The issues of near field interaction detection in developed combined shear force/ emission microscope

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Abstract. The shear force microscopy is one of few AFM methods which allow to scan the surface in the non-contact regime. The advantage of this technique is the possibility of investigation soft and fragile samples. The detection of near field interactions is performed by measurement of the oscillation amplitude of the scanning tip, which oscillates laterally to the surface. Although the method is used effectively in various applications (i.e. SNOM), the origin of the phenomena is still discussed and investigated. In this article we describe some particular experiments performed in order to develop the home built shear force microscope which was combined with emission measurement module. We also present the solution, which allows to prevent unwanted behaviour of the PID regulator, when the electrical field is applied to the tip.

1. Introduction
The shear force microscopy (also the acronym SHFM is being used) is classified as a non-contact method, which means that the scanning tip moves over the surface in the attractive forces area. This AFM technique, due to very small forces acting between the tip and sample, can be used for measurements of very soft samples without a risk of damaging them. Due to still discussed matter of the tip-sample interaction mechanisms, specific experiments should be performed in order to apply the best possible detection method. Therefore recognizing of the role and influence of all factors is crucial. In this article principle experiments and technical solutions of specific measurement problems are described as an insight into the development process of home built shear force microscope which was also utilised to perform the measurement of local electrical properties of the surface as well as the LAO (Local Anodic Oxidation) process. The specific details of the construction of the setup as well as the experimental results were described in several papers [1,2] and its simplified diagram is shown in figure 1. The instrument was built using optical fibre Fabry-Perot interferometer for tip oscillation amplitude measurement. This feature allowed to obtain quantitative information about tip’s oscillation amplitude and to bias the tip. By using tungsten, electrochemically etched wire as the scanning tip, the measurement of the current emission from the surface was performed.
2. Principle of tip-sample interaction

In order to set the optimal parameters for tests, one should understand the tip-sample interaction principles. A number of papers has been devoted to the problem of understanding the phenomena of the shear force and both theoretical and experimental approaches were presented. As an origin of the tip-sample interaction, different physical mechanisms were proposed: Van der Vaals forces, capillary forces [3], viscosity and elastic forces [4,5], electrostatic forces [6], “knocking” [7,8], “kinking” [9], torsion [10] or friction [11]. Despite published theories and experiments, no final answer about the nature of those interactions was found yet and new ideas are presented, like surface-depend elastic-dissipative mechanism [12]. It must also be emphasized that in some papers one cannot find full information about the measurement conditions, thereby any referring to them can be difficult or impossible. One should be aware, that the environmental conditions should be controlled and described due to its impact on measurements [13,14]. Thereby we were encouraged to perform the experiments in order to check if we could obtain a similar result. The principle issue of configuration is the oscillation amplitude of the tip. It is commonly known, that the tip should oscillate with as small amplitude as possible. On the other hand, there is a limitation of a detection system. In our case we could obtain 0.01 nm RMS of resolution in a bandwidth of 100 Hz. This allowed us to apply typically 1 nm of free amplitude of oscillation of the tip. Thereby we did not observe the behaviour described by Lapshin et al. [10], who proposed a torsion phenomena which as a source of repulsive force could be the main mechanism of the tip-sample interaction. The non-contact interaction was also proposed by Bodo-Epoy et al. [15] as a conclusion of a similar experiment. When the oscillation amplitude is relatively large (comparable or larger than the attractive forces area), the situation can be observed as shown in figure 2.

![Figure 1. The simplified diagram of combined shear force-emission microscope.](image1)

When the surface of the sample is not perpendicular to the tip, similar behaviour like in intermittent contact measurements can appear, thereby the tip literally touches the surface. In the experiment described by Lapshin et al., the oscillation amplitude was between 40 nm and 120 nm therefore such even could be observed. It is also possible, that even when the tip is perpendicular to the surface, but the oscillation amplitude of the tip is about a 100 nm, the tip is “immersed” relatively shortly in the attractive forces area, thereby it approaches as long as the interactions are stronger, and then already some knocking can be observed. Thus the oscillation amplitude of the tip of the order of few nanometers can guarantee that the tip will always be in the attractive forces area (for set point values larger than 0.7).

