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# INTRODUCTION TO A QUARTZ TUNING FORK COMBINED WITH SCANNING PROBE MICROSCOPE

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*We discuss various aspects of the quartz tuning fork, ranging from its original purpose as a high quality factor resonator for use as a stable frequency reference, to more exotic applications in sensing and scanning probe microscopy. We show experimentally how to tune the quality factor by injecting energy out of phase with the current at resonance, hence tuning the sensitivity and the response time of the probe to external disturbances. The principle of shear force scanning probe microscopy is demonstrated on a simple profiler constructed with equipment available in a teaching laboratory. Furthermore, our system is developed for measuring the interaction parameters of surface nano- materials.*

## Introduction

Due to its high stability, precision, and low power consumption, the quartz crystal tuning fork has become a valuable basic component for frequency measurements. For instance, since the late 1960s, mechanical pendulum or spring based watches have largely been replaced by crystal watches, which are sufficiently stable for most daily uses. The key component of these watches is mass produced at very low cost [1].

In this paper, we will discuss the quartz tuning fork, a tiny component that includes a high quality factor resonator, which is used in a wide range of applications. A good understanding of its working principles will enable us to understand many applications to oscillators and sensors. We will show how the interpretation of the quality factor depends on the measurement technique and how an external active circuit can be used to tune the quality factor. Finally, we will demonstrate the use of the tuning fork as a force sensor and use it in some applications of scanning probe microscopy.

## Quartz tuning fork

The basic principle of the tuning fork [2–4] is well known to musicians: two prongs connected at one end make a resonator whose resonance frequency is defined by the properties of the material from which it is made and by its geometry. Although each prong can be individually considered as a first approximation to analytically determine the available resonance frequencies, the symmetry of the two prongs in a tuning fork reduces the number of possible modes with a good quality factor [3, 5].

The tuning fork appears as a metallic cylinder 8 mm in height, by 3 mm in diameter, holding a two-terminal electronic component (Fig. 1 *a*). The packaging of the tuning fork can easily be opened by using tweezers to clamp the cylinder until the bottom of the cylinder breaks. A more reproducible way to open the packaging is to use a model-making saw to cut

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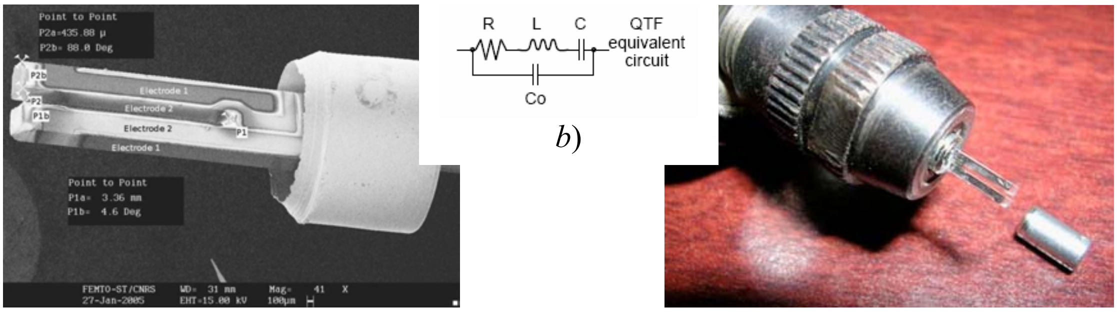
the metallic cylinder, keeping the bottom insulator as a holder to prevent the contact pins from breaking (Fig. 1 *c*).

Quartz tuning forks are primarily designed for frequency control and time base application. Furthermore, application of quartz tuning fork resonators seems to be an attractive alternative to the described conventional mass measurement techniques, since the tuning fork resonators combine the high Q-factor in air of a quartz resonator and the flexural oscillation mode of a cantilever [6]. A number of tuning fork designs were developed that exploits the mechanical resonance such as flexure, extensional, torsion and shear modes. The sensitivity of these mode frequencies to external perturbations such as mass loading, force, pressure, and temperature quartz oscillators are suitable for sensor technology [7–12].




