NON-CONTACT FRICTION FORCE MICROSCOPY
EXPLOITING LATERAL RESONANCE ENHANCEMENT

M. LABARDI, M. ALLEGRINI
INFM and Dipartimento di Fisica, Università di Pisa, Via Buonarroti 2, I-56127 Pisa, ITALY;
e-mail: allegrin@df.unipi.it

SUMMARY
A method for sensing the dissipation occurring when a sharp atomic force microscopy (AFM) tip is oscillated laterally above a surface at distances typical of non-contact mode AFM operation is established and demonstrated. Dissipation is detected by measuring the damping of lateral resonant modes of the AFM cantilever, excited independently after the lateral resonant mode identification, when the tip is approached to the surface in the AFM non-contact or tapping regime. Preliminary images on a gold/glass sample as well as force versus separation curves give a hint of the potential of the technique. This method may provide a means of achieving sensitivity on the nanometer scale to different materials, or to adsorbate presence, even on delicate surfaces not explorable by the more invasive contact-mode friction force microscopy.

Keywords: Non-contact atomic force microscopy, Friction force microscopy, Cantilever, Resonance, Force curve.

1 INTRODUCTION
Dissipation phenomena are known to take place in close proximity mechanical interactions, even at distances of several nanometers, owing to the presence of surface contaminants or adsorbates. Atomic force microscopy (AFM) is a powerful tool for the study of tribological effects on the nanometer scale. Friction phenomena are evidenced when the sharp probe of an AFM is moved while contacting the sample surface, either laterally in a continuous way (friction force microscopy, FFM) [1] or vertically by vibrating the tip of a few nanometers (force modulation microscopy) [2]. Further evidence of such dissipative behaviour is found in shear-force microscopy [3] (exploited in near-field optical microscopy, SNOM, for the distance regulation of the optical probe) as well as in the force vs. separation (FS) curves recorded by dynamic force microscopy [4].

In order to perform a more accurate investigation, we have combined the customary technique for the obtainment of FS curves, already developed in our laboratory [5], to the exploitation of lateral resonance modes of AFM cantilevers [6]. Such modes have been recently pointed out as not suitable for distance stabilization purposes [7] in tapping-mode AFM, but on the other hand, they look convenient for a sensitive measurement of shear friction during the tip approach to the surface. A simple model that provides indications for the better exploitation of the different vibrational eigenmodes of cantilevers will be given in Sect. 3.

Similar friction measurements are usually performed in shear-force microscopy with the tapered optical fiber probes used in SNOM [3], in order to stabilize the tip to sample distance to several nanometers. The effective mass of such optical fiber tips can be consistently bigger than the one pertaining to an AFM cantilever tip, so that even higher sensitivity can be anticipated for the latter sensor. We have investigated the capability of AFM to sense material difference or adsorbate presence on a surface while operating in the non-contact mode. Such non-contact friction force microscopy (NC-FFM) may result of convenience for imaging of delicate surfaces as well as for material discrimination or adsorbate detection in ambient conditions.

2 MATERIALS AND METHODS
The instrument used is a home-made AFM/FFM [8] integrated to a commercial controller and acquisition system, operated in air at room temperature. The AFM sensors adopted are n+-doped silicon AFM cantilevers of non-contact mode type (spring constant 21-78 N/m, fundamental resonance frequency 260-410 kHz, nominal tip radius 5 – 10 nm) from Nanosensors™.

It is well known that AFM cantilevers exhibit many flexural eigenmodes [6], that can be pictorially compared to the harmonic modes of a vibrating string, the main difference being that cantilevers may have a free end or one end (the tip) clamped by some restoring force. Additionally, torsional eigenmodes of the beam are also possible. The beam motion has a direct influence on the tip location. In particular, flexural oscillations induce vertical as well as longitudinal tip displacements, while torsional oscillations induce lateral tip motion.

