

Resonance frequency as a function of the time needed by epoxic glue to fix a probe

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In Atomic Force Microscope based in Quartz Tuning Fork, it is usual to attach, by epoxic glue, a probe to one of the prongs of the Quartz Tuning Fork and employ it as force sensor. This paper shows that the resonance frequency changes as a function of the required time by the epoxic glue to dry. Results of the resonance frequency shift measured at different moments of the experiment are also reported. It has been found that it is necessary to wait at least 10 hours to obtain a stable mechanical system. The results conclude that the studied phenomenon becomes important in the frequency-modulation AFM, due to the evolution of the resonance frequency along the entire experiment.

Keywords: Resonance frequency; quartz tuning fork; atomic force microscope.

En el microscopio de fuerza atómica basado en osciladores de cuarzo, es usual adherir, por medio de pegamento, una sonda a uno de los brazos de dicho oscilador y utilizarlo como un sensor de fuerza. El presente trabajo muestra que la frecuencia de resonancia cambia como función del tiempo requerido por el pegamento para secar. Los resultados del cambio en la frecuencia de resonancia en distintos momentos del experimento también se reportan. Se ha encontrado que es necesario esperar al menos 10 horas para obtener un sistema mecánico estable. Los resultados concluyen que el fenómeno estudiado se vuelve importante en los microscopios de fuerza atómica basados en la modulación de la frecuencia de oscilación, debido a la evolución de la frecuencia de resonancia durante el experimento.

Descriptores: Frecuencia de resonancia; oscilador de cuarzo; microscopio de fuerza atómica.

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1. Introduction

Since Karrai [1] used the Quartz Tuning Fork (QTF) as a force sensor, several configurations of force microscopes using the piezoelectric detection have been reported [2-4], having as a common element, a probe attached to one of the prongs of such QTF. The attachment is usually obtained by epoxic glue [5] and the mechanical coupling becomes very effective, as demonstrated by the high resolutions that are obtained, in particular, on Atomic Force Microscope (AFM) [6-8]. Actually, it was demonstrated that the angle of interaction also affects the interaction between probe and sample [9]. On the other hand, it is well known that the QTF has a very narrow resonance frequency; it means that the quality factor is very high for low mass probes [10]. This feature is an advantage in frequency modulation mode AFM [11] and, consequently, it is very important to study the resonance frequency as a function of the determining parameters as quality factor, loaded mass and ambient conditions [12]; particularly, the time needed by the epoxic glue to dry, which is an important factor for the QTF sensor performance, has never been reported.

The present work explores the resonance frequency behavior in a QTF-probe system by using frequency modulation and determining the minimum time required to have a

mechanical system with enough stability to be used in frequency modulation mode AFM.

2. Experimental methods

The experiment has been performed with a standard Quartz Tuning Fork (QTF) with resonance frequency of 32,764 Hz, without probe, in self-excited mode [13]. The obtained current has been measured by a homemade lock-in [14]. In order to get a Lorentzian shape in the current, the excitation signal reported in [15] has been added a phase of 90°. The probe is a monomodal optical fiber with one end sharpened by chemical etching [16]. In order to allow the probe to have a resonance frequency higher than that of the QTF, 2 mm free at the end of the QTF prong are provided [17]. The used epoxic glue is a drop of cyanoacrylate adhesive [18] of approximately 5 L volume. Once the probe has been positioned on the prong of the QTF and the epoxic glue added, the experiment is begun immediately, even when the epoxic glue is still liquid. At that moment, the resonance frequency reaches a value of 32,095 Hz, due to the added load mass, *i.e.* the probe and epoxic glue. At the same time, the quality factor (Q) changes from 7814.2 to 2139.6 for the same reason. Figure 1 shows the QTF resonance frequency without probe and with probe at the beginning of the experiment (inset plot). In order to

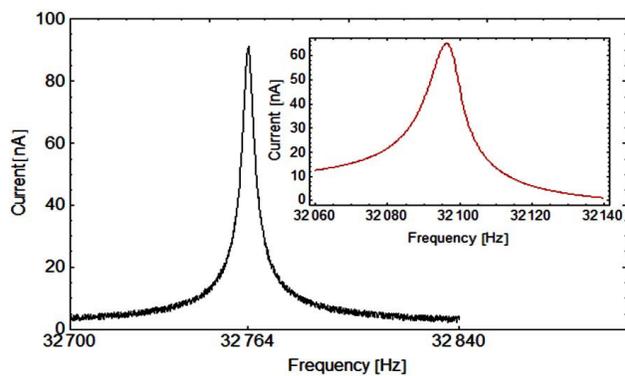


FIGURE 1. Resonance frequency of the QTF without probe. The inset shows the resonance frequency with probe at the beginning of the experiment.

check the resonance frequency shift, the excitation frequency has been swept from 32,060 to 32,140 Hz with steps of 0.05 Hz, with a sinusoidal function of 30 mV of amplitude.

3. Results

The results have been obtained continuously along ten hours. Figure 2 shows a collection of resonance frequencies during the entire experiment. It can be seen that the resonance frequency shift is very steep at the beginning of the experiment, during the first 15 minutes, due to the fast evaporation of the solvent of the epoxic glue. After this interval of time, the frequency shift increases monotonically for the next 8 hours of the experiment. In the last two hours, the resonance frequency shift is less than 0.2 Hz; it means that the glue is completely dry, so an efficient mechanical coupling has been obtained, see the inset of Fig. 2.

