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SURFACE IMAGE ARTIFACTS CONNECTED WITH THE GEOMETRY AN ATOMIC FORCE MICROSCOPE TIP

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In paper the effect convolution arising at surface scanning by method of atomic force microscopy is considered. The processes occurring in system "tip-surface" are analyzed. The estimation of critical parameters of a tip and of a tip point, also a degree of influence of these parameters on a surface is made at scanning. It is shown, that of the step form tip allows to minimize many artifacts and to receive correct topography of a surface with complex morphology.

1. Introduction

Scanning probe microscopy (SPM) is subdivided into scanning tunnel microscopy (STM) and atomic force microscopy (AFM). Both of a method mutually supplement each other and allow receiving rather extensive information on various characteristics of a surface. However there is an essential difference between them from the point of view of the spatial resolution. If STM really shows the resolution down to atomic, for AFM the level of the molecular-atomic resolution (especially in lateral planes) is while an unattainable level. A principal cause here is that resolution AFM very strongly depends from geometrical sizes of the microscope tip point.

Even though atomic force microscopy [1,2] has been successful in imaging surfaces with atomic resolution, it is still doubtful whether true atomic resolution is really obtained. Most images reported show perfect crystal lattices or defects much larger than atomic scale defects. On the other hand, the situation in scanning tunneling microscopy is quite different and images with point defects are routinely obtained [3]. This is usually attributed to the fact that the tunneling current is laterally localized in an area of few angstroms in diameter, while in atomic force microscopy the effective part of the probing tip is laterally much larger. So the atomic resolution is not obtained by a point interaction but by a superposition (convolution) of several interactions between the atoms in the tip and the sample. This assumption is justifiable if one considers that even in the case of a diamond tip and a diamond sample, using typical loads, the tip-sample contact area is larger than a single-atom one [4,5]. The two surfaces (tip and sample) are generally deformed when they are in contact [6]. For softer materials this tendency for larger contact areas under load is even more prevalent [4]. For materials with layered structures (e.g. pyrolytic graphite) the assumption that the tip drags a flake of the material as it scans the surface has proved to be very fruitful [7] and provides results in agreement with the experiments. Especially for the layered materials the same considerations of flake-like tips (or multiple-atom tips) can be also applied to the STM imaging mechanism. However, the usual case in STM pictures is the imaging of single-point defects in a variety of materials. This excludes the possibility of laterally large effective tips as has been shown [8].

In a word, there are two physical mechanisms that make the tip-sample contact area become of some considerable size: a) loads (even the lowest ones) result in a flat contact area of considerable size; b) especially for layered materials the tip drags a flake of the sample probed and this flake is the effective tip. The main difference between the two cases is that while in the first case the material of the tip is in general different from the material of the sample, in the second case the effective flake like tip is of the same material. At last, tip with big radius is not able to track precisely sharp relief with big height drops on the surface [9-11], this is third physical factor that makes the tip-sample contact area becomes of considerable size.

Thus, for topographic AFM studies of larger scale structures the macroscopic shape of the tip becomes very important. So, it is necessary to have a limiting small radius of the microscope's tip. However, as for the further analysis tip parameters are the most important ones, so we can rule out the possibility to study the second factor in the system of "tip-surface".

2. The processes being in the system "tip-surface"

Here several various cases can be arisen. The first case is recesses or rises on the surface of half-sphere-typed with the radius much bigger than the tip radius. Here surface topography is tracked with maximum accuracy. Therefore there are no problems in data interpretation for these two cases. But such "ideal" surfaces which can be investigated a tip of the big radius, in atomic force microscopy meet seldom.

The second case: there has been a recess with sharp edges on the surface; in this case its lateral sizes are bigger than tip ones. The tip falls until back control system touches the recess bottom. Here at the expense of interaction of recess front edge with lateral side of tip we obtain not the image of recess lateral edges but the image of tip lateral surface. There have been taken place size distortion in the direction of scanning surface and data loss as a result. There has been a step with sharp edges up on the surface. The tip is moving over the surface measuring surface relief till the beginning of the interaction of its lateral surface with the step edge. The relief of the tip lateral surface starts to be represented and the value of distortion and data loss depends on the distance between the tip and surface: the less is the distance, the less is distortion**.** In this case to minimize given distortion it is possible only by means of a tip with small radius of a point.

The third case: the apparent sizes of features can be also affected by the topography around them. For example, if one investigates spherical particles adsorbed on a rough substrate their shape will be partially determined by the topography of the substrate around them (we assume that spherical particles dimensions are in the order of the tip's radius of curvature). fig.1 shows schematically this distortion: even if two particles are exactly the same, they will be imaged differently if one is on a peak and the other in a valley, due to convolution of the tip and the sample. The thick line shows the apparent height corrugation. The particle in the valley seems lower ($h_1<h_2$) and more flattened ($w_1>w_2$) than the particle on the peak.

