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INVESTIGATION THE STRUCTURED MATERIAL SURFACES USING THE QUARTZ TUNING FORK BASED ON AN ATOMIC FORCE MICROSCOPY WITH CONTROLLABLE Q-FACTOR IN TWO MODES OPERATION: "INTERMITTENT CONTACT" AND "SHEAR-FORCE"

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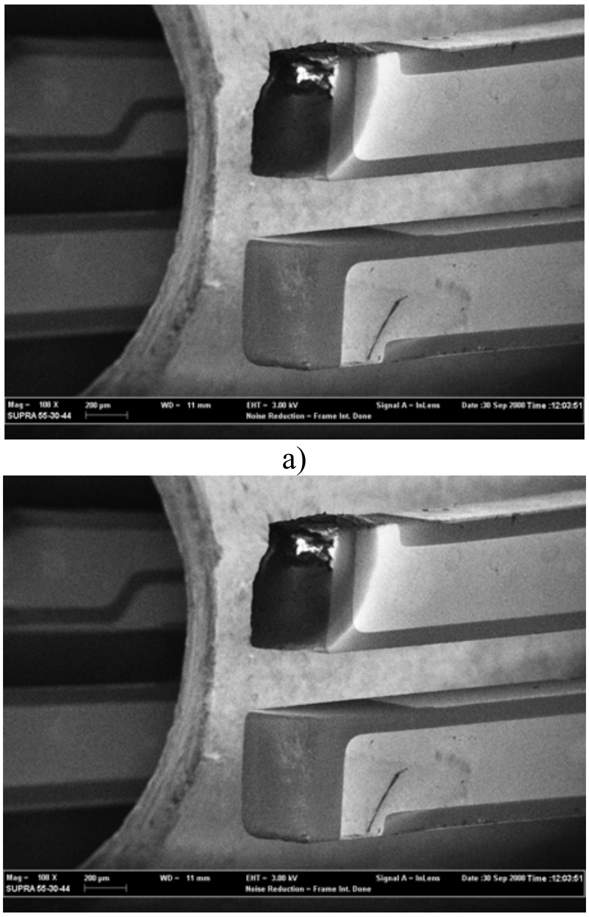
We present a combination of an atomic force microscopy with a quartz-crystal tuning fork in ambient conditions. A silicon cantilever tip was attached to one prong of the tuning fork to realize shear-force and intermittent contact mode Fork-AFM operation. By electronically adjusting the quality factor of the probe, called *Q*-control, it was possible to tune the quality factor *Q* and correspondingly change the overall scanning time. It was also seen that tuning fork with low quality factors could increase stability with the changed signal and so improve the imaging resolution. Measurements on the different samples were used to demonstrate this technique.

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 For instance, since the late 1960s, mechanical pendulum or spring based watches have largely been replaced by crystal watches, which are sufficiently stable for most daily uses.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]. Recently, tuning fork based shear force detection, as implemented in a large number of near-field scanning optical microscopes (SNOM), has proven to be an easy and reliable method by which to control the distance between the probe and sample by Karrai and Grober [2]. In following, Giessibl et al. [3] has employed them for atomic resolution AFM imaging. Tuning forks have been used as sensors at low temperatures and in high magnetic fields by Rychen et al. [4]. It is said that at this moment the applications of tuning fork are rather widespread.

Recently, electronic circuits have been developed that allow the quality factor *Q* of the cantilever to be varied in a controlled manner [5, 6]. More details on the relation between the *Q* of the probe and the microscope sensitivity can be found in the literature [5, 6, 7]. We have presented atomic force microscopy results, which use a quartz tuning fork with *Q*-control in ambient conditions, and investigated the system with *Q*-control and without *Q*-control of the tuning fork to improve the shear-force detection sensitivity with the diamond tips [8, 9]. We have also show that *Q* values of tuning forks can be decreased significantly using the technique of *Q*-control. Furthermore, with lower values of the quality factor, we can decrease the recording time for scanning images and hence, images of smaller size could be acquired faster, which may allow for real time imaging and high resolution.

