Frequency modulation torsional resonance mode AFM on chlorite (001)

Ayhan Yurtsever, Alexander M. Gigler and Robert W. Stark

Center for NanoScience (CeNS) and Dept. Earth and Environmental Sci., Ludwig-Maximilians-Universität München, Theresienstr. 41, 80333 Munich, Germany

E-mail: stark@lrz.uni-muenchen.de

Abstract. In this paper, we discuss torsional resonance mode atomic force microscopy in frequency modulation (FM-TR-AFM) under ambient conditions. Freshly cleaved chlorite (001) exhibiting brucite-like and mica-like surface areas were investigated in constant amplitude operation in order to visualize topography and frictional properties. The measurements in frequency modulation allow the characterization of dissipative effects due to changes in the lateral forces between tip and sample.

1. Introduction

Mapping in-plane surface properties by atomic force microscopy (AFM) enables the characterization of friction, shear stiffness, and other tribologically relevant properties with nanometer resolution. Torsional resonance imaging resembles an AFM shear force experiment which is strongly linked to these tribological surface properties. In contrast to classical resonant imaging such as intermittent contact or non-contact mode, the tip is oscillating parallel (instead of normal) to the sample surface reducing peak repulsive forces. Several approaches have been demonstrated in order to achieve a laterally oscillating tip. A direct excitation and analysis of torsional resonances in contact mode allows to characterize frictional properties of hard samples [1]. Atomic steps on graphite could be resolved by an AFM sensor attached to the prong of a tuning fork [2].

For standard AFM setups, a special cantilever holder was introduced, which allows for the direct excitation of torsional resonances [3]. This improves the coupling to torsional oscillations and reduces possible cross talk between flexural and torsional vibrations. Two piezoelectric elements drive the cantilever to a small torsional oscillation. Typical Q-factors are on the order of Q = 1000. Since the twisting angle is very small, the tip oscillates nearly parallel to the surface with typical amplitudes smaller than 1 nm. Thus, the torsional resonance mode allows for the quantitative characterization of in-plane sample properties such as friction or shear stiffness [4]. By shaking the cantilever base at the torsional resonance frequency Pfeiffer et al. [5], could measure the energy dissipation on the Cu (100) surface in UHV. Kunstmann et al. [6] used frequency-modulation for a combined flexural and torsional measurement mode. They derived additional information on dissipation from the torsional oscillation response on insulating surfaces in UHV.

Frequency modulation (FM) techniques in non-contact atomic force microscopy are powerful tools to achieve atomic resolution on a great variety of samples, including semiconducting [7] or conductive [8, 9] samples. The imaging process has been investigated extensively by theory [10-12] and
In addition to imaging, FM techniques in AFM allow to study energy dissipation. The dissipation is strongly associated with the mechanical and chemical properties of the surface [16, 17]. In this work, we demonstrate frequency modulation [18] for torsional resonance mode AFM (FM-TR-AFM). Both, frequency shift and energy dissipation are measured under ambient conditions. The frequency shift provides information on the conservative interaction, while the dissipation signal also contains information on energy loss.

2. Materials and methods
All experiments were carried out on a Dimension 3100 atomic force microscope equipped with a NanoScope IV controller and a torsion resonance (TR) mode adapter (Veeco Metrology Inc., Santa Barbara, CA). Here, two dither piezos were driven out of phase in order to generate a torsional oscillation of the cantilever. Frequency modulation feedback was realized using an external phase-locked-loop unit (Nanosurf Easy PLL, Liestal, Switzerland) connected to the microscope controller via a signal access module, as shown in figure 1.

Figure 1. Scheme of the feedback circuit for torsional resonance mode in frequency modulation. The torsional vibration of the cantilever was measured as the lateral deflection signal of the segmented photodiode. The signal was amplified, phase shifted, and connected to the dither piezos for positive feedback. The frequency shift – mainly induced by conservative tip-sample interaction – is used for distance regulation. An automatic gain control (AGC) keeps the oscillation amplitude constant. Thus, the AGC error output corresponds to the local energy dissipation.

