Surface Characterization with Nanometer Lateral Resolution Using the Vibration Modes of Atomic Force Microscope Cantilevers

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Abstract. The combination of ultrasound with atomic force microscopy (AFM) opens the high lateral resolution of scanning probe techniques to ultrasonics. Atomic force acoustic microscopy (AFAM) and lateral atomic force acoustic microscopy are techniques which use the vibration modes of AFM cantilevers. In the AFAM-mode the cantilever is vibrating in one of its flexural resonances while the sensor tip is continuously in contact with the sample surface. With this imaging technique a contrast can be obtained which depends on the elasticity of the sample surface. Quantitative values of a local elastic constant can be obtained. Shear stiffness and friction phenomena can be investigated in ultrasonic friction force microscopy by evaluating the torsional resonances of AFM cantilevers.

Introduction

In atomic force microscopy [1] a sharp sensor tip attached to a micro-fabricated cantilever is scanned over a sample surface using piezoelectric devices. Tip-sample interaction forces lead to a deflection of the cantilever being measured by an optical beam deflection detector. For topography measurements the tip-sample forces are kept constant by a feedback loop and the feedback signal to the piezoelectric positioning devices is displayed as a color coded height image. Different dynamic operation modes of the atomic force microscope are known [2]. In such modes the cantilever vibration amplitude, phase, or resonant frequency is recorded. In general, dynamic operation leads to an improved force sensitivity compared to quasi-static operation allowing one to detect smaller forces. The flexural and torsional resonance frequencies of commercial cantilevers of a few 100 µm length are predominantly in the ultrasonic range between 20 kHz and several MHz. In atomic force acoustic microscopy [3] and ultrasonic friction force microscopy [4], the sensor tip of the AFM is in contact with the sample surface while the cantilever vibrates. The radius of the tip-sample contact area which defines the lateral resolution ranges between several nm and several 10 nm. Due to the repulsive tip-sample forces which determine the mechanical boundary conditions of the cantilever, the flexural and torsional resonant frequencies of the cantilever increase. The shift of the resonant frequencies can be used to image lateral and normal sample surface stiffness and elasticity. If the amplitude of vibration is increased above a critical amplitude, the resonance curves develop plateaus or asymmetries which are typical for nonlinear oscillators. Furthermore, higher harmonics and sub-harmonics of the frequency of excitation are observed in the spectra. These effects are caused by the nonlinearity of the tip-sample interaction forces, mainly by adhesion and friction.

1. Imaging of Vibration Modes of AFM Cantilevers

We used an optical Michelson-heterodyne interferometer to analyze the modes of vibration of commercial AFM cantilevers made of crystalline silicon [5]. The cantilevers were of approximately rectangular shape, see Fig. 1. To allow handling, commercial sensor beams are fabricated in one piece with a chip of mm size to which one end of the beam is fixed. For excitation of vibration the chip of the cantilevers was glued to an ultrasonic shear wave transducer with a center frequency of a few MHz. The laser beam of the interferometer with a spot size of a few micrometers in diameter was focused onto the cantilever beam. With the help of stepper motors the surface of the cantilever was scanned, and calibrated images of vibration amplitude and phase were generated.

