Vibration amplitude of a tip-loaded quartz tuning fork during shear force microscopy scanning

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This Note reports on experimental results obtained with a recently published vision method for in-plane vibration measurement [Sandoz *et al.*, Rev. Sci. Instrum. **78**, 023706 (2007)]. The latter is applied to a tip-loaded quartz tuning fork frequently used in scanning probe microscopy for shear-force monitoring of the tip-sample distance. The vibration amplitude of the tip-loaded prong is compared to that of the free one and the damping induced by tip-surface interactions is measured. The tuning-fork behavior is characterized during approaches from free space to surface contact. Tip-surface contact is clearly identified by a drastic reduction in the prong vibration amplitude. However, no differences were observed between hydrophilic and hydrophobic surfaces. Experiments reported here show that the vibration amplitude of the quartz tuning fork in free space is a good estimate of the vibration amplitude of the tip interacting with the sample surface during shear force sample-tip feedback. The experimental setup for measuring the amplitude is easily integrated in an inverted microscope setup on which the shear force microscope is installed for simultaneous scanning probe and optical microscopy analysis of the sample. © *2008 American Institute of Physics.* [DOI: 10.1063/1.2965137]

Shear force microscopy (SFM) is the only scanning probe microscopy technique in which the tip-sample distance is controlled independently of the physical quantity mapped by the probe. However, the vibration of the tip induces spatial averaging and the lateral resolution is no longer solely limited by the probe tip radius but also by the tip vibration amplitude. Among the various methods presented previously for detecting the tuning-fork motion over the surface and measuring tip-sample interactions, quantitative analysis was performed on piezoelectric excitation of the vibration through a tuning fork using a voltage to vibration amplitude constant,[1,2] interferometric methods,[3] capacitive measurement,[4,5] two or four quadrant-photodiode shadow measurement,[6,7] and injection of the reflected laser beam in the laser cavity.[8] None of these techniques provide full two-dimensional (2D) description of the tip motion, since they are always based on the projection of the motion field in a given direction.

We proposed recently a technique based on image processing for in-plane vibration amplitude measurement.[9] We implemented this method on a quartz tuning fork loaded with a sharp tungsten tip as represented in Fig. 1. The prong end faces were polished and a pattern of dots was etched by focused ion beam following a 2D periodical grid. The periodical grid forms complementary spatial frequencies in the spectral domain and any grid displacement is evaluated through the spectral phase shift induced. A subpixel resolution is thus obtained in the prong displacement measurement, in the range of 10^{-2}–10^{-3} pixel by using a standard charge couples device (CCD) camera. The usual 2π phase ambiguities associated with phase measurements are removed by choosing a finite number of periods in the dot pattern and by identifying the central dot by correlation. Digital dot-pattern position measurements are thus performed on image sequences recorded during tuning-fork vibration. In-plane displacements of the prong end face are retrieved, allowing vibration amplitude measurement.

The tip-loaded tuning fork forms a high-Q device ($Q \sim 1600$) that we used as shear force probe in a scanning microscope by detecting the vibration damping due to tip-surface interactions.[1,10] The experimental setup is depicted in Fig. 2. The tip-loaded tuning fork is approached coarsely from the inspected sample by means of a Z-axis piezoelectric transducer (PZT). The sample is mounted on a three-axis closed loop PZT (PI P517) fixed on the stage of an inverted optical microscope. The latter is illuminated by means of a strobed light emitting diode (LED). The stroboscopic illumination is required since the tuning-fork frequency (~33 kHz) is outside the bandwidth of the video camera (25 frames/s). In practice the illumination frequency is shifted by 2 Hz with respect to the tuning-fork dither frequency in order to explore the prong position excursion with an apparent frequency of 2 Hz, i.e., compatible with the video rate of the CCD camera. Since we choose a transparent glass slide as inspected sample, images can be recorded from either the sample surface or the shear force probe end face simply by shifting the focus position along the Z direction.

