Mechanical-Diode Mode Ultrasonic Friction Force Microscopy

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Abstract. We report on a novel technique, Mechanical-Diode Mode Ultrasonic Friction Force Microscopy, based on the study of the additional ultrasound-induced torsion of a cantilever when the cantilever tip scans in contact with a sample surface at low frequency in the presence of shear surface ultrasonic vibration of sufficiently high amplitude (lateral mechanical-diode effect). Data recorded on Si(111) are discussed. The results can be understood by considering the interaction of the tip with the lateral surface sample potential, and the presence of an ultrathin viscous liquid layer at the tip-sample contact which develops hydrodynamic pressure when sheared at ultrasonic velocities.

1. Introduction

In Ultrasonic Force Microscopy (UFM), the detection of normal surface oscillations at frequencies much higher than the cantilever resonance frequency is based in the mechanical-diode (MD) effect, or static vertical cantilever displacement observed when the normal distance at a tip-sample contact is modulated at ultrasonic frequencies with sufficiently high amplitude, due to the nonlinearity of the tip-sample interaction force [1, 2]. UFM has already demonstrated its ability to provide information about nanoscale sample materials properties such as elasticity and adhesion [3]. Scanning Acoustic Force Microscopy (SAFM) [1] was implemented for the study of field amplitudes and phase velocities of Surface Acoustic Waves. SAFM has also been applied to transversal surface acoustic waves by exploiting the MD effect in the cantilever torsion due to in-plane oscillations [4,5]. Nevertheless, little is known about the physical mechanisms responsible of the cantilever behavior in this case.

Recently, Ultrasonic Friction Force Microscopy (UFFM) was proposed for the study of friction and stick-slip phenomena at ultrasonic velocities [6-9]. UFFM is based on the analysis of the torsional contact resonances of AFM (Atomic Force Microscopy) cantilevers in contact with a sample surface. Here, we demonstrate the detection of a torsional MD cantilever response when the tip of an AFM cantilever scans over a surface at low frequency in the presence of shear surface ultrasonic vibration excited from a piezo beneath the sample. Data recorded on Si(111) using different ultrasonic excitation amplitudes and tip-sample loads will be discussed. The results can be understood by considering the lateral ultrasonic force expected from interaction of the tip with the lateral surface sample potential. As in ref. [7], we have also observed a vertical lift-off of the cantilever as a result of the shear surface ultrasonic vibration. Our results are consistent with the presence of an ultrathin viscous liquid layer at the tip-sample contact which develops hydrodynamic pressure when sheared at ultrasonic velocities, as suggested previously [7].
Figure 1. MD-UFFM on Si(111). The tip is scanning forwards at low velocity, and additional shear ultrasonic vibration of ≈ 4 MHz is input to a piezo beneath the sample, modulated in amplitude with a triangular shape. The maximum ultrasonic amplitude $A_m$ is varied in each case, and the normal set-point force $F_o$ is kept constant.

2. Experiment
The experimental set-up for Mechanical Diode - Ultrasonic Friction Force Microscopy (MD-UFFM) measurements was implemented in our lab by appropriately modifying a commercial AFM (Nanotec). A shear-wave piezoelectric transducer was mounted below the sample with its polarization perpendicular to the longitudinal axis of the cantilever. The sample was bonded to the piezo using a thin layer of crystalline salol to ensure good acoustic transmission. A function generator was used to excite shear ultrasonic vibration. The ultrasonic vibration was modulated in amplitude with a triangular-shaped envelope as shown in Fig. 1, and the torsion and deflexion of the cantilever were monitored with the standard AFM four-segment photodiode. The samples consisted in small pieces of a polished Si(111) wafer, rinsed in alcohol and blown dry under $N_2$ flux. Rectangular Si cantilevers with nominal spring constants of 0.15 N m$^{-1}$ for bending and 25 N m$^{-1}$ for torsion were used.

3. Results and discussion
Fig. 1 shows typical MD-UFFM cantilever responses recorded on Si (111) while scanning forwards at low velocity, in the presence of shear ultrasonic vibration at the tip-sample contact of ≈ 4 MHz modulated in amplitude with a triangular shape. When scanning backwards, the torsional responses were the mirror image to those in Fig. 1, and the deflexion curves were identical.

In both scanning directions, the initial torsion of the cantilever diminishes as the amplitude of shear ultrasonic vibration at the tip-sample contact increases. After a critical shear ultrasonic amplitude, the MD torsional cantilever response remains flat. Fig. 2 illustrates a physical explanation for these results. In the absence of ultrasound, when scanning at low velocity on a flat surface, the cantilever is subjected to an initial torsion $t_o$ due to friction. At the typical low AFM scanning velocities, nanoscale friction proceeds by the so-called stick-slip mechanism [10]. At a sticking point, the tip is located at a minimum of the sum of the periodic surface potential and the elastic potential of the cantilever; the lateral displacement of the cantilever support relative to the sample introduces an asymmetry in the total potential that facilitates the jumping of the tip to the next energy minimum site. Most of the time the tip sticks to a surface point, and then slips to a next sticking point with some energy dissipation.
Figure 2. Physical model for MD-UFFM (see text). The surface atoms are laterally displaced due to the shear surface vibration, but due to its inertia, the cantilever cannot follow the surface lateral displacements at ultrasonic frequencies not coincident with a torsional cantilever resonance.