In this paper, we describe an implementation of a combination between the above system *Q*-control and atomic force microscopy (AFM) NT-206 (Microtestmachines Co., Belarus) [10] based tuning fork two operation regimes: i) tapping mode and ii) shear-force mode. Also we discuss the advantages and disadvantages of these modes of operation and extend possibility of analyzing the properties of surface of nano materials with high precision and resolution.



a)

b)

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

QUARTZ TUNING FORK

A tuning fork is a simple metal two-pronged fork with the tine formed from a *U*-shaped bar of elastic material (usually steel). A tuning fork resonates at a specific constant pitch when set vibrating by striking it against a surface or with an object, and after waiting a moment to allow some high overtones to die out. The pitch that a particular tuning fork generates depends on the length of the two prongs, with two nodes near the bend of the *U* [11 – 13].

The tuning fork appears as a metallic cylinder 8 mm in height, by 3 mm in diameter, holding a two-terminal electronic component (fig. 1a). 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 the metallic cylinder, keeping the bottom insulator as a holder to prevent the contact pins from breaking (fig. 1b).

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 [14]. 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 [15 – 18].

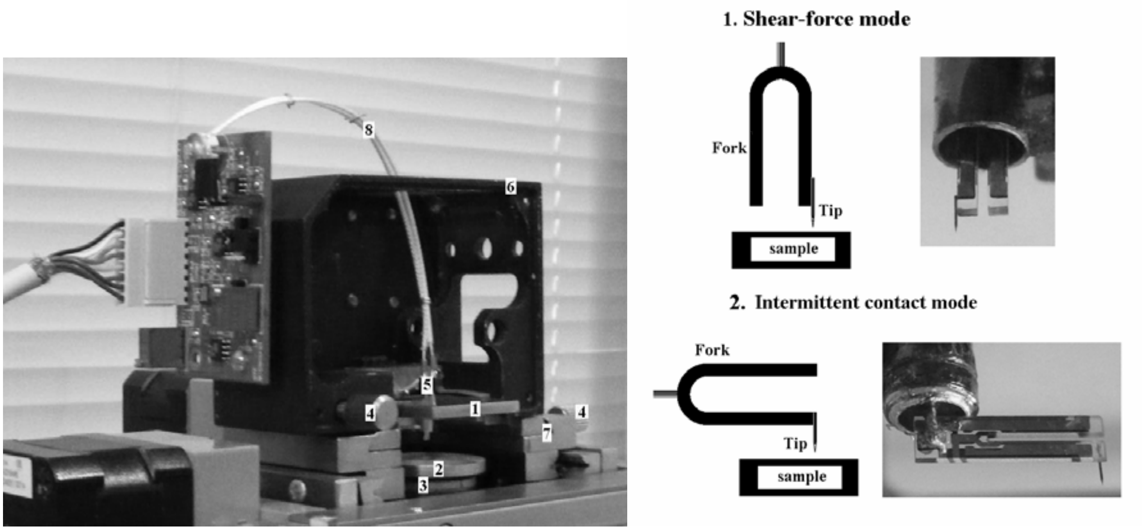
In our experiments, as usual, a tuning forks of a commercially available type fabricated for “quartz” clocks is used (type 74-530-04 of ELFA Company, standard resonance frequency 32757 Hz, and theory quality factor Q = 15000). The QTF was modeled in a standard way as a series *R*-*L*-*C* circuit. The *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 [8]. 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

$$k = \frac{E}{4} w \left(\frac{t}{l} \right)^3 , \qquad (1)$$

Where $E = 7{,}87 \cdot 10^{10}$ 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 $k \approx 7$ kN/m, which agrees reasonably well with our experimental result.

TUNING FORK COMBINED WITH AFM NT-206 (FORK-AFM)

The scanning probe microscope, based on the above described quartz tuning fork has been developed in our laboratory. The mechanical part of the tuning fork that connecting to ato-mic force microscopy NT-206 (Microtestma-chines Co., Belarus) [10] is shown in fig. 2a. This 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 (4) are fixed to the holder in the standard arrangement for probe-sample coarse and fine approaching. The tuning fork sensor (5) as the heart of the system is attached to the holder with cyanoacrylate glue, and connected to the AFM through the cable (8). The holder with tuning fork is placed on the base plate (2) and secured using the outside metal box (6). The metal box could be moved in the sample state (7) AFM NT-206.



a) b)

Fig. 2. a) – Photography of header of AFM NT-206 using a quartz tuning fork (Fork-AFM) (1 – holder, 2 – base plate, 3 – piezoscanner, 4 – screws, 5 – tuning fork sensor, 6 – metal box, 7 – sample state, 8 – cable); b) – Principle of two operation modes: shear-force and intermittent contact mode.