*a*) *c*)

Fig. 1. SEM image of tuning fork displaying the layout of the electrodes (*a*). The equivalent circuit of tuning fork (*b*). A tuning fork just removed from its packing, and the metallic enclosure that would otherwise keep it under vacuum (*c*)

In our experiments, as usual, authors used a tuning forks of a commercially available type fabricated for "quartz" clocks (type 74-530-04 of ELFA Company), with the standard resonance 32768Hz, and theory quality factor Q=15000. The QTF was modeled in a standard way as a series R-L-C circuit. The R-L-C model provides a convenient electrical analog of the mechanical properties of the tuning fork. (Its mass *m*, stiffness or spring constant *k*, and damping due to internal and external dissipative forces are represented by *L*, *C*, and *R* respectively.) This model is usually further improved by the inclusion of a parallel shunt capacitance *Co* corresponding to the package capacitance (See in Fig. 1 *b*). The admittance was measured as a function of frequency using a signal synthesizer and lock-in amplifier. The theoretical spring constant is obtained from the formula $k=\frac{E}{4}w\left(\frac{t}{l}\right)^3$ [13], where $E=7,87.10^{10}\,N/m^2$ is the Young modulus of quartz. The length (*L*), thickness (*T*) and width (*W*) of the tuning fork used are 6.01, 0.35 and 0.61 mm, respectively. Using these parameters, we obtain k ≈ 7 kN/m, which agrees reasonably well with our experimental result. Furthermore, the forks are inexpensive, have high amplitude and phase sensitivity, high mechanical quality factor Q, large spring constants which allow the detection of piconewton forces and the acquisition of true atomic resolution images. The techniques for feedback control is electronic, hence greatly simplify the design and the implementation of the feedback control mechanism.

## Force sensor aspects

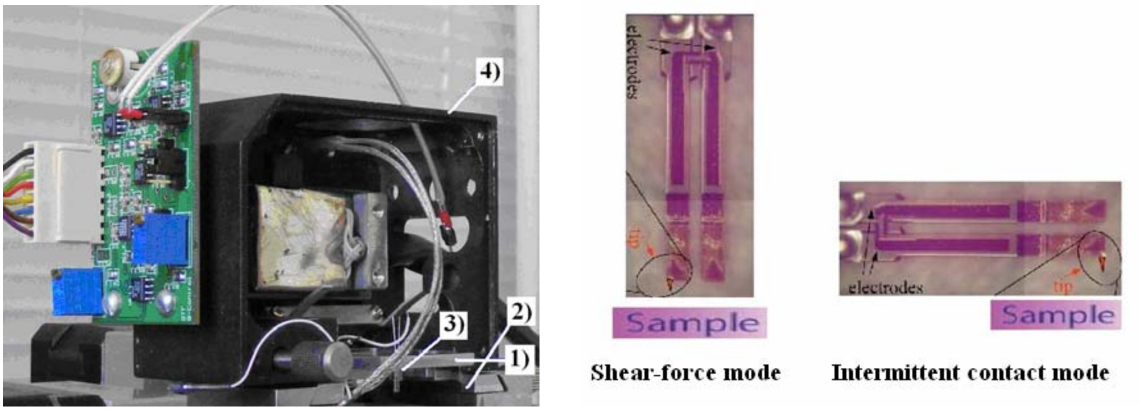
As in any case in which a stable signal that is insensitive to its environment can be obtained, we can ask how the geometry of the resonator might be disturbed to lead to a sensitive sensor. One solution for the tuning fork is to attach a probe to one prong, which is sensitive to the quantity to be measured.

Applying a force to a probe disturbs the tuning fork's resonance frequency, which can be measured with great accuracy to yield a sensitive sensor. The probe can be a tip vibrating over a surface whose topography is imaged, leading to tapping mode microscopy [14], or a shear force scanning probe microscope [15]. A topography measurement can be combined with the measurement of other physical quantities [16] such as the electrostatic force [17], magnetic force [18], or the evanescent optical field [19, 20]

We have seen that due to the vibration of the prongs of the tuning fork with a displacement component orthogonal to the sides of the prongs, a fraction of the energy stored in the resonator is dissipated at each oscillation by interaction with the surrounding viscous medium, leading to a drop in the quality factor and a sensitivity loss. Furthermore, it could influence the symmetry of tuning fork.