In Fig. 2, $E$ corresponds to the total potential acting upon the tip when scanning forwards at low velocity. Due to this potential, the tip is subjected to the force given by the derivative curve plot below. When the tip lies in the minimum energy site crossed by the dashed line, the corresponding force is zero. Due to the different time-scales, we may consider that the tip-sample potential brought about by scanning at low velocity is frozen during a shear ultrasonic vibration period. The shear ultrasonic wave transmitted through the sample introduces in-plane oscillations at the sample surface, in the direction perpendicular to the long cantilever axis. Atomic species within the tip-sample contact area will be subjected to shear ultrasonic vibration, but the inertia of the cantilever will hinder its rotation out-of-resonance. The relative lateral displacement of the surface atoms relative to the tip will lead to a time-dependent variation of the total potential acting upon the tip at ultrasonic time scales. We define the lateral ultrasonic force as the average force that acts upon the cantilever during each ultrasonic cycle in the presence of shear ultrasonic vibration,

$$F_{ult}^l(x_l, A) = \frac{1}{T_{ult}} \int_{t_0}^{t_n} F(x_l - A \cos \frac{2\pi}{T_{ult}} t) dt$$

where $x_l$ is the new lateral equilibrium location of the tip in the presence of lateral ultrasonic vibration of amplitude $A$, which defines the new equilibrium torsion of the cantilever, $A$ is the amplitude of shear ultrasonic vibration and $T_{ult}$ refers to the ultrasonic time period.

The experimental results in Fig. 1 can be quite well qualitatively explained by this model. We are currently performing numerical simulations and calibrating our instrument [11]. As the shear ultrasonic excitation amplitude is increased, the magnitude of the ultrasonic force also increases, leading to a reduction of the initial cantilever torsion. The torsion curve for the smallest shear excitation amplitude of 1.8 V in Fig. 1 clearly shows that a minimum amplitude is needed to observe a lateral mechanical diode response of the cantilever.
Figure 3. MD-UFFM on Si(111) The tip is scanning forwards and backwards at low velocity, and additional shear ultrasonic vibration of ≈ 4 MHz is input to a piezo beneath the sample, modulated in amplitude with a triangular shape. The maximum ultrasonic amplitude $A_m$ is kept constant, and the normal set-point force $F_o$ is varied in each case.

This minimum amplitude can be understood as the one needed in order that the lateral surface displacement sweeps a lateral nonlinear tip-sample force regime (see Fig. 2). The model also predicts a maximum ultrasonic force when the ultrasonic lateral displacement reaches the next zero-force lateral position. Previous studies of the cantilever response to shear sample ultrasonic vibration at the torsional cantilever resonances (UFFM) [8, 9] have indicated that sliding friction sets in for shear surface ultrasonic vibration amplitudes larger than a critical value. The flat MD-UFFM responses in Fig. 1 after a critical shear ultrasonic amplitude may well indicate a sliding regime that proceeds at a constant lateral force.

As in ref. [7], we observe a lift-off (vertical deflexion) of the cantilever as a result of the excitation of shear ultrasonic vibration at the tip-sample contact. As can be appreciated in Fig. 1, the cantilever deflexion increases linearly as the shear ultrasonic vibration amplitude is increased. Slight deviations of the linear shape of the deflexion curve are observed when the maximum deviation of the initial cantilever torsion is reached. Those deviations may be related to a coupling of the cantilever lateral and vertical motions at the onset of a sliding regime.

Fig. [3] shows MD-UFFM responses on Si(111) recorded at different normal set-point forces, including the torsion curves recorded in both forward and backward scans. From Fig. 3 it is noticeable that for higher normal loads, the magnitude of MD signal increases, and a higher critical shear ultrasonic amplitude is required to reach the flat torsion regime attributed to sliding. These results are also in agreement with the model sketched in Fig. 2. For higher loads, the magnitude of the surface interatomic potential is expected to be larger [12]. In Fig. 3, the distance between the torsion curves recorded in forward and backward scans is proportional to the magnitude of the friction force. The results indicate that friction reduces as a result of the excitation of shear ultrasonic vibration at the tip-sample contact, and that in this case friction vanishes in the flat MD torsional response regime. Physically, the onset of a lateral ultrasonic force is necessarily related to a reduction of friction (see Fig. 2). The lift-off (deflexion) signals that accompany the MD torsional response have been attributed to the presence of an ultrathin viscous liquid layer at the tip-sample contact which develops.
hydrodynamic pressure when sheared at ultrasonic velocities [7]. The shape of those lift-off curves is essentially different from the typical UFM MD deflexion response that results from the excitation of normal ultrasonic vibration [13]. The presence of a squeezed liquid layer at the Si surface - Si tip contact has been previously considered to explain a reduction of friction in ambient conditions as a result of the excitation of normal ultrasonic vibration at amplitudes not sufficiently large to break the tip-sample contact during the ultrasonic period [14]. The properties of boundary lubricants offer interesting opportunities for the control of nanoscale friction [15,16], as well as the excitation of mechanical resonances [17]. A further discussion of these points will be provided elsewhere [11].

4. Summary & Outlook

We have presented a novel procedure to study nanoscale friction and probe the surface interatomic potential and the properties of confined liquid lubricants, namely MD-UFFM. Experimental MD-UFFM data recorded in ambient conditions on Si(111) using different shear ultrasonic excitation amplitudes and set-point forces have been discussed. As in previous reports [7], we observe a lift-off (deflexion) of the cantilever as a result of the excitation of shear ultrasonic vibration at the tip-sample contact that can be explained by the presence of an ultrathin viscous layer that develops hydrodynamic pressure when sheared at ultrasonic velocities. Our results demonstrate that shear ultrasonic vibration can lead to a reduction and suppression of nanoscale friction. Promising applications of MD-UFFM include the control of friction, the study of friction at soft materials and the characterization of confined lubricants for nanoscale applications. MD-UFFM will allow us to study the dynamic frictional response to shear forces in nanostructures with surface and subsurface sensitivity.

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