There are two basic methods of dynamic operation: intermittent contact mode and shear – force (or lateral mode) operation. In fig. 2b, we demonstrate basic principle of Fork-AFM in ambient conditions using a quartz tuning fork in these both modes. As shown in fig. 2b – lower part, the tip is mounted perpendicular to the tuning-fork prong so that the tip oscillates normally to the sample surface. This is the intermittent contact mode. In the lateral force sensor mode (fig. 2b – upper), the tip is mounted parallel to the tuning fork prong and oscillates nearly parallel to the surface of sample.

A constant sine wave voltage is applied to the one of the connectors of tuning fork to drive the fork sensor. The other connector of tuning fork is connected to a reference signal generator of the lock-in amplifier. When the probe is approaching nears the sample surface, the oscillation of the sensor is damped due to probe-sample force interactions, resulting in a decrease in the output signal of the lock-in amplifier. The decreased signal is compared with a set-point of the feedback circuit and the resulting difference is fed back to the scanner via the high voltage amplifier in order to control the probe-sample distance during scanning. A detailed analysis of the operation of the system was presented in [8], and so we will not repeat it here.

TUNING THE QUALITY FACTOR (*Q*-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 [5, 7, 8, 9, 20]. Furthermore, 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. 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.

Fig. 3 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). In our experiment, the resonance frequency shift is associated with a feedback loop phase that is not exactly equal to –90°.

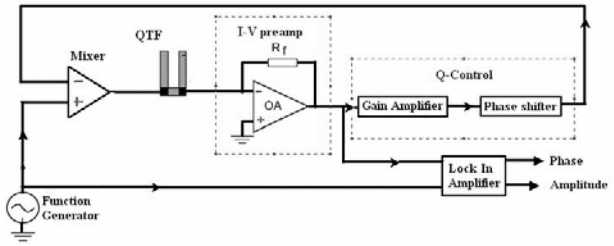


Fig. 3. Principle diagram of Q-control feedback circuit and the block diagram of I-V preamplifier and the *Q*-control system.

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. Fig. 4 displays a measurement of the decreasing of the quality factor based on a discrete component implementation of the circuit in fig. 3.

FORK-AFM IMAGING

To make sure that our system is able to obtain high resolution images of various kinds of sample in two operation modes, we have carried out the related experimental setup in two materials: fiber plastic and hologram. Here we only use the silicon cantilever tips with the radius about 10 nm. The process and the results of gluing tip are described in the [19]. Furthermore as the result in the [8, 9] the images with the high resolution are achieved when using the *Q*-control with the low pre-setting quality factor. There fore here we only present the results when using the low pre-setting quality factor for investigating the topography images of these materials.

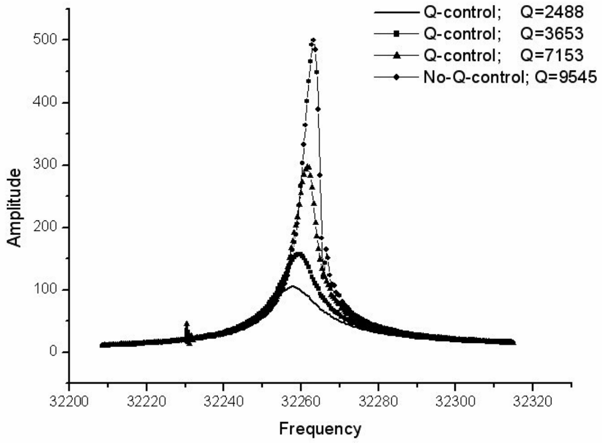


Fig. 4. Result of using *Q*-control, the amplitude response as a function of the driving frequency with different feedback g under condition of the phase shift – 90 degree.