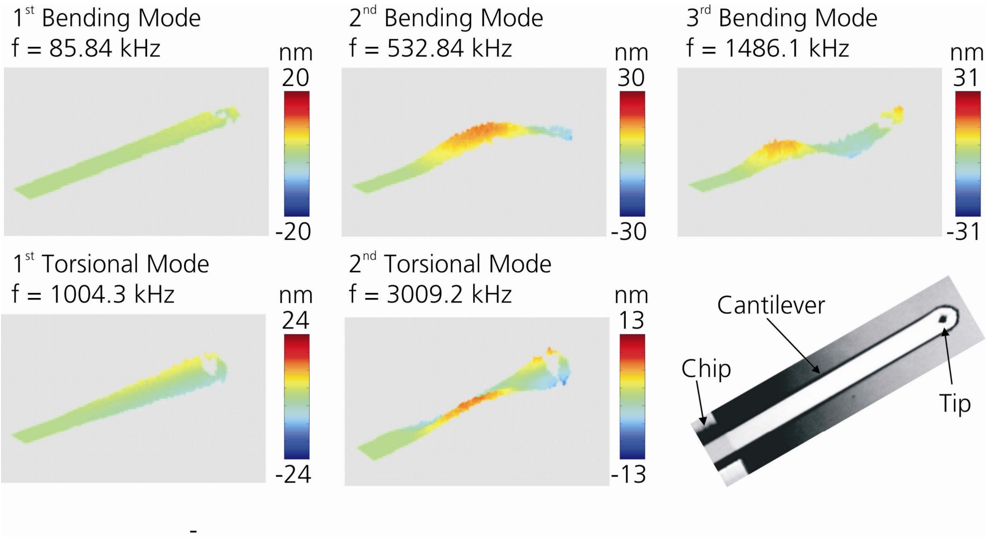


Figure 1. The first three bending modes and the first two torsional modes of a rectangular silicon cantilever measured with the optical interferometer. In each figure, the clamped end of the cantilever is in the lower left corner. An optical micrograph of the cantilever is also shown. The nominal dimensions of the examined type of cantilever are 225 × 28 × 3 μm^3 (length × width × thickness).

The resonant frequencies of the modes can be used to calculate effective dimensions of the cantilever. This yields more precise data than the geometrical dimensions given by the manufacturer. Furthermore lateral bending modes were observed. The lateral mode is a flexural mode with the deflection of the beam in the width direction. As the oscillation is perpendicular to the laser beam of the interferometer the lateral movement is not detectable. However, due to the trapezoidal shape of the cross-section of the cantilever, the lateral deflection is accompanied by upward or downward deflection that can be measured with an interferometer [6].

Apart from the theoretically expected resonances of the cantilever in the spectra, there remained often signals similar to resonances but with frequencies not theoretically expected. These spurious resonances can disturb the shapes of the resonance curves or they may cause double or multiple peaks. Measurements as well as finite element simulations showed that the additional resonances are vibration modes of the chip to which the cantilever is fixed. The resonant frequencies and the modes of the chip depend on how it is clamped or fixed. For the interferometric measurements the chip was glued to the ultrasonic transducer and its lowest resonant frequencies were in the MHz range. In commercial AFM instruments the chip is often clamped into a holder by a small spring. More than half of the chip is mounted in the holder with a smaller part protruding over its border (Figure 2). This protruding part can be excited to bending vibration with frequencies of several 100 kHz [7].

2. Contact Resonance Spectra

For a stiffness measurement, the sensor tip of the AFM is brought in contact with the sample surface with a certain static load given by the static deflection of the cantilever multiplied with its spring constant. The static deflection of the cantilever is kept constant by the feedback loop of the AFM. In the AFAM mode (Figure 2) the sample is coupled to a longitudinal wave ultrasonic transducer generating out-of-plane vibrations of the sample surface, which excite bending modes of the cantilever via the tip-sample contact. A shear-wave transducer polarized perpendicularly to the length axis of the cantilever is used to excite torsional or lateral modes of the cantilever for lateral AFAM. Alternatively, the vibrations of the cantilever can be excited at its base by a piezo element integrated in the cantilever holder [8]. Torsional modes are excited by two piezo elements in thickness mode that are driven with sinusoidal signals 180° out-of-phase [9].