The sketch of our NC-FFM setup is reported in Fig. 1. To obtain the flexural and torsional eigenmode spectra of our AFM cantilevers, a special holder has been designed, able to induce preferential rotations of the cantilever chip, that we label flexural and torsional, around the x and y directions depicted in Fig. 1, respectively. Such holder is mounted in replacement of a standard AFM cantilever holder. Longitudinal and lateral excitation spectra of the cantilever are recorded with the bidirectional optical lever method [9], used in our system for the cantilever position sensing (top of Fig. 1). Mixing of longitudinal and lateral modes is present, but the comparison among spectra is sufficient for the lateral resonance identification. After the latter task is accomplished, the non-contact AFM is operated with two independent excitation frequencies $f_1$ and $f_2$. 
Figure 1: Sketch of the NC-FFM. The dashed upper rectangle indicates the AFM head, comprising a cantilever holder and the bidirectional optical lever detection system (the sample scan stage is not reported).

The usual non-contact AFM excitation at frequency $f_1$ is provided by the AFM controller, operating with the slope detection method [10], symmetrically (in phase) to both PZT slabs 1 and 2 acting on the cantilever chip in order to excite preferentially flexural resonances. In addition, the lateral resonance frequency $f_2$ is applied asymmetrically, only to PZT 1, in order to favour torsional mode excitation. Such configuration is adopted for convenience in our setup even during imaging, but in general it is not strictly necessary to provide such asymmetrical excitation to enable torsional modes. Such modes will be excited, though less effectively, also by providing normal excitation at the torsional eigenfrequency, because of inherent mixing between the two movements of bending and twisting, owing to the imperfect symmetry of the mechanical system. Non-contact imaging is thus performed while acquiring simultaneously the amplitude (or phase) of the lateral vibration, demodulated by a lock-in amplifier (Fig. 1).

Thin gold islands (10-30 nm height) deposited on glass have been used as samples and examined in ambient air. Force vs. separation curves have been traced to explore the behaviour of the shear-friction interaction as compared to the $z$-direction interaction, as a function of distance between tip and surface. Owing to the short range character of the friction forces investigated, such curves have been recorded by stabilizing the tip/sample distance in the AFM non-contact mode, and subsequently changing the setpoint of the oscillation amplitude at $f_1$. In such way, the distance is changed of very small amounts to permit the accurate exploration of the onset of frictional force in non-contact regime.

3 RESULTS AND DISCUSSION

Flexural and torsional response spectra are shown in Fig. 2(a) and (b) respectively in the frequency range of interest (550-650 kHz). Dashed plots are obtained when the excitation is asymmetrical. It is evident how the resonance at 574.6 kHz shows up preferentially with such an excitation. Other spectral features are present that on the other hand behave similarly when switching the excitation mode from symmetrical to asymmetrical. This means that the overall coupling of the excitation to the vibrating system is different, but does not provide the excitation of different modes.

An AFM image in the non-contact mode taken on a gold island deposited on glass (Fig. 3) shows, along with the topographic relief of gold (a), the presence of a different lateral vibration amplitude (b), discriminating the different materials. In particular, when the tip is located on the elevated gold island, dissipation seems reduced. Such difference may be related to the different material comprising the sample as well as to the different amount of adsorbates present on the two substances.

To investigate further this point, we have taken FS curves on the surface of the same sample. A typical approach curve is reported in Fig. 4. Trace A shows the set amplitude value (manually decreased with the passage of time by means of the AFM controller) while trace B shows the corresponding $z$-position of the sample, displaced vertically with respect to the tip by the PZT scanner of the AFM (positive values mean the sample is displaced towards the tip). Three regimes are contained in the present FS curve:

Figure 2: Excitation spectra of the bending (a) and twisting (b) movements of the cantilever.

Figure 3: Non-contact AFM topography (a) and friction (b) images on a sample of gold islands on glass. Image size is 1 x 1 µm², pixel-to-pixel resolution 24 nm.
scanning motion in the contact mode are due to the friction forces arising from constant velocity lateral flexural as well as torsional movements of the beam. The bidirectional optical lever method detects mode \[12\] as well as of observations of other groups on the basis of our previous investigations in the contact mode. Our preliminary findings can be interpreted as follows.

I. Dynamic attractive mode. The tip oscillation is high and the sample approach acts as to shift the resonance frequency to lower values. This kind of slope detection method \[10\] is very sensitive to distance changes, so that big variations in amplitude do not correspond to marked z-position changes. Imaging resolution in this operating mode is however rather low due to the long range of the forces involved.