**Tuning fork combined with AFM NT-206 (Fork-AFM)**

Based on the described quartz tuning fork, the scanning probe microscope has been developed in our laboratory. In following, we present the mechanical part of the tuning fork that connecting to atomic force microscopy NT-206 (Microtestmachines Co.,Belarus) [21]. As shown in Fig. 2 *a*, the mechanical part consists of two major units: a holder (1) and a base plate (2). The holder is designed as the unit holding most of the mechanical components of the shear force microscope. Two fine-pitch screws are fixed to the holder in the standard arrangement for probe-sample coarse and fine approaching. The tuning fork sensor (3) as the heart of the system is attached to the holder with cyanoacrylate glue. The holder with tuning fork is placed on the base plate (2) and secured using the outside metal box (4).



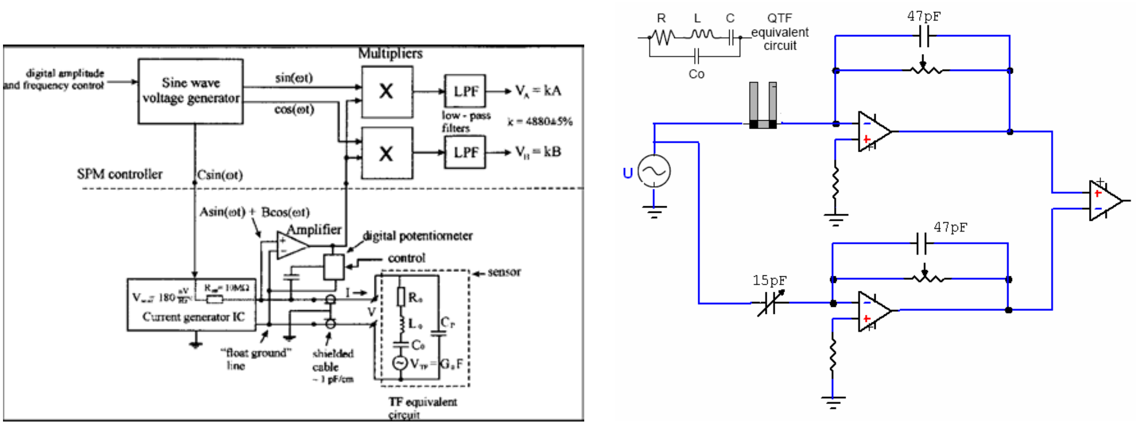

*a*) *b*)

Fig. 2. Photography of header of AFM NT-206 using a quartz tuning fork (Fork-AFM) (*a*). Principle of two operation modes: shear-force and intermittent contact mode (*b*)

In the dynamic operation modes, the tip gluing the prong is deliberately vibrated. The tip is mounted on tuning fork to allow the external excitation of an oscillation. There are two basic methods of dynamic operation: intermittent contact mode and shear-force (or lateral mode) operation. In Fig. 2 *b*, we demonstrate basic principle of shear force microscopy in ambient conditions using a quartz tuning fork in these both modes. As shown in Fig. 2 *b*, the tip is mounted perpendicular to the tuning-fork tine so that the tip oscillates normally to the sample surface. The mode used was the intermittent contact mode. The second mode is the lateral or shear-force mode. In the lateral force sensor mode, the tip is mounted parallel to the tuning fork prong and oscillates nearly parallel to the surface of sample.

For receiving the signal and controlling the oscillation from tuning fork with AFM NT-206, we have used two systems with the different principle operation. Firstly, we have used the transducer instrument of D. V. Serebryakov *et al.* [22]. We used for our experiment an improved version of the transducer electronic circuit that described in Fig 3 *a*. When using this system, we were able to achieve the high quality factor Q, simultaneously decrease the response time. However, for the transducer of D. V. Serebryakov *et al.*, because of its synchronization, it is rather difficult to repair and change.

