3 V, color of the largest amplitudes is white, (b) topography, gray scale covers 1 μm from dark (low) to light (high).

amplitude distribution obtained on a Si wafer. An offset of 9 V calculated from the mean value of the raw data was subtracted. The gray scale is from 0 V (black, smallest ultrasonic amplitudes) to 0.3 V (white). The scan size is 100×100 μm^2, the scan rate was 1 Hz and 512 points per line were sampled. The repetition frequency was adjusted to be at least $1/\Delta t$, where Δt is the time interval between two image points. the horizontal lines are typical artefacts of AFM scans probably due to material transfer between tip and sample. All other features are reproducible. The corresponding topography image is shown in Fig. 4(b). The gray scale covers 1 μm of corrugation. As can be seem from an image taken with a smaller scan size (3×3 μm^2, in Fig. 5) the smallest features which can be resolved are about 100 nm large, which is comparable to the tip diameter. We also took a 10×10-μm^2 image on a freshly cleaved mica surface and the mean ultrasonic amplitude was comparable to the one on the wafer. As the surface was absolutely flat and homogeneous, we observed no change in amplitude except noise and artifacts.

High-frequency ultrasonic vibrations can be transmitted into the soft cantilever because of tip-sample interaction forces. When the tip is in contact with the sample surface it is attracted by adhesion forces, and the repulsive forces also

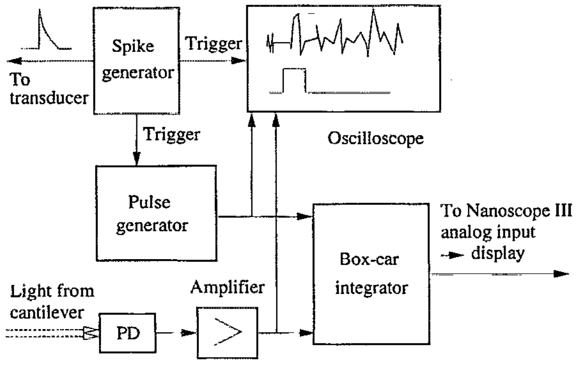

FIG. 3. Block diagram of the electronics. The signal from the photodiode is amplified by 60 dB and fed into a boxcar integrator. Its output signal is used for image buildup. The gate provided by a pulse generator is delayed such that it coincides with the first ultrasonic signal.

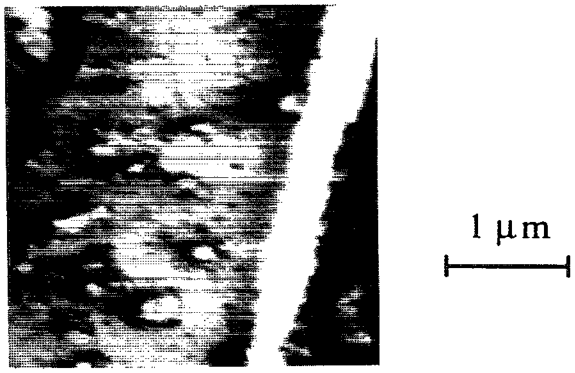

FIG. 5. Details of the wafer surface showing a lateral resolution of approximately 100 nm obtained, the gray scale covers 0.2 V.

present result from the deformation of the tip and the sample surface. The tip with the cantilever can be regarded as a point mass m suspended between two fixed surfaces by a soft spring with the spring constant k of the cantilever, and a spring with spring constant k^* given by the derivative of the tip-sample interaction forces.[12] In the AFM contact mode the tip-sample interaction force is slightly repulsive. The force derivative k^* is negative and under ambient conditions larger than k ($k \simeq 0.1$ N/m). Therefore, the resonance frequency ω_0' of the cantilever with the tip in contact with the surface is larger than the resonance frequency ω_0 of the free cantilever: $\omega_0'^2=(k-k^*)/m \gg \omega^2=k/m$. The forces increase the effective resonance frequency of the cantilever.

Apart from the wafer and mica samples mentioned above, we also carried out measurements on thin glass samples. As all samples had a thickness of less than 1 mm, the ultrasonic attenuation can be neglected and, hence, the amplitude was approximately the same for all materials. Measurements of the absolute amplitude of the detected signal would only be possible after a calibration of the knife-edge detector. However, in a separate experiment, the amplitude of the surface vibration was measured by interferometry and was approximately 5 nm on the wafer. As the scanned surface area was smaller than the ultrasonic wavelength (0.15–0.3 mm at 20 MHz), all parts of the surface region can be thought to move uniformly with the same phase and amplitude. Therefore, the changes in the detected amplitude are caused by a change of the coupling between the surface and the tip of the AFM. These differences in coupling can be due to the geometry of the surface—all edges show a decrease of the ultrasonic amplitude—a change in elastic or chemical surface properties, or even layers of adsorbate which are not well bound to the surface, as well as differences in thickness of the adsorbed water layer. A rough estimation shows that the kinetic energy which the tip and the cantilever gain during the ultrasonic vibration, might be large enough so that the tip can jump out of the water layer.

Common adhesion potentials[14] indicate that a linear approximation of the interaction force should not be sufficient for a vibration amplitude of several nm. Such large amplitudes should lead to nonlinearity in the signal. However, we could not detect such a behavior so far. This might be due to the additional adhesion caused by the adsorbed water film always present in usual laboratory air conditions, or by the large ultrasonic amplitudes employed in our measurement. Further investigations will be necessary in order to fully understand the transmission of the ultrasonic signal to the cantilever via the tip, and the image contrast obtained. It is of particular interest to clarify wether the features obtained in our images stem from local variations of elasticity, as it was observed in scanning microdeformation microscopy.[15]

In summary, we should like to stress that the monitoring of the transmission of ultrasound is an attractive method for probing of tip-sample interaction forces because of the large signal/noise ratio obtained and in order to construct acoustic microscopes yielding a resolution beyond the diffraction limit.[16,17]

Note added in proof: Recently, we became aware of research work carried out by two groups discussing the expected nonlinear transmission of ultrasound in an AFM.[18,19]

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