II. Dynamic repulsive mode (or tapping-mode). The tip is in an average repulsive regime, but contacts the sample only intermittently. A well-known instability point is crossed \[11\]. Imaging resolution in this operating mode becomes higher.

III. Contact mode. The z-position of the sample is intentionally increased so to push the system in a deep contact condition. The oscillation amplitude is no longer stabilized by the feedback loop and the value read on the plot is the resulting vibration due to a profound modification of the system’s resonance spectral behaviour owing to the contact constraint.

The behaviour of the torsional vibration amplitude (trace C) and phase (D) shows an onset of dissipative behaviour in the regime of repulsive tapping (II), while the resonance is damped further when switching to contact mode (III). By adjusting the setpoint within regime II, moderate change in both lateral amplitude and phase are recorded. The image of Fig. 3 has been acquired in regime II. Imaging attempts in regime I have not lead to remarkable results.

Our preliminary findings can be interpreted as follows, on the basis of our previous investigations in the contact mode \[12\] as well as of observations of other groups \[7,13\]. The bidirectional optical lever method detects flexural as well as torsional movements of the beam. Friction forces arising from constant velocity lateral scanning motion in the contact mode are due to the phenomenon of kinetic friction \[1\], while lateral vibration due to an ac electrical excitation of ferroelectric samples (dynamic-contact electrostatic force microscopy DC-EFM \[13\]) is due instead to static friction, since the tip sticks to the surface and follows its piezoelectric sideways motion. On the other hand, the excitation of a resonance implies a grow-up of the oscillation amplitude, due to energy storing of the elastic system, and thus it turns out not to be feasible in the contact mode, where the tip constraint is strong \[12\]. An exception can be found when a small tip motion is able to excite a cantilever oscillation taking place at a different location of the elastic beam. This happens for instance for the first flexural eigenmode of AFM cantilevers in contact conditions, so that signal enhancement can be obtained by operating DC-EFM at such resonance frequency \[14\]. The same mechanism is more difficult to realize for torsional eigenmodes. When the tip is not in contact, though, resonant oscillations are able to build up. Increased sensitivity due to the resonance condition can be thus exploited for the measurement of proximity-induced friction, similarly to what made in shear-force microscopy, where a SNOM tip is oscillated laterally and the damping of such oscillation is used to regulate the distance of the sensor from the sample on the scale of tens of nanometers. The interaction mainly responsible for such damping is rather well known in ambient conditions. It is composed by a hydrodynamical damping part and a shorter range component due to surface adsorbates. Both these contributions are not present in vacuum, but evidence of a residual damping effect, not related to physical contact \[16\], suggests that different dissipation mechanisms could be present and take part also in ambient air. In the case of tapping-mode AFM, for instance, formation of liquid necks has been suggested to explain longer range dissipation \[15\]. Thus, alternative dissipation probing techniques as the one here proposed may contribute to enlighten on these controversial issues.

4 CONCLUSIONS

Friction experienced by the tip in non-contact conditions is basically different from the one in contact. It is mostly related to the presence of adsorbates or contaminants at the surface, although other effects can be also responsible for dissipation as argued for the shear-force case. The present technique could contribute to a deeper understanding of the origin of dissipation found in such nanometer scale system, that is at present still unclear and debated. Preliminary results have shown that damping of torsional cantilever eigenmodes due to shear friction forces in the AFM repulsive tapping regime can provide compositional sensitivity in the imaging of surfaces, exploitable on delicate samples not explorabile by contact techniques. The distance dependence of such shear interaction shows a sudden onset of the interaction when switching to the repulsive tapping regime, with a moderate dependence of the amount of dissipation on the amplitude setpoint. Systematic studies as a function of the set amplitude, sample composition and type of ambient (air or controlled atmosphere) must be carried.

Figure 4: Force vs. separation (FS) curve for the approach of the NC-FFM system (silicon tip on gold/glass sample). (A) Amplitude of flexural vibration at \(f_1\). (B) z-position of the sample. (C) Amplitude and (D) phase of the torsional vibration at \(f_2\).
out for a more complete understanding of the sensitivity and potential of the present technique as regards the discrimination of materials or adsorbates on a surface.

5 ACKNOWLEDGEMENTS

We gratefully acknowledge EC for financial support within the TMR Network ERBFMRXCT98-024.

6 REFERENCES