Silicon cantilevers with a flexural resonance frequency of 117 kHz and a nominal spring constant of 27 N/m were used (ZEIHR Nanosensors, Neuchatel, Switzerland). Typically, the resonant frequency of the first torsional eigenmode was 910 kHz. The torsional oscillation of the cantilever was maintained by feeding the amplified and phase shifted torsion signal to the driving piezos attached underneath the cantilever holder. The PLL proportional and integral gains allowed to track the resonant frequency while an automatic gain control (AGC) kept the oscillation amplitude constant (CA-Mode). The amplification in the amplitude control loop is adjusted by a PI controller to keep the oscillation amplitude constant. The frequency shift of the oscillation was used as a control signal for the z-feedback loop. Measurements were carried out in constant frequency shift mode, where the frequency shift of the resonant frequency of the cantilever was kept constant during FM-TR-AFM imaging. Image processing was done using WSxM [19].

A clinochlore (pennine) specimen (Mg,Fe²⁺)₅Al[(OH)₈][AlSi₃O₁₀] from Rimpfischwäng, Zermatt, Switzerland (Sample #17984 of the Bavarian Mineralogical State Collection, Munich) was cleaved. The mineral belongs to the chlorite group of phyllosilicates. It consists of alternating T-O-T (tetrahedral-octahedral-tetrahedral) and brucite-like layers. The T-O-T layer corresponds to phlogopite mica. The brucite-like surface exhibits OH groups and is slightly positively charged as compared to the mica-like layer which presents oxygen at the surface. The step height is 0.5 nm for a brucite-like layer and 1 nm for a T-O-T layer.
3. Results and Discussion

We imaged a freshly cleaved chlorite surface in TR-mode AFM using the frequency modulation technique with constant oscillation amplitude. The frequency shift data corresponds to the conservative interaction due to feedback errors while the energy dissipation image corresponds to the damping or frictional behavior of the sample. The information of the energy dissipation was extracted from the AGC feedback signal, which showed the variation in amplitude of the cantilever excitation signal. The power needed to keep the torsional oscillation amplitude constant is directly proportional to the power dissipated at the tip-sample contact. Figure 2 shows simultaneously measured maps of (a) topography, (b) frequency shift, and (c) energy dissipation as measured with the AGC on the mineral surface. The experimental data were obtained in a constant amplitude operation of the FM-TR-AFM. A cross-section of the topographic data shows that the triangular shapes are brucite flakes with a typical step-height of 0.5 nm.

As feedback control parameter, the frequency shift was used. Thus, image (b) only reveals the control error which results in a derivative picture of the topography. The cross-section shows that despite of edges, the frequency was maintained well. The excitation signal represents the energy needed to keep the torsional amplitude constant. Thus, it gives a direct measure for the dissipated energy, which shows a clear contrast for the different domains of the chlorite surface. In addition to the different mechanical properties of the T-O-T and brucite-like layers of the crystal, these are also charged differently. This surface charge density also influences the interaction between tip and surface due to corresponding changes in the surface potential. From the dissipation cross section, it is obvious that the oscillator loses more energy on the brucite flakes as compared to the T-O-T layer. This dissipative contrast might be due to different wetting properties of the respective surfaces or due to differences in the elemental friction processes at the atomic scale.

![Figure 2](image)

**Figure 2.** (a) Topographic image of the chlorite (001) surface. Image (b) shows the local detuning (z-feedback error). Image (c) shows the two dimensional mapping of the piezo excitation amplitude which was measured simultaneously with topography in ambient air. The cross sections below the respective images were extracted as indicated by the white lines.
4. Conclusions
We have demonstrated torsional resonance mode atomic force microscopy using the frequency modulation technique under ambient conditions. On a chlorite surface, image contrast between exposed T-O-T and brucite-like layers could be achieved in the dissipation signal. The fundamental mechanism of this contrast still needs to be investigated. Mapping energy dissipation in this mode opens a wide field for quantitative tribological analyses. Thus, frictional properties can be accessed, which so far have not been accessible by conventional dynamic AFM.

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