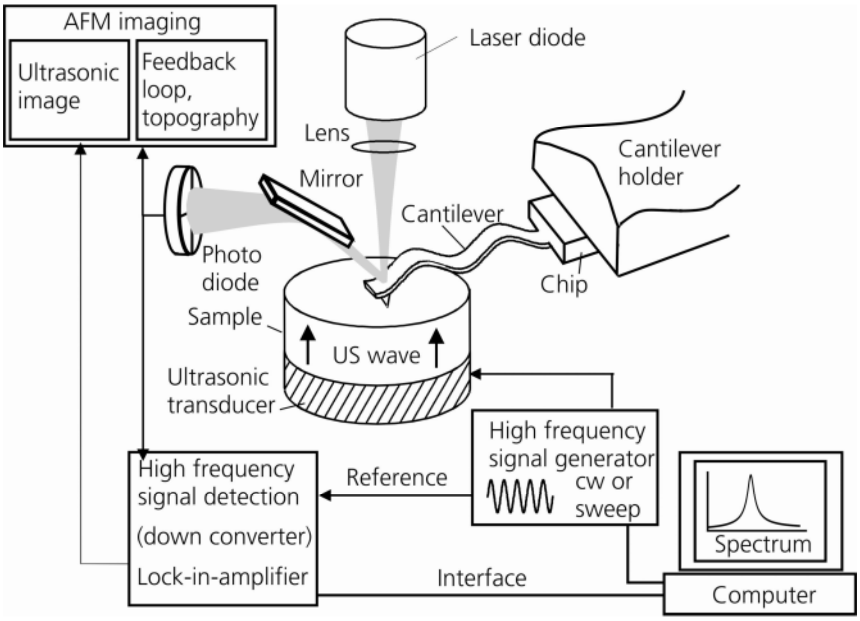


Figure 2. Set-up for AFAM or lateral AFAM imaging. An AFM with additional ultrasonic equipment is used. In the figure, the size of the cantilever is strongly exaggerated compared to the chip, the holder and the other components. An ultrasonic transducer is coupled to the sample and excites sample-surface vibrations which are transferred into the cantilever via the sensor tip. The high-frequency ultrasonic vibration is excited by a signal generator and detected by a fast lock-in-amplifier or a network analyzer.

The ultrasonic vibrations of the cantilever are evaluated either by a fast lock-in-amplifier or by a network analyzer. In the spectroscopy mode, the frequency of excitation is swept, while the sensor tip remains at one fixed position of the sample surface. Contact-resonance spectra are acquired in this way. In the imaging mode the sample surface is scanned, the feedback loop of the AFM holds the static deflection of the cantilever at a predefined value. The frequency of excitation is set close to a contact-resonance frequency and amplitude or phase of the ultrasonic vibration of the cantilever are stored. In this way an acoustic image is generated parallel to the topography image. Recently, feedback loops [10] or set-ups have been used which allow either to follow the contact resonance frequency or to store a spectrum in every image point [11].

The AFM cantilever beam is a small mechanical continuum whose resonant frequencies depend on its mechanical boundary conditions. One end of the beam is fixed while the other end carries the tip which senses interaction forces towards the sample surface. The forces normal and lateral to the surface depend on the physical and chemical properties of tip and sample, on their distance, and on the ambient conditions. Different physical forces such as Van-der-Waals forces, electrostatic forces, magnetic forces, adhesion, friction, and elastic forces can contribute to the interaction. If the tip is in contact with the sample surface with a high static load of several 100 nN or more, elastic repulsion forces dominate. The force is a nonlinear function of the tip-sample distance. However, if the amplitude of vibration of the tip is small enough, the force curve can be approximated by its derivative in the set-point, the contact stiffness. In this case the cantilever is a linear oscillator whose resonance frequencies depend on the contact stiffness, and the resonance curves exhibit a Lorentzian shape (Figure 3).