A dual frequency synthesizer (Tektronix AFG320) delivers the driving signals for both the tuning fork and the LED. A lock-in amplifier provides the magnitude of the current output of the tuning fork as well as its phase with respect to the excitation sinewave. The lock-in outputs allow the detec-

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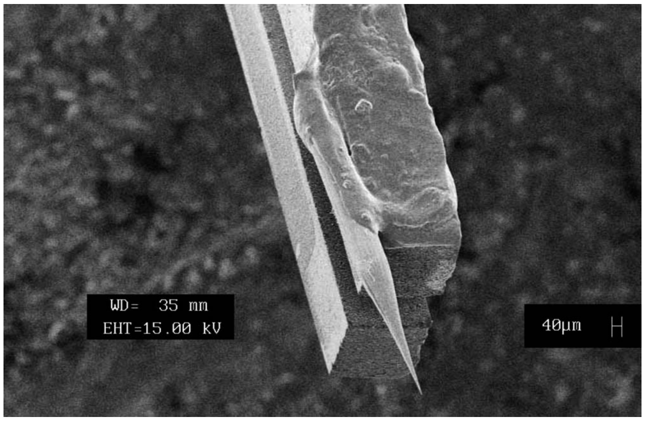

FIG. 1. Scanning electron microscopy image of the tip-loaded tuning fork.

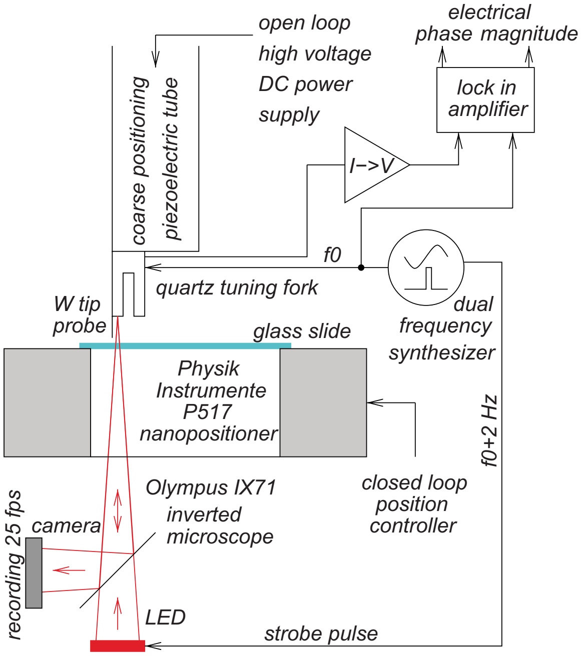

FIG. 2. (Color online) Experimental setup.

tion of the tuning-fork resonances as well as the determination of suitable setting points for shear force experiments. These outputs are also fed into a computer used for the servocontrolling of the (x,y,z) position of the three-axis PZT with nanometer resolution in order to perform lateral scanning at constant tip-sample distance.

Figure 3 shows the resonance curves obtained for the tip-loaded and unloaded prongs. As could be expected, a smaller vibration amplitude is observed for the tip-loaded one, about 85% of the free one's. The figure presents also the magnitude of the lock-in signal as well as the phase difference between the excitation signal and the current due to direct piezoelectric effect in the tuning fork. The latter signal is of practical interest since it exhibits an extremum that can be convenient for the servocontrol of the tip position.

Figure 4 presents the calibration curve of the prong vibration amplitude versus the excitation voltage. We observe a linear behavior and a constant ratio between the loaded and unloaded prongs. This means that such calibration allows the deduction in the vibration amplitude of the tip-loaded prong from the unloaded one's. Practically, this allows the observation of the unloaded prong a few millimeters aside from the shear force interaction.

Figure 5 presents the tip-loaded prong vibration amplitude observed while the shear force probe is approached to the surface until contact (approximately image 280) and then retracted (approximately image 540). The vibration damping is clearly observed, about 50% in the case of the figure. We observed that the damping level depends on the experimental conditions. Our interpretation is that because of the tip contact with the surface, the prong behaves as a double encastred beam. Then the vibration amplitude at the prong end level depends on the length and stiffness of the tip glued to the prong. Indeed we observed a damping level increase after a large number of surface approaches. Our interpretation is that tip-end damaging due to successive approaches is responsible for a harder contact with the surface.