Fig. 5a – e show topographical images obtained in the shear and tapping mode, respectively for fiber plastic and hologram. We have succeeded in getting images with discernible bit line in both cases. From this line profile (fig. 5c), the maximum height of the feature indicated by an arrow in the image is about 30 nm. Furthermore by comparing force images obtained in both shear mode (fig. 5a, d) and tapping mode (fig. 5b, e) with one type of tip, we found that a much better signal could be achieved in tapping mode operation. To explaining for this result, we bring out some assumption for explanation in the following way: in the shear-force mode, because the tip oscillates parallel to the surface of sample, the area contact between tip and sample is about 30 – 40 nm. Therefore, in this process, the instability such as signal drift or tip contamination maybe appear and influence the results scanning. And in the intermittent contact mode, the area contact between tip and sample is much smaller than shear force mode (about 10 – 15 nm). As a result, the region contact between tip and sample may achieve the atom interaction, thus it prevents sample damage, and we could obtain the images with high contrast resolution. These results show that this system Fork-AFM with good, sharp tip can receive high-resolution images of samples in ambient conditions.

DISCUSSIONS & CONCLUSIONS

We have described the method of using the quartz tuning fork as the sensitive sensor for AFM. Furthermore, we then discussed the use of the *Q*-control circuit as a tunable system, which could be increased or decreased the *Q*-factor 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 operation modes: shear force and intermittent contact modes. The scanning time could be reduced so that for the images of smaller size one could acquire the high resolution images almost in realtime, which may allow for recording high resolution video. Reproducible topographic images have been obtained on hard and soft samples. From the received results, one can also think of the combination of the tuning fork with the AFM that allows to inexpensively implementing a variety of scanning probe micros copies for investigation the properties of nano-materials.

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REFERENCES

1. Reference tuning fork 74-530-04 of ELFA Company, (C-MAC MicroTechnology, [http: // www.cmac.com]).
2. Karrai K., Grober R. D., Piezoelectric tip-sample distance control for near field optical microscopes//Appl. Phys. Lett. – 1995. – Vol. 66. – P. 1842-1844.
3. Giessibl F.J., High-speed force sensor for force microscopy and profilometry utilizing a quartz tuning fork//Appl. Phys. Lett.–1998. – Vol. 73. – P. 3956-3958.
4. Rychen J., Ihn T., Studerus P., Herrmann A., Ensslin K. A low-temperature dynamic mode scanning force microscope operating in high magnetic fields//Rev. Sci. Instrum. – 1999. – Vol. 70. – P. 2765-2768.
5. Lei F.H., Nicolas J.-L., Troyon M. Shear force detection by using bimorph cantilever with the enhanced Q-factor//J. Appl. Phys. – 2003. – Vol. 93. – P. 2236-2243.
6. Anczykowski B. In Handbook of Nanotechnology/Ed. Bharant Bhushan. (Springer-Verlag erlin Heidelberg). – 2004. – P. 449.
7. Lei F.H, Angiboust J.F., Qiao W. Shear force near-field optical microscope based on *Q*-controlled bimorph sensor for biological imaging in liquid//Journal of Microscopy. – 2004. – Vol. 216. – P. 229–233.
8. Vo Thanh Tung, Influence of *Q*-control on Shear-force Detection with Quartz Tuning Fork in Atomic Force Microscopy//Молодежь в науке. Прил. к журн. “Весці Нацыянальнай акадэміі навук Беларусі” – 2007. – Минск: Белорус. Наука. – 2008. – Vol. 3. – P. 85-89.
9. Vo Thanh Tung, Chizhik S.A, Quartz tuning fork atomic force microscopy using quality-factor control//Physics, Chemistry and Application of Nanostructures: Rev. and Short Notes to Nanomeeting-2007 Minsk, World Scientific Pub Co Inc. – 2007. – P. 535-538.
10. http://microtm.com/nt206/nt206r.htm
11. Forrer M.P. A flexure-mode quartz for an electronic wrist-watch//Proceedings 23rd ASFC. – 1969. – P. 157-162.
12. Yoda H., Ikeda H., Yamabe Y. Low power crystal oscillator for electronic wrist watch// Proceedings 26th ASFC. – 1972. – P. 140-147.
13. Ong P.P. Little known facts about the common tuning fork//Phys. Educ. – 2002. – Vol. 37. – P. 540-542.
14. Matsiev L.F., Application of flexural mechanical resonators to simultaneous measurements of liquid density and viscosity//Ultrasonic symposium. – 1999. – Vol. 1. – P. 457-460.
15. Chuang S.S. Quartz Tuning Fork Crystal Using Overtone Flexure Modes//35th Annual fre-