Therefore, we have carried out the different transducer, with the principle of using voltage to control tuning fork. Because the key to implementing tuning forks for force detection is to accurately measure the fundamental resonance of the tuning fork as a function of applied force, thus this can be done either by shaking the fork at its mechanical resonance and monitoring the induced voltage or by directly driving the tuning fork with a resonant voltage and measuring the induced current. In this experiment, a standard operational amplifier circuit is used to convert the net current to a voltage (Fig. 3 *b*). The current to voltage (*I –V*) gain of the circuit has been calibrated from dc to 100 kHz. Sweeping the driving frequency f and simultaneously recording the corresponding induced voltage V, we can obtain the relationship between the frequency (f) and the output voltage (V). Under the resonant condition, the tuning fork arm has the biggest displacement that corresponds to the maximum or peak output voltage amplitude in f–V curve. Thus, the resonant frequency can be determined. The upper current-to-voltage converter will sense both the piezoelectric currents from the tuning fork oscillation and these additional stray currents. The lower op-amp in Fig. 3 *b* is not always necessary. However, it is present in order to cancel currents from stray capacitance (Co and other capacitance from wires). The lower op-amp *(I–V)* converter allows us to subtract stray currents by adjusting the variable capacitor away from the resonance.



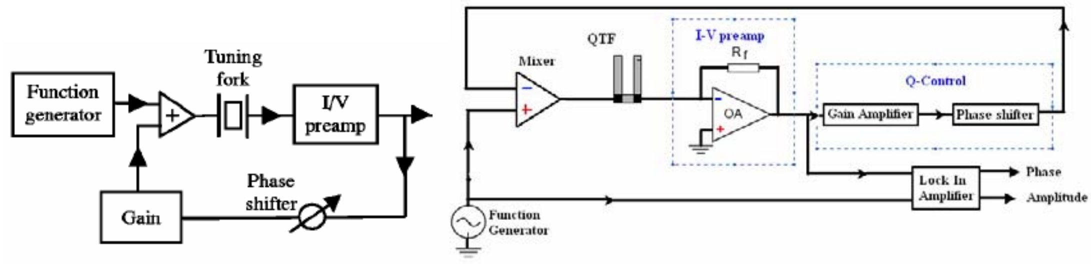

*a*) *b*)

Fig. 3. Circuit diagram of the transducer of D. V. Serebryakov et al (*a*). Essential features of tuning fork sensing system. Current through the tuning fork is converted to a voltage by an op-amp (OA). When the tip is attached to the tuning fork, the admittance due to Co can become a significant fraction compared to the RLC branch of the equivalent circuit (*b*)

## Quality factor tuning

The quality factor *Q* is widely used when discussing oscillators, because this property is useful for predicting the stability of the resulting frequency around the resonance following, for instance, the Leeson model which relates the phase fluctuations of the oscillator with the quality factor of the resonator and the noise properties of the amplifier used for running the oscillator.

We can infer that the quality factor can be increased by injecting energy into the tuning fork during each cycle. Similarly, the quality factor can be decreased by removing energy during each cycle. These two cases can be accomplished by adding a sine wave at the resonance frequency with the appropriate phase (Fig 4 *a*). However, in practice, a quartz tuning fork works at a low enough frequency to allow classical operational amplifier based circuits to be used for illustrating each step of quality factor tuning.




*a*) *b*)

Fig. 4. Principle diagram of Q-control feedback circuit (*a*). The block diagram of I–V preamplifier and the Q-control system (*b*)

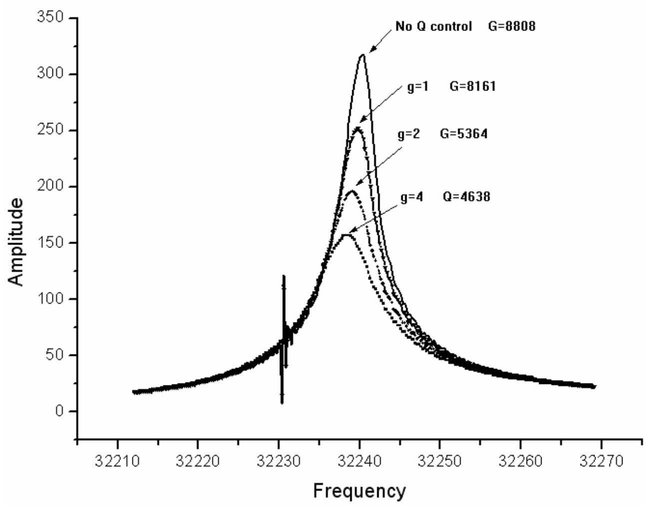



Fig. 5. Result of using Q-control, the amplitude response as a function of the driving frequency with different force feedback g under condition of the phase shift -90 degree