Near field microscopy has been widely used for investigating the sensitivity of tip-substrate interactions with respect to the chemical properties of both the tip and sample surfaces.[11] Attraction and friction forces between the tip and

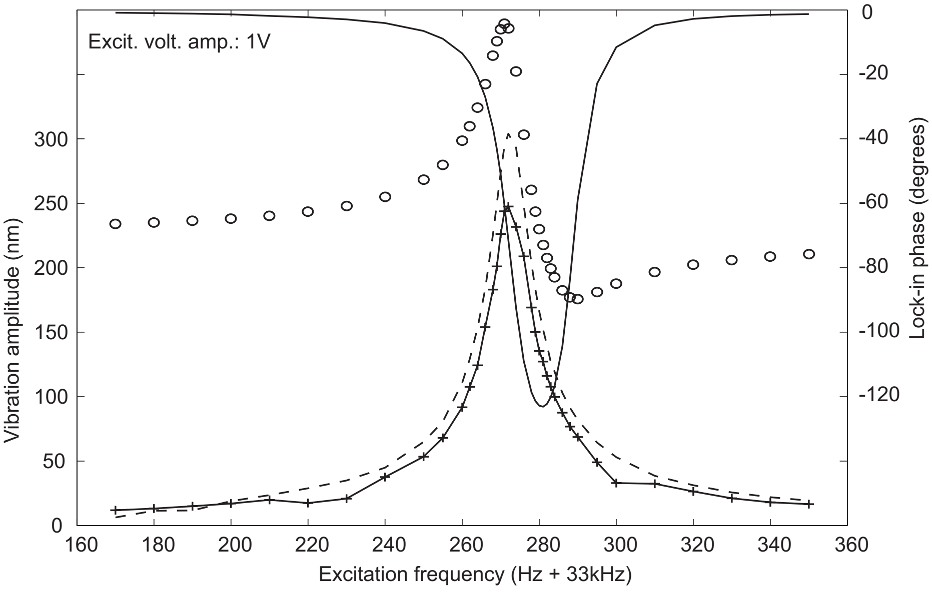

FIG. 3. Vibration and electrical characterization of a tip-loaded tuning fork around resonance. Dotted line: free prong vibration amplitude; solid line plus crosses: tip-loaded prong vibration amplitude; solid: lock-in phase (deg); circle: lock-in magnitude.

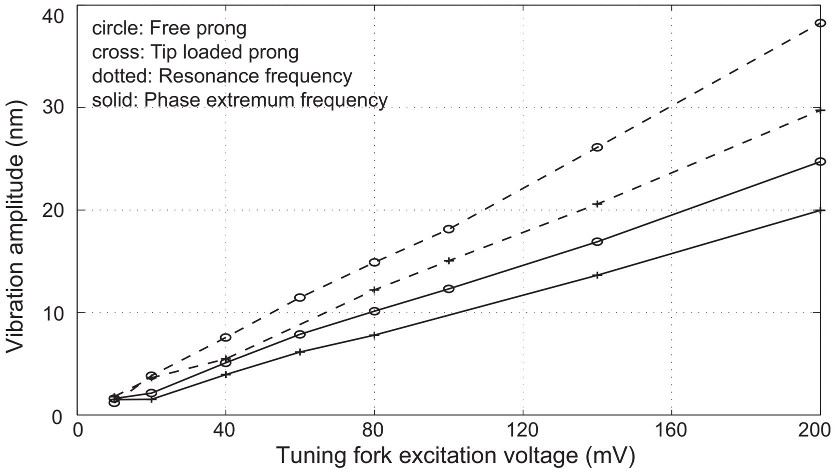

FIG. 4. Calibration of the vibration amplitude of the tip-loaded and unloaded prongs versus excitation voltage for two servocontrol configurations.

sample can be mapped at the nanometer scale and allow the local identification of surface properties.[12–14] For instance, hydrophobic and hydrophilic surfaces were clearly distinguished by lateral force microscopy (LFM).[15] We checked if our SFM was capable of such surface property discrimination. For that purpose, the surface approach-retraction experiment described above was carried out successively on glass plates with and without hydrophobic surface silanization. We did not notice any difference between the two surface types, neither in the damping level nor in the transitory regime between far field and near field. We alternated several times hydrophilic and hydrophobic plates in order to exclude any tip damage disturbance. One may point out that cantilevers used in LFM exhibit a smaller force constant [about $0.1-100\ \mathrm{N/m}$ (Ref. 15)] than tuning forks [tens of kN/m (Ref. 16)], which makes them much more sensitive to the magnitude of the surface-probe interaction forces. This difference is a sufficient reason for not discriminating the hydrophobic/hydrophilic surface types in our case.