3. Experiments

In order to recognize specific issues of shear force phenomena as well as to develop the setup, several experiments were performed.
3.1. The resonance curve vs. tip-sample distance and approach curve

One of the most important behaviours of the tip-sample interaction is a correlation between the tip-sample distance and resonance curve. This experiment was done by sweeping the excitation signal while the tip's oscillation amplitude was recorded (figure 3). As presented in many papers [14,16,17], the resonance peak shifts into the direction of higher frequencies. However, according to Vaccaro, the resonance frequency should decrease [18]. Also, the approach curve was measured as a correlation between both: tip oscillation amplitude and phase shift and tips sample distance (figure 4). The obtained curve agrees with typically presented results [19, 20], but it should be mentioned that the 0 value of distance is set arbitrarily.

![Figure 3](image1.png)

**Figure 3.** The resonant curves taken for different tip-sample distance.

![Figure 4](image2.png)

**Figure 4.** The approach curve showing tip oscillation amplitude and phase shift as a function of tip-sample distance.

3.2. Optimization of tip oscillation frequency and detection method.

One of the crucial issues in techniques where the oscillating tip is utilised is the choice of the excitation frequency in order to provide the best sensitivity of near field detection (low vertical noise) as well as a low response time (high scanning bandwidth). Due to different approaches presented in papers, the verification of the best solution of force detection was performed. In order to test the quality of system response, additional square Z signal was added in order to force the Z feedback to respond. The PID regulator setting was optimised in order to obtain the best possible result. It should be emphasized that the added signal was small enough (simulating 2 nm step height) to keep the tip-sample distance in the attractive force area in order to prevent the tip and sample damage in the case of insufficient quality of feedback response. The results of Z feedback reaction are presented in figure 5. The test was performed for certain tip's oscillation frequencies around the resonance. The detection of interferometric signal was made with analogue lock-in amplifier (Stanford 530) in two modes: R (amplitude only) and X (amplitude and phase: Rcosθ). The best detection condition can be found for the response signal most similar to square, with steep slopes, small overshoots and oscillations. The result of test is shown in figure 5. One can see that the best results were obtained when the X signal from the lock-in amplifier was used. It seems to be obvious, because the phase signal allows to obtain relatively large detection sensitivity (see approach curve). This conclusion was also described by Ruiter et al. [21], where the bandwidths of phase and amplitude were theoretically and experimentally compared and concluded that the bandwidth of amplitude signal is attenuated with 12 dB/oct, while of phase signal with 6 dB/oct. In the case of X detection, the excitation frequency does not affect significantly the response, so practically any frequency from the resonance range could be used. This method however would not be reliable when inhomogeneous samples were scanned, because the phase shift also varies with material properties of the surface. Thereby this phenomena would introduce the artefacts. Such solution was however suggested by Lei et al. [22] as a reliable method of signal detection for Z feedback.
The amplitude detection shows a significant difference of responses for certain frequencies. Relatively good results were obtained for frequencies located on slopes of the curve. However usage of the right slope seems to be unreliable if the frequency is too high and the behaviour of “tip to sample sticking” appears (the crossed mark). This behaviour is connected to the fact of moving the resonance peak into direction of higher frequencies and a risk of nonlinear correlation of detected signal – tip-sample distance. Basing on this experiment, the procedure of adjusting PID settings was developed and performed before the measurements.

3.3. The electrical field influence on tip-sample interaction

Due to development of the instrument as a device for investigation of electrical properties of the surface, it was crucial to recognize the influence of the electrical field on tip-sample interaction. In order to do that, the square voltage signal was applied to the tip-sample biasing circuit while the tip was in the typical scanning position, but no scanning process was running (figure 6). The relation between the applied voltage and Z feedback response is presented on graph (figure 7).