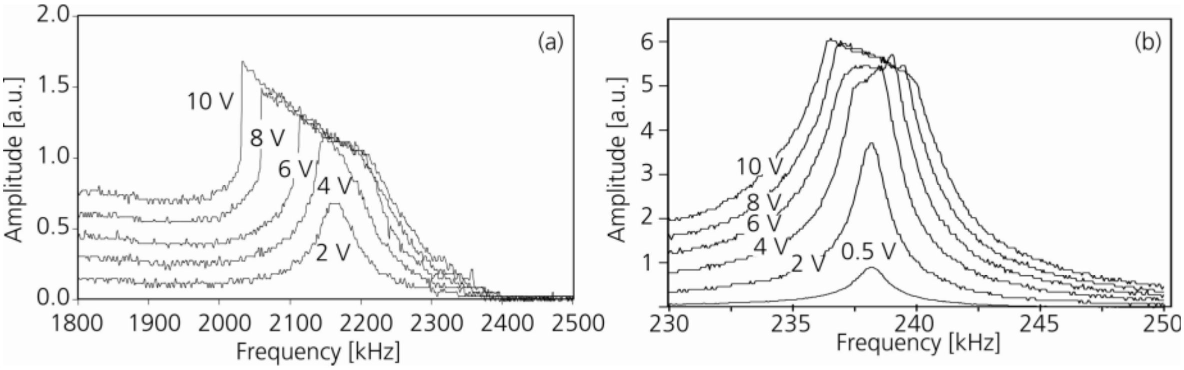


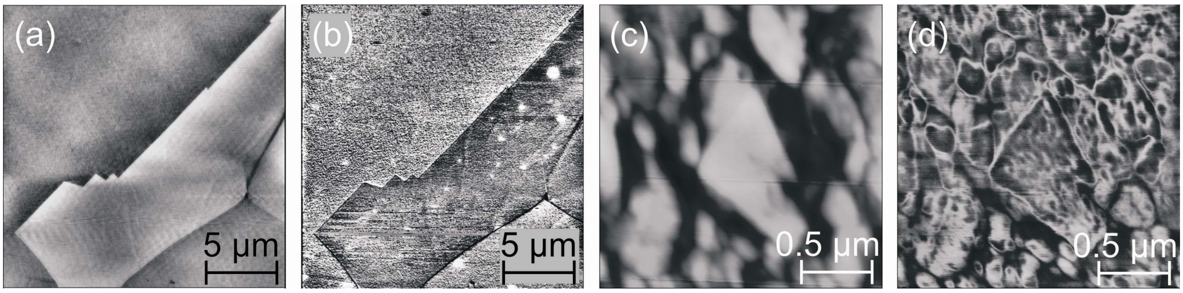
Figure 3. Contact resonances of a flexural mode excited in the AFAM mode (a) and of a torsional mode (b) with increasing amplitude of excitation. The voltages in the figures are the amplitude of the cw voltage applied to the ultrasonic transducer which is proportional to the amplitude of surface vibration.

For a quantitative measurement, free spectra and contact resonance spectra of at least two flexural modes are measured at low amplitudes. The experimental spectra are fitted to the theoretical model yielding the contact stiffness. The tip position is a free parameter in order to make the fit self-consistent. With the help of a force model, as for example Hertzian contact mechanics, an elastic constant of the sample surface can be calculated from the contact stiffness.

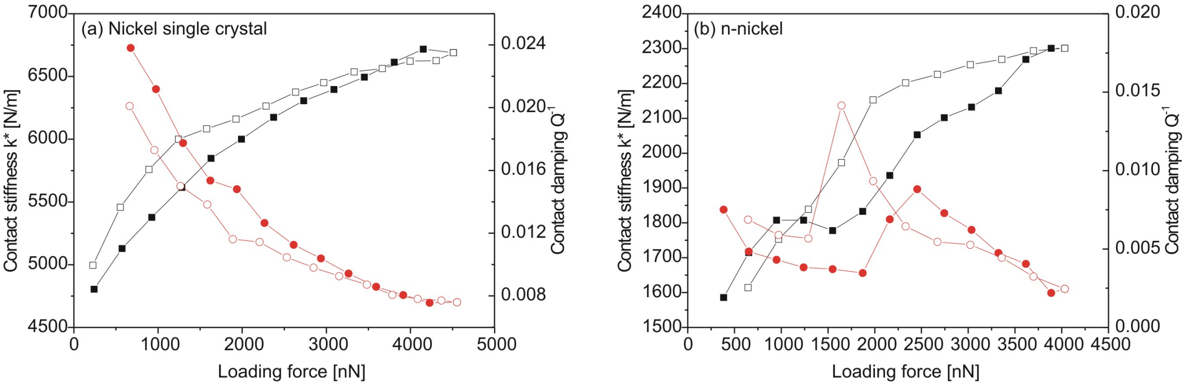
If the amplitude of vibration is increased, the nonlinearity becomes visible in the resonance curves [12]. Figure 3 shows contact resonance spectra of a flexural mode (a) and of a torsional mode (b). At low amplitude of excitation (2 V and 0.5 V, respectively) applied to the transducer below the sample the linear approximation is valid, and the resonance curve has Lorentzian shape. With successively increasing amplitude of excitation the resonance curve becomes asymmetric. In the spectra of the flexural mode (a) a steep jump develops at the left side of the curves. This behavior is typical for nonlinear resonators with softening behavior, i.e. the average contact stiffness sensed by the tip decreases with increasing tip-sample amplitude. The nonlinearity of the tip-sample force was exploited in ultrasonic force microscopy [13]. A slightly different behavior is observed for the torsional modes (b) [14]. At low lateral surface vibration amplitudes the sensor tip remains in elastic contact with the sample surface, and the cantilever behaves like a linear oscillator with viscous damping and a certain set of resonance frequencies. If the surface vibration is increased above the critical amplitude, the maximum of the resonance curves does not increase anymore and the curves develop a plateau at the highest excitation amplitudes. Numerical simulations of a nonlinear oscillator driven by a dry friction element produced curve shapes as in the experiment. This led us to the conclusion that the plateaus in the resonance curves indicate the onset of stick-slip in the relative tip-sample oscillation.