Figure 4 *b* illustrates a possible implementation of the circuit including an amplifier, a phase shifter, a bandpass filter, and an adder. The feedback gain defines the amount of energy fed back to the resonator during each period; the phase shift determines whether this energy is injected in phase with the resonance (quality factor increase) or in phase opposition (quality factor decrease). The resonance frequency shift is associated with a feedback loop phase that is not exactly equal to 90°. The phase shift was set manually, using a variable resistor and an oscilloscope in XY mode, until a circle was drawn by an excitation signal and by the phase-shifted signal, allowing for a small error in the setting. Figure 5 displays a measurement of quality factor decrease based on a discrete component implementation of the circuit in Fig. 4.

## Fork-AFM imaging

To make sure that the high resolution images of our system for a lot of different materials are obtained in two modes operation, we have carried out the related experimental setup in two materials: alumina ($Al_2O_3$) and hologram. We have used tungsten tips with the radius about 30-70 nm. Figures 6 *a–d* show topographical images obtained in the shear and tapping mode, respectively for alumina $Al_2O_3$ and hologram. We have succeeded in getting images with discernible bit line in both cases. However by comparing force images obtained in both shear mode (Fig. 6 *a*, *c*) and tapping mode (Fig. 6 *b*, *d*) with one type of tip, we found that a much better signal could be achieved in tapping mode operation. These results show that a tuning fork with good, sharp tip using these transducers setup can receive high-resolution images of samples in ambient conditions.

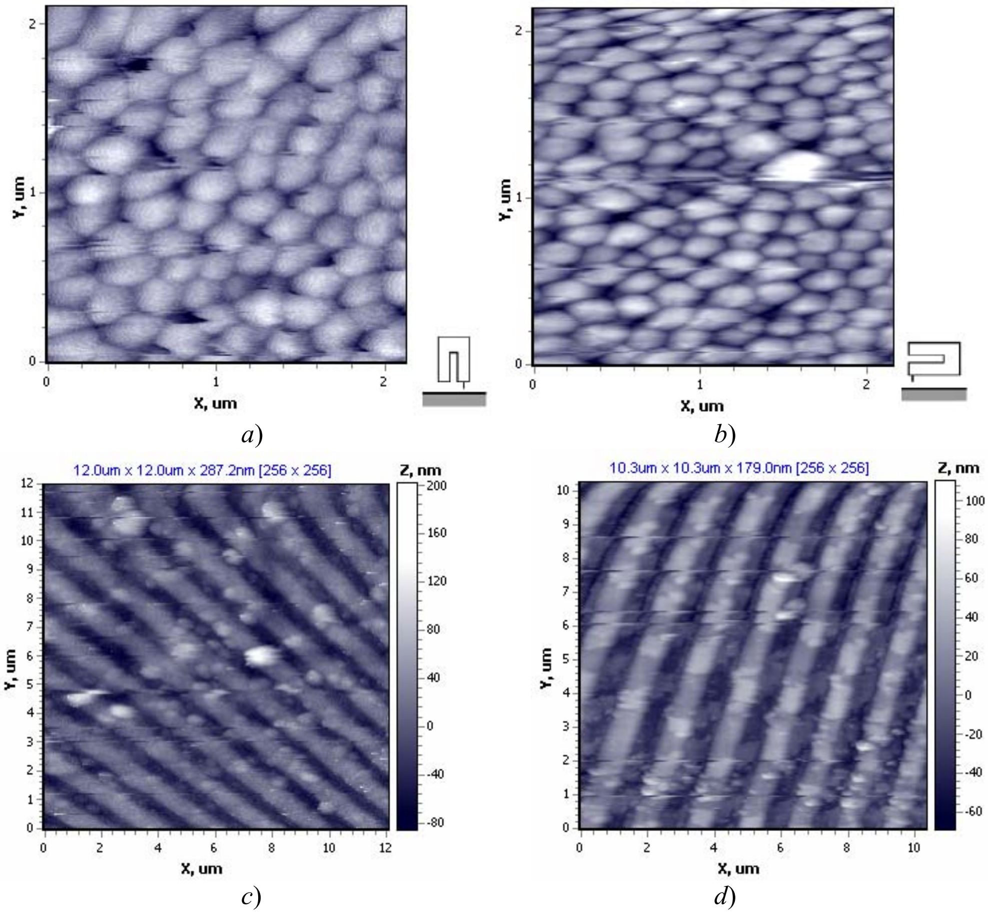
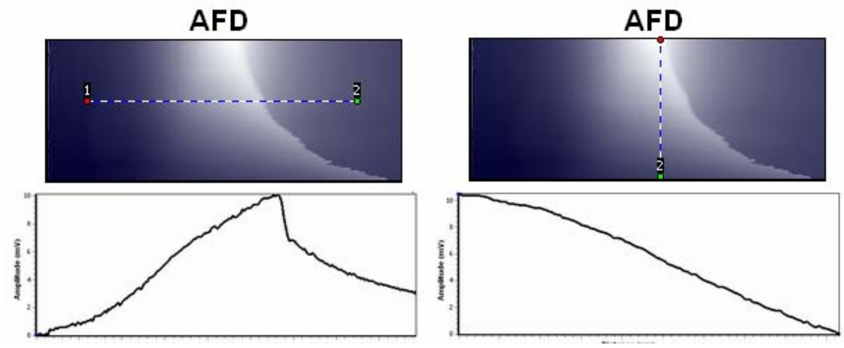