As predicted, the response of Z feedback proved that the electrical field changes the forces acting between tip and sample and makes the feedback retract the tip from the surface. In the described setup, the measurement of the emission current is performed after the tip-sample distance is settled in certain position. Then the tip moves to another location, settles the distance and another measurement of emission current is taken. In order to prevent the response of Z feedback on applied electrical field, the tip-sample distance is “frozen” with a developed extension module for PID regulator. The simplest way to solve such a problem is an application of a sample-hold buffer on the output of the PID module (figure 4). Than the switching signal can “freeze” tip’s position over the surface and applied tip-sample bias voltage does not affect the emission conditions. However, when the bias is applied, the
measured signal changes and makes the PID output signal change. But as long as the feedback loop is open, the PID output signal changes without any control. This fact introduces a risk, that after releasing the “hold” signal, Z actuator will receive a signal irrelevant to the tip-sample distance and it can be very rapidly retracted or crashed into surface. This behaviour should be eliminated, thereby a new solution was developed and another sample-hold module was connected to the input of PID module. This allowed to make the input signal on PID unchanged during the bias applying. One must note, that even the smallest offset between measured signal and the set point can still make the integrating channel of the PID change the output signal, and cause unwanted changes of the tip-sample distance after disabling “freeze” signal. This issue can be solved by shortening the capacitance in feedback circuit of operation amplifier which is responsible for integration process. Such a solution allows to restore a tip’s position and appropriate conditions of signals very quickly. Moreover the risk of tip damage is minimized. In figure 8 the signal traces for different solutions of PID development are shown. It should be emphasized that utilisation of digital PID allows to solve those issues much easier if the “freeze” option is available or one has a full access to the code and can stop the calculations at any moment if necessary.

![Figure 8](image.png)

Figure 8. Theoretically predicted behaviour of the Z feedback for different solutions of the signal “freezing”. The analogue PID signals traces for different solutions of “freezing” the feedback loop: no sample-hold devices applied (black, dotted line), sample-hold device placed on the output of the PID (black, dashed line), sample-hold device placed on input and output of the PID (grey, dashed line), all block of the significant signal processing channels in PID are frozen (black, solid line). Please note, that some real features (overshoots, oscillations) are not shown in order to make the graph more clear.

3.4. The result of measurements of real object

One of the most popular sample in AFM measurements is HOPG (Highly Oriented Pyrolite Graphite). In presented result (figure 9) a relatively large scan field was measured with specific features which can be resolved on both: topography and emission map pictures.

![Figure 9](image.png)

Figure 9. Topography (on left) and current emission map (on right) measured with developed shear force/ emission microscope on the surface of the HOPG sample.
The measurement was taken in ambient (relative humidity 35 %, temperature 23 °C), with typical scan settings: free tip oscillation amplitude: 1 nm, set point 0.9, scan rate 0.1 Hz. One can see clearly areas of increased emission from the surface, that correlates with the surface. However it is crucial to be aware of possible artefacts appearance due to i.e. varying of an active field of emission from the tip or disturbed tip-sample distance on very steep structures. Such interpretation issues are commonly known in AFM measurement techniques.

4. Conclusions
In the article we have presented some of experiments performed in order to develop the home-built construction of a combined shear force/emission microscope. By recognizing several issues, we could improve significantly the quality of work of the instrument as well as compare our results of experiments to the published ones by other groups. The results we obtained mostly lead to the same conclusion, however we disagreed with some theories concerning the origin of the shear force phenomena. According to our experiments, the electrical field is one of the several mechanisms observed as tip-sample interaction in this near field detection technique. By applying specific solution of hold-sample module, we could prevent the unwanted behaviour of the Z feedback after disabling the “freeze” mode, which was used, when the emission current from the surface was measured.

The experience we could gather is also used in a development of other systems and solving problems connected to other methods of near field measurements. Further experiments are required in order to recognize specific phenomena’s influence on the tip-sample distance at certain measurement conditions.

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