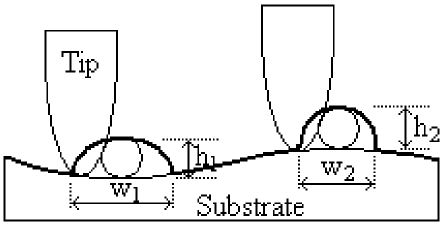

Fig.1. The effect of particle shape at surface local topography.

The fourth case: a tip with a relatively large cone angle fails to penetrate into deep and narrow grooves on the sample surface. This leads to underestimation of their depth and smoothing of their edges (fig. 2(a)). One point of recess edge touches lateral surface of the tip. Tip displacement trajectory do not describes recess surface, but tip surface. Although the height of protrusions can be recorded quite accurately their edges are being smoothed and their lateral size is overestimated (fig. 2(b)).

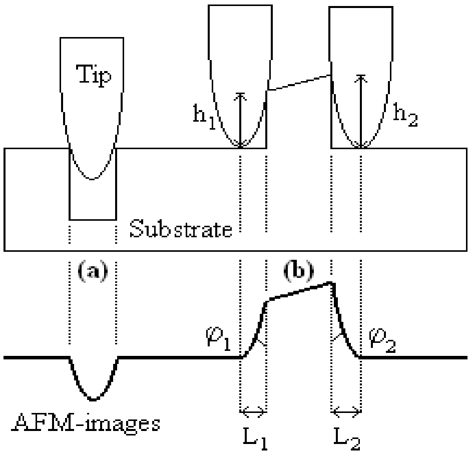

Fig. 2. The effect of tip geometry at AFM imaging (thick line): a) a groove and b) a protrusion.

Due to finite size of the tip artifacts are produced and the resulting image is a convolution between the shape of the tip and the topography of the sample. Therefore is this case interaction between the tip and surface changes from the system “tip-surface” to the system “tip-cluster surface”, tip displacement trajectory describes the image of the tip itself. In this case there is a restriction connected with geometry of a tip. There can be areas in which the image basically is defined by the form of a tip and geometry of its fastening. As a first approximation these so-called “dead zones” can be defined as follows: $L_1 \approx h_1 \times tg\left(\varphi_1 - \alpha\right)$ and $L_2 \approx h_2 \times tg\left(\alpha + \varphi_2\right)$, where α – a corner of fastening of a tip with cantilever. At $\varphi_1 \geq \alpha$, that is typical for the majority industrial cantilevers, having a corner of fastening $\alpha \geq 15°$, the area of “dead zone” L_1 for "step" is defined only by radius of curvature of a tip. The size of area of “dead zone” L_2 for conic tips with a corner of convergence 22° (φ_2 = 11°) and α = 20° turns out significant and is $L_2 \approx 0,6 \times h_2$.

Thus, the brief analysis of the artifacts arising in system "tip-surface" has shown, that accuracy of convolution task decision in AFM to those above, than it is less radius of a tip curvature and than less corner of convergence of a tip cone.

3. The critical physic mechanical parameters of the tip

So to obtain a clear AFM-topography of the surface with complex relief it is necessary to have the tip with low radius and high mechanical stiffness. But tips from Si_3N_4 which are widely applied in atomic force microscopy though have high mechanical stiffness have such lacks, as the big radius of a tip curvature and the big corner of a tip point. In the first case the possibility of achievement of the high AFM resolution, and the second lack not is excluded is correct to measure complex surfaces. All this does not provide an opportunity of precision measurements of a surface topography by means of AFM. From this point of view more perspective are tungsten tips. However all technologies of tungsten tips making until recently had the certain restrictions.

How can we reduce the radius of the tip? If we use most widespread method of electrochemical etching the magnitude of the obtained tip is defined by the relationship:

$$R = D\sqrt{\frac{\gamma \Delta L}{4\sigma_{ur}}} \quad , \tag{1}$$

where D is diameter of tip wire; ΔL is length of separating part; γ is specific density of tip agent; σ_{ur} is ultimate resistance to tip agent breakage. For tungsten being one the conventional materials at producing the tip specific weight γ=1, 89×10^5 N/m^3; σ_{ur} = 7, 11×10^8 N/m^2. It follows that it is possible to obtain the tip with reasonable values of tip radius ≤10 nm from wire of low diameter (≤10^{-4}m). However the tip with such diameter has low mechanic stiffness in longitudinal and lateral direction and mechanical, thermal and quantum fluctuations can worsen stability of atom force microscope operation. Let us evaluate amplitude of longitudinal and lateral oscillations of tip of two tips in diameter 10^{-3}m (Tip1) and 10^{-4}m (Tip2) at their similar length 10^{-2}m. It is known that mean square of amplitude of quantum $\langle X_q^2 \rangle$ and thermal $\langle X_T^2 \rangle$ fluctuations of oscillating system on resonance frequency is defined as