quency cont. symp. – 1981. – P. 130-143.

16. Itoh H., Aoshima Y., Egawa T. Model of a quartz crystal tuning fork using torsion spring at the joint of the arm and the base//Proceedings IEEE. – 2001. – P. 592-596.
17. Blaauwgeers, R., Blazkova, M., et al. Quartz tuning fork: thermometer, pressure – and viscometer for Helium liquids//Low temperature physics. 2007. – Vol. 146. – P.537-562.
18. Zhang J., O'Shea S. Tuning forks as micromechanical mass sensitive sensors for bio- or liquid detection//Sensor and Actuators B: Chemical. – 2003. – Vol. 94. – P. 65-72.
19. Vo Thanh Tung, Chizhik S.A, Chikunov V.V, Nguyen T.V., Tran X.H, Influence of additional mass on quartz tuning fork in dynamic operation mode//Proceeding of 7th Int. BelSPM-7, Belarus. 2006. – P. 236-240.
20. Friedt J.-M., Carry É. Introduction to the quartz tuning fork//Am. J. Phys. – 2007. – Vol. 75. – P. 415422.

АТОМНО-СИЛОВА МІКРОСКОПІЯ ПОВЕРХОНЬ ЗА ДОПОМОГОЮ КВАРЦОВОГО КАМЕРТОННОГО ДАТЧИКА В “ЛАТЕРАЛЬНО-СИЛОВОМУ” І “НАПІВКОНТАКТНОМУ” РЕЖИМАХ

Во Тхань Тунг, С.А. Чижик,
В.В. Чикунов, Чан Цуан Хоай

У роботі описується конструкція атомно-силового мікроскопа с датчиком у вигляді камертона на основі кварцового кристала. Кремнієве кантилеверне вістря було закріплено до зубця камертона таким чином, що дозволило реалізувати для камертонового АСМ латерально-силовой (shear-force) і напівконтактний (intermittent contact) режими. За допомогою системи електронного регулювання добротності зонда, так називаного *Q*-контролю, можливе настроювання параметра добротності *Q* і, відповідно, зміна повного часу сканування. Було помічено, що шляхом зменшення параметра добротності можна збільшити стабільність вимірюваного сигналу й у такий спосіб поліпшити розподіл для формованих зображень. Для демонстрації методики використовувалися зразки різних типів.

АТОМНО-СИЛОВАЯ МИКРОСКОПИЯ ПОВЕРХНОСТЕЙ С ПОМОЩЬЮ КВАРЦЕВОГО КАМЕРТОННОГО ДАТЧИКА В “ЛАТЕРАЛЬНО-СИЛОВОМ” И “ПОЛУКОНТАКТНОМ” РЕЖИМАХ

Во Тхань Тунг, С.А. Чижик,
В.В. Чикунов, Чан Цуан Хоай

В работе описывается конструкция атомно-силового микроскопа с датчиком в виде камертона на основе кварцевого кристалла. Кремниевое кантилеверное острие было закреплено к зубцу камертона таким образом, что позволило реализовать для камертонного АСМ латерально-силовой (shear-force) и полуконтактный (intermittent contact) режимы. С помощью системы электронного регулирования добротности зонда, так называемого *Q*-контроля, возможна настройка параметра добротности *Q* и соответственно изменение полного времени сканирования. Было замечено, что путем уменьшения параметра добротности можно увеличить стабильность измеряемого сигнала и таким образом улучшить разрешение для формируемых изображений. Для демонстрации методики использовались образцы различных типов.