In order to check the mechanical coupling after long periods of time, frequency sweeps at 12, 24, 36 and 48 hrs, have been conducted; the results are shown in Fig. 3 with black, red, blue and green marks, respectively. It is clear that the change in the resonance frequency is around 0.1 Hz, which

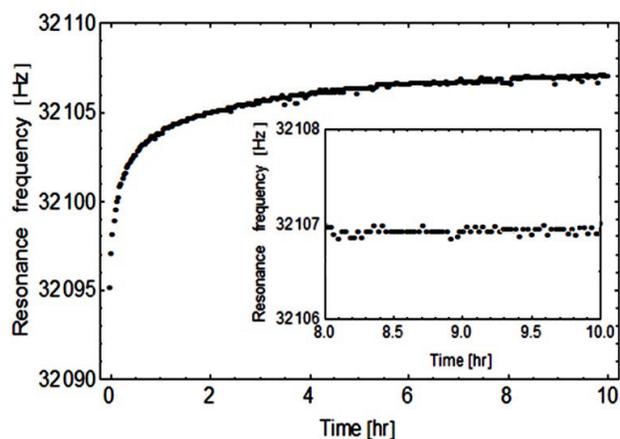


FIGURE 2. Resonance frequency shift in time. Inset: for the last two hours of the experiment the frequency shift is less than 0.2 Hz.

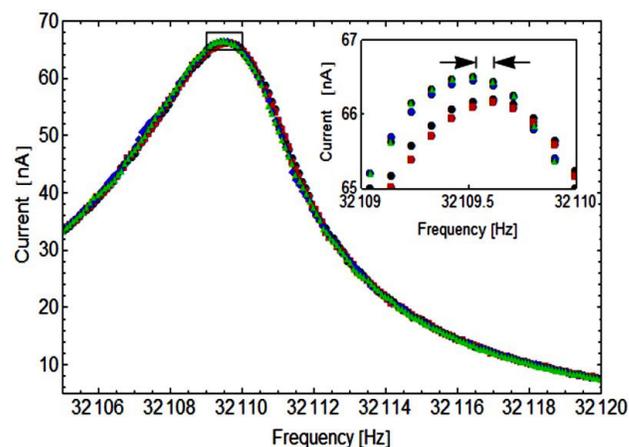


FIGURE 3. Resonance frequency shift after 12 hrs (black), 24 hrs (red), 36 hrs (blue) and 48 hrs (green). The inset is a zoom of the black rectangle. The distance between arrows indicates 0.1 Hz.

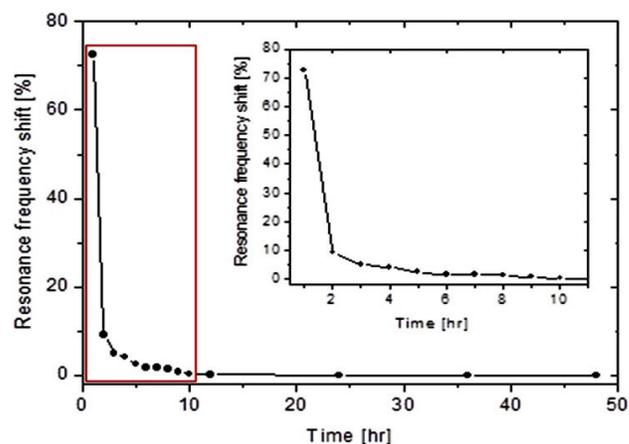


FIGURE 4. Resonance frequency shift in percentage of the total change in the experiment. The inset is a zoom of the red rectangle.

is enough to preserve the high resolution of a frequency and phase sensitive AFM.

Finally, the resonance frequency shift as a percentage of the total change in the experiment has been measured. It has been found that the total shift in the experiment is about 12 Hz respect with the value at the beginning. From Fig. 4, it can be seen that in the first hour the change is about 77% of the total, this is a consequence of the fast evaporation of the solvent in the used epoxic glue, as has been aforementioned. The rest 23% is reached along nine hours, it means, a very slow change but clearly evident.

4. Discussion and conclusions

It is well known that one of the main purposes in the Atomic Force Microscopy (AFM) is to obtain the highest lateral resolution as possible. For this reason, it is important to study the determinant parameters related to such resolution; particularly, it has been demonstrated that the drift on resonance frequency can affect the lateral resolution in an AFM based

on QTF [19]. Consequently, it is important to ensure that the resonance frequency will not change along an entire experiment, even in the case where such experiment has a duration of several hours. This way, it has been found that the resonance frequency changes about 77% of the total change, in the first hour after the probe has been glued, and 99% after 10 hrs. This phenomenon is due to several factors, but the most dominant of those is the evaporation of the solvent in the used epoxic. According to the results, it can be verified that after 12 hours the resonance frequency has changed about 0.1 Hz; despite it is only two orders of magnitude bigger than the drift of the bare QTF [20], it is still enough to obtain high resolution images.

The effect of the time needed by the epoxic glue to dry on the resonance frequency of a QTF-probe system has been studied. The results have shown that it is necessary to wait at least ten hours, under the described experimental conditions, in order to obtain the mechanical stability of the force sensor and ensure that the resonance frequency will not change beyond 0.1 Hz. Experiments realized at 12, 24, 36 and 48 hours confirm this fact. Finally, it is necessary to develop a mechanical model to predict the effect of the liquid layer of epoxic glue in the mechanical behavior and compare the theoretical results with the experimental ones presented in this work.

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