Fig. 6. Shear (*a*, *c*) and intermittent contact (*b*) mode topographical images of (*a*, *b*) alumina $Al_2O_3$, (*c*, *d*) hologram using tungsten tips. The dimension of images is noted in the figure

## Fork-AFM in measurement of the property surface of material

In following, we introduce an experimental investigation of shear mode tip-sample interactions coefficients of materials at the nanometer scale, obtained from the amplitude-phase-distance spectroscopy measurement, using quartz-crystal tuning-fork shear-force microscopy

(Fork-AFM). From our systems, the amplitude-frequency-distance characteristic was measured at during approach only or during retraction only to avoid backlash effects. In this measurement, we obtained the change of the amplitude-frequency when the tip approaches to the surface sample very slowly. Figures 7 show 2-D image taken on in order to investigate the distance dependence of these interactions on polyethylene polymer. In these images, the amplitude oscillation of tip, that corresponding to vertical axis and defined by light-scale, was shown as a function of frequency (horizontal axis) and distance between tip and sample (vertical axis).




*a*) *b*)

Fig. 7. The 2D images of characteristic of spectroscopy amplitude-frequency-distance that operating in the dynamic shear-force. The 2-D grey scale images show the amplitude of the tuning fork vs. frequency (horizontal) and distance (vertical). In (*a*) the lower graph shows the amplitude as a function of frequency at fixed distance along the line 1-2 in the grey scale image; In (*b*) the amplitude is shown as function of distance along the line 1-2 at the resonant frequency

Figure 8 shows the results of the dependency between oscillation amplitude and oscillation frequency on the distance between tungsten tip and polyethylene surface. From the measured results of amplitude and frequency, using equations that calculated in [23, 24], the damping force, the elastic force and the shear modulus and viscosity coefficients are calculated and shown in Fig. 9 *a*, *b*. Furthermore, in our study, we constructed a controllable Q-factor system, so that we could investigate the tip-sample interaction through the variation of the pre-set quality-factor (meaning the Q-factor at the free oscillation, when the tip is far from the sample surface, which we call hereafter $Q_\infty$ for short). The influence of the Q-factor on the interaction components of tip-sample is investigated. These results show that the capabilities of the combination of a tuning fork-based AFM can be extended to quantitatively analyze the properties of surface of nano materials with high precision.