$$\left\langle X_q^2 \right\rangle = \frac{\hbar\omega}{K_M} \tag{2}$$

$$\left\langle X_T^2 \right\rangle = \frac{\kappa T}{K_M} \quad , \tag{3}$$

where k is Boltzmann’s constant; T is temperature; K_M is effective stiffness of oscillating system; ω_0 is its resonance frequency equal to $\sqrt{K_M /m}$; m is equivalent mass of oscillating system. Lateral K_{ML} and longitudinal K_{MV} stiffness of tip can be determined with sufficient accuracy using the following expressions:

$$K_{ML} = \frac{3\pi E D^4}{64\, L^3} \tag{4}$$

$$K_{MV} = \frac{\pi E D^2}{4L} \tag{5}$$

where E is Young's modulus, L is tip length. If we substitute in formulae (2–5) typical parameter magnitudes we get following values for thermal and quantum fluctuations (table 1 and table 2).

Table 1

	Tip 1	
	Longitudinal oscillations	Lateral oscillations
L, (m)	10^{-2}	10^{-2}
D, (m)	10^{-3}	10^{-3}
K_M, (N/m)	$1,5\times10^{7}$	$3,1\times10^{4}$
ω_0, (Hz)	$3,2\times10^{5}$	$1,2\times10^{4}$
$[<X_q^2>]^{½}$, (m)	$1,8\times10^{-18}$	$3,5\times10^{-17}$
$[<X_T^2>]^{½}$, (m)	$3,3\times10^{-14}$	$6,7\times10^{-13}$

As it follows from table 1 and table 2, tip 2 has deficient mechanical stiffness. But if the tip diameter is 10^{-3}m tip radius is prohibitively big (see formula (1)).

Table 2

	Tip 2	
	Longitudinal oscillations	Lateral oscillations
L, (m)	10^{-2}	10^{-2}
D, (m)	10^{-4}	10^{-4}
K_M, (N/m)	$1,6\times10^{5}$	$2,9\times10^{1}$
ω_0, (Hz)	$2,1\times10^{5}$	$2,8\times10^{3}$
$[<X_q^2>]^{½}$, (m)	$1,7\times10^{-17}$	$1,1\times10^{-16}$
$[<X_T^2>]^{½}$, (m)	$3,1\times10^{-13}$	$2,1\times10^{-11}$

In addition, it is necessary to estimate of mechanical stability of a tip point. Let us evaluate amplitudes of quantum and thermal fluctuations of a tip point in the form of the cone in length 1nm and diameter at the base 0, 5 nm according to formulae (2 – 5) (Table 3).

Table 3

	Point of a tip	
	Longitudinal oscillations	Lateral oscillations
L, (m)	10^{-9}	10^{-9}
D, (m)	5×10^{-10}	5×10^{-10}
K_M, (N/m)	$7,9\times10^{1}$	$0,2\times10^{1}$
ω_0, (Hz)	$1,1\times10^{12}$	$6,1\times10^{11}$
$[<X_q^2>]^{½}$, (m)	$1,1\times10^{-12}$	$6,3\times10^{-12}$
$[<X_T^2>]^{½}$, (m)	$1,2\times10^{-11}$	$1,3\times10^{-10}$

As it follows from table 3 the lateral thermal fluctuations of a tip point has large amplitude ~10^{-10}m. In accordance to the calculations the amplitude of tip point oscillations can reach one angstrom. But is must be confirmed by the presence of "blurred" sections on SPM-images of the surface at the atomic resolution. However, virtually experimental data do not confirm this fact. These oscillations occur on the frequency of the main lateral mechanical resonance of a tip point equal to ω_0 = 6, 1×10^{11} Hz. As frequency bands of electron system stability of SPM control do not exceed ω_{max}= 10^3 Hz, minimum time of measurement is $\tau_1 = 10^{-3}$ s. It excels the time when averaging of a tip point oscillations is taken place with frequency ~100 kHz equal to $\tau_2 = 2Q/\omega_0$ where Q is quality factor of oscillating system. As a result of averaging the accuracy of mean position determination of tip point increases by $n = \sqrt{\tau_1 / \tau_2}$. If we assume Q =100 we get $n \approx 10^3$. In this case amplitude of a tip point thermal fluctuations is $\Delta X_{Tmin} = \{[<X_T^2>]^{½}\}/n = 10^{-13}$m, i.e. the less of 1/100 of angstrom that is quite enough for practical purposes. Thus a tip's point mechanical stability of typical geometry and sizes do not have a significant influence on reading surface image on SPM.