3. AFAM Experiments on Nickel Samples

Examples of topography and AFAM images of nickel samples are shown in Figures 4 (a)-(d). In the topography image of a polycrystalline nickel sample (a) grain boundaries are visible as steps. The AFAM image (b) reveals a difference in the grey levels between the different grains. The mechanical anisotropy of nickel leads to a difference in local elasticity and contact stiffness sensed by the tip. Figures 4 (c) and (d) show a topography image (c) and an AFAM image (d) of a nanocrystalline nickel sample with a grain size of 30 nm. The AFM images indicate that the grains visible form agglomerates of similar crystallographic orientation having diameters of several 100 nm [15].



Figures 4. Topography (a) and (c) and AFAM images (b) and (d) of a polycrystalline and a nanocrystalline nickel sample, respectively. The height scale of the topography images (a) and (b) is 20 nm. The AFAM images show the amplitude of vibration coded in grey levels. The AFAM images were taken at the first flexural contact resonance at a frequency close to 700 kHz.



Figures 5. Contact stiffness k* (black squares) and contact damping Q^{-1} (red circles) as a function of static loading force measured by AFAM on a (111) nickel single crystal and on nanocrystalline nickel. The loading force was first increased (closed symbols) and subsequently decreased (open symbols).

AFAM contact resonance spectroscopy measurements were made on nanocrystalline nickel, on polycrystalline nickel and on (111)-oriented crystalline nickel. Silicon cantilevers with a spring constant around 40 N/m and with a diamond coated tip were used. The static load was first increased to a predefined value of approximately 4500 nN and was subsequently decreased. Contact resonance spectra were recorded during the loading-unloading cycle. By fitting theoretical curves to the experimental spectra contact stiffness and contact damping as a function of load were obtained (Figures 5). Polycrystalline nickel and nickel single crystal showed a similar behavior. The tip-sample contact radius having dimensions of several 10 nm is much smaller than the grain size of the polycrystal. If the tip is placed at a location far from a grain boundary, the individual grain therefore behaves like a small single crystal. Figure 5 (a) shows typical loading-unloading curves. The contact stiffness increases with increasing static load due to the increase of contact area. A slight hysteresis is visible, i.e. the unloading curve does not overlap completely with the loading curve. As the contact stiffness is higher during unloading, it can be concluded that the hysteresis is due to tip-sample adhesion forces. The contact damping decreases with increasing load. The loading-unloading curves show a quite different behavior on nanocrystalline nickel. The contact stiffness shows discontinuities similar to pop-in events observed during nanoindentation experiments, and the damping increases concurrently. We believe that this is caused by dislocation nucleation and presently examine this hypothesis in more detail.

4. Summary

Flexural and torsional vibration modes of AFM cantilevers can be measured interferometrically with a high signal-to-noise ratio. The resonance frequencies and Q-values of the modes change when the tip of the cantilever is in contact with a sample surface. The contact resonances can be measured in an atomic force microscope with additional ultrasonic equipment. The sensitivity of the contact resonance spectra to tip-sample interaction can be exploited to examine the elasticity and the damping of a sample surface on a nm-scale.

Acknowledgments

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