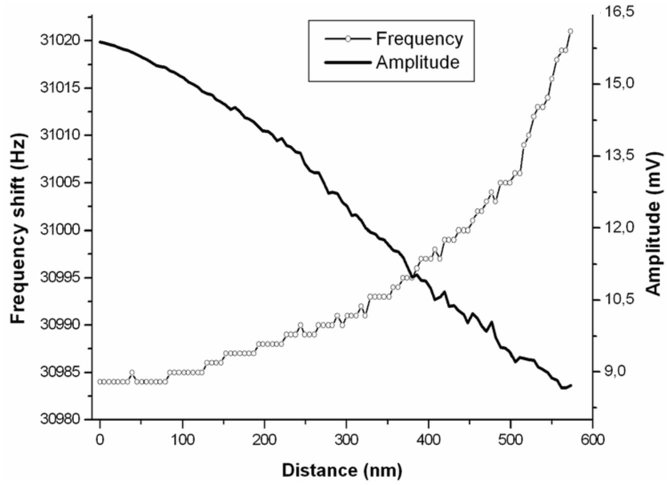
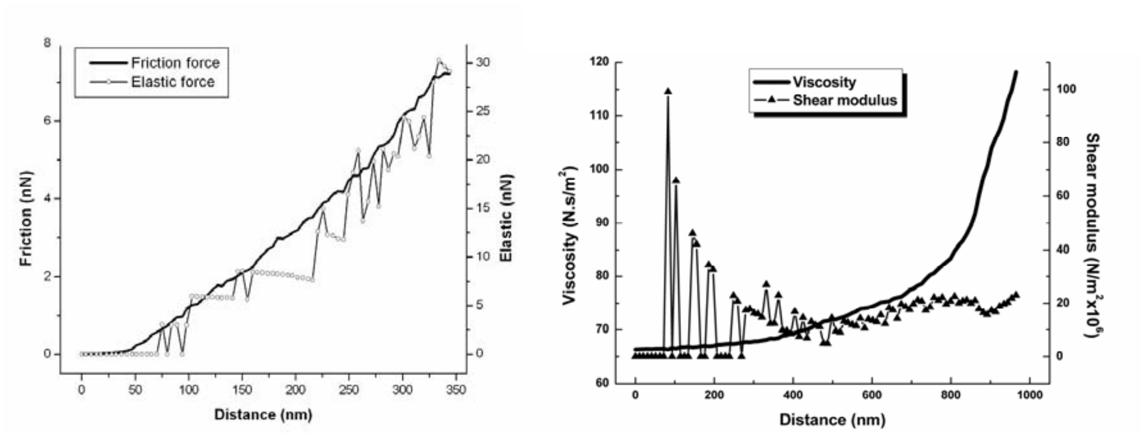



Fig. 8. The curves demonstrating the dependence of amplitude and frequency as a function of the tip-sample distance when the tip approaches to the surface of polyethylene




*a*) *b*)

Fig. 9. The curves demonstrating the dependence of *a*) force interactions (damping and elastic force), *b*) shear modulus and viscosity coefficients, as a function of the tip-sample distance when the tip approaches to the surface of polyethylene

## Discussions & Conclusions

We have discussed the quartz tuning fork, a two-terminal electronic component whose use is essential in applications requiring an accurate time reference. We have shown its basic principle when used as a high quality factor resonator packaged in vacuum. Furthermore, we then described the use of the Q-control system as a tunable quantity, which could be increased or decreased by injecting energy in phase or out of phase respectively with the input voltage. The oscillator is thus a limit condition of an infinite quality factor when the losses are compensated by the in phase injection of energy.

We have demonstrated atomic force microscopy using quartz tuning with and without Q-control fork in air conditions in two modes operation: shear force and intermittent contact modes. We can decrease the recording time and images of smaller size could be acquired well, which may allow for real time imaging and high resolution. Reproducible topographic images have been obtained on hard and soft samples. Currently we are using the two types of tips: tungsten tips and commercial Contact silicon cantilever CSC21/15 chips in the setup in order to experimentally verified that our system can expand to measure for all materials with the better resolution, especially for biological applications. Furthermore, some applications in investigation of the interaction parameters is mentioned in our study. One can also think of the combination of the tuning fork with the AFM NT-206 that allows to inexpensively implement a variety of scanning probe micros copies for investigation the properties of nanomaterials.

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