4. The tungsten tips with a step form

As it was already marked, the tip should have extremely small radius of a tip point and simultaneously possess sufficient mechanical stiffness. At manufacturing a tip by a method of electrochemical etching it is not possible to provide performance of both these conditions, since there is a connection between initial diameter of a wire and the received radius of a tip point (fig. 3(a)). Therefore, with the purpose of reception of a tip with small radius of a tip point and high mechanical stiffness the technique in which basis the method of reception of a tip of the step form lays at electrochemical etching (fig. 3(b)) has been developed. Thus at the initial stage etching goes in regular intervals on all length of the part of a tungsten wire shipped in a solution. After some time the wire is a little extended from a solution and process proceeds, and etching already goes on smaller, in comparison with initial, to diameter. It allows ~8×10^{-4} m to spend at initial diameter of a wire a final stage of etching at working diameter ~4×10^{-5} m, that as a result gives a tip with small enough radius of a point (≤20 nm) and, owing to the geometrical form, high mechanical stiffness. Such step form tip has optimum parameters (fig. 4). Those tips allow of artifacts being at the investigation of surfaces with complex relief to be minimized.

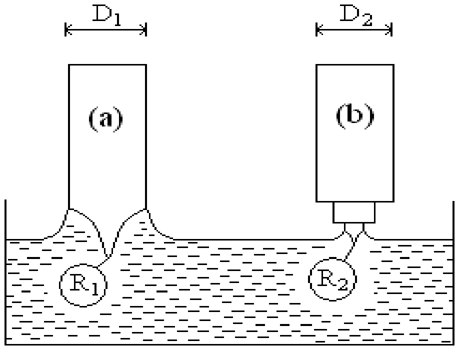

Fig. 3. Classical (a) and step (b) methods of tungsten tips electrochemical etching.

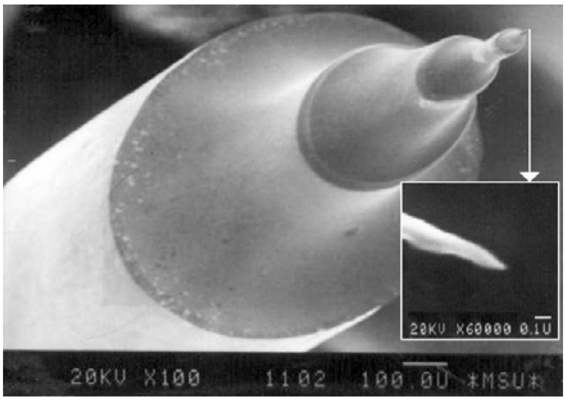

Fig. 4. SEM-images of a step tungsten tip and a tip point.

Thus, the decision of a task on reduction of a “dead zone” at AFM measurements of deep recesses and protrusion surfaces has demanded development of special manufacturing techniques an atomic force microscope tips – technology of step form tips electrochemical etching. In this case the form of a tip is in advance known and can be correctly considered at interpretation of results of AFM measurements. All this makes this type tips extremely perspective for application in analytical atomic force microscopy.

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S.C. Ələkbərov

ATOM-QÜVVƏ MİKROSKOPUN İYNƏSİNİN HƏNDƏSİ FORMASI İLƏ BAĞLI OLAN SƏTHİN GÖSTƏRMƏSİNİN ARTEFAKTLARI

Məqalədə atom-qüvvə mikroskopiya metodu ilə səthin tədqiqi zamanı təzahür edən konvoliyusiya effektinə baxılmışdır. “İynə-səth” sistemində olan proseslər araşdırılmışdır. İynənin və iynə ucu itiliyinin kritik parametrləri, eyni zamanda bu parametrlərin skan zamanı səthə təsir dərəcəsi qiymətləndirilmişdir. Göstərilmişdir ki, pillə formasında olan iynə artefaktların əksəriyyətinin minimallaşdırmasına və mürəkkəb morfoloqiyaya malik olan səthin korrekt topoqrafiyasının alınmasına imkan verir.

С.Д. Алекперов

АРТЕФАКТЫ ИЗОБРАЖЕНИЯ ПОВЕРХНОСТИ, СВЯЗАННЫЕ С ГЕОМЕТРИЕЙ ИГЛЫ АТОМНО-СИЛОВОГО МИКРОСКОПА

В работе рассмотрен эффект конволюции, возникающий при исследовании поверхности методом атомно-силовой микроскопии. Проанализированы процессы, происходящие в системе «игла-поверхность». Произведена оценка критических параметров иглы и острия иглы, а также степени влияния этих параметров на поверхность при сканировании. Показано, что игла ступенчатой формы позволяет минимизировать многие артефакты и получить корректную топографию поверхности со сложной морфологией.

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