Finally the prong vibration amplitude was measured during SEM scanning of a grating with a period of 280 nm. The tip distance was maintained by servocontrolling the phase difference between the tuning-fork excitation signal and the current induced by the direct piezoelectric effect. Figure 6 presents the results obtained in a case where the servocontrol fails in some points, as described by the phase variation diagram. In normal conditions, i.e., when the phase keeps close to the setpoint value of $-150°$, the prong vibration is as large as in free space. This indicates that tip-surface contact is avoided, as deduced from Fig. 5. However, for positions where the servocontrol fails to maintain the phase difference, the vibration amplitude is damped, indicating tip-surface contact. An excellent agreement can be noticed between both diagrams. However, the surface damage observed in the grating profile around pixel 55th does not produce a vibration damping nor a drastic phase variation. Then a tip-surface contact can be excluded in this zone.

Results presented in this Note were obtained with a homemade SFM that is not optimized at this stage. Image processing was performed on images of a dot pattern carved on each prong end with period of 3 μm. The latter is observed with a 20× objective (numercial aperture of 0.5) and a standard CCD camera. Subnanometer amplitudes can be addressed with this approach in optimized conditions, i.e., highly stable device and environment, use of a scientific grade camera, with a high magnification (40×) objective with a wider numeric aperture, and a dot-pattern period close to the diffraction limit ($<2\ \mu$m). This method is restricted to the observation of (at least partially) transparent samples but provides a quite simple and unique way of following the tuning-fork vibration amplitude during SFM scanning. Images sequences were processed *a posteriori* using MATLAB routines but the algorithms are compatible with real-time implementation.

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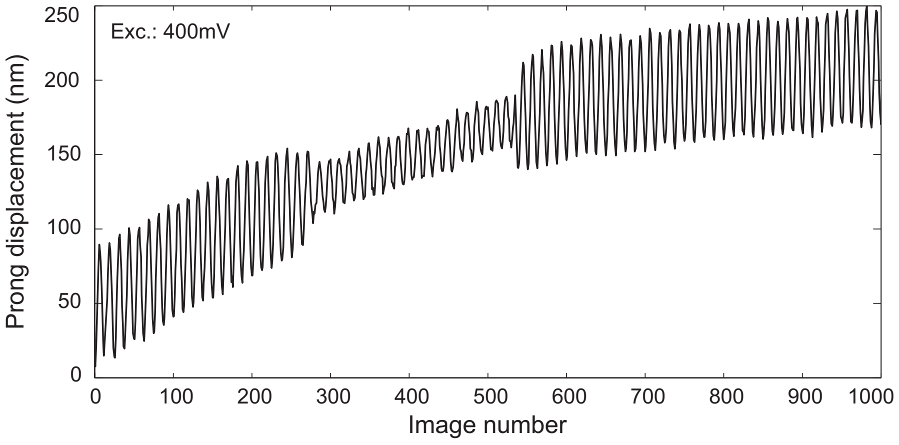

FIG. 5. Modulation of the vibration amplitude of the tip-loaded prong during a tip-surface approach and backward.

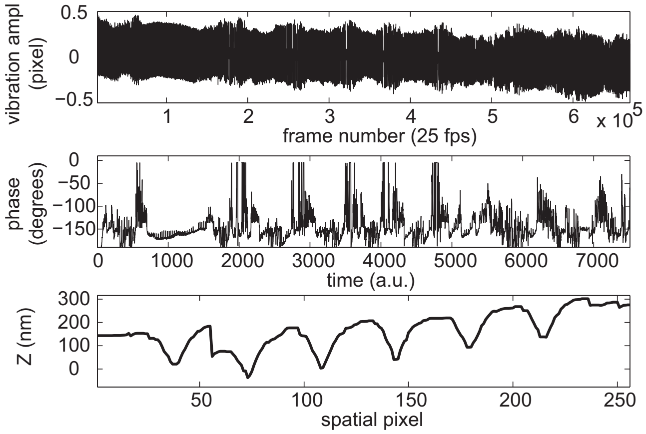

FIG. 6. Vibration control during SFM scanning of a surface grating; up: prong vibration amplitude; middle: phase variation between tuning-fork excitation voltage and induced current; down: reconstructed surface profile.

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