AN ALTERNATIVE APPROACH TO USING ATOMIC-FORCE MICROSCOPY IN THE SURFACE STUDIES

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A new research "MD mode" regime based on the atom force microscopy was developed. "MD mode" regime allows exploring any nonconductive surfaces with complex topography and large differences of heights. Moreover, it allows excluding uncontrollable interaction of probe with sample, and artifacts caused by scanning process. The experimental results demonstrating the possibility to use atomic force microscopy with "MD mode" for the measurement of number properties of the surface are given.

1. Introduction

Atomic-force microscopy (AFM) is the powerful method for studying the nanocrystalline material surfaces [1, 2]. Continuous improvement of methods and instruments for AFM has led to the development of number of new procedures and techniques for the investigation of surfaces. However, some AFM operators have serious problems in experiment result interpretation. It is connected with artifacts arising during the surface investigation with the complex relief in atmospheric ambience, because materials with complex surfaces are difficult to investigate using conventional AFM modes [3-5].

"Contact mode" techniques have a high resolution, but can be applied only to very limited group of surfaces which must be rigid and smooth. One more weak point of "contact mode" in application to complex surfaces is that they roughness of the latter commonly leads to the catching of the probe during scanning so that the probe quits its moving operation over the surface.

"Tapping mode" is a rather good mode for scanning of the complex surfaces without abrasion and catching of the probe, but the resolution of "tapping mode" is commonly worse than that of "contact mode".

"Non-contact mode" provides conditions for avoiding probe's pollution, destruction, abrasion and catching. However, a probability of probe catching to the surface is rather high in studying of the complex surfaces by "non-contact mode".

All above problems are solved by using so-called "MD mode" regime [6-8]. In this paper we demonstrate the possibilities of the "MD mode" regime and report the results of scanning various surfaces.

2. "MD mode" regime and experimental details

"MD mode" regime operates in the following manner. As usual, a chosen section of the surface is scanned by probe with some step. But the path the probe is going along in the vertical plane is very different from the probe trajectory in conventional AFM modes [9]. "MD mode" probe operation is shown in Fig.1. In the beginning the tip of the probe is high enough over the surface (Fig.1, (1)) so as to avoid any probe-surface interaction. Then, the tip starts moving from this position down to the surface and stops after the contact with the surface is reached (Fig.1, (2)). At this stage topographic measurements of the surface coordinate are carried out. As the measurements are performed in full contact with the surface, their resolution is comparable with "contact mode" resolution. After the measurements are completed the tip is removed from the surface back to the starting position (Fig.1, (3)). At the end of a scanning step the tip is shifted in lateral direction to the next point, as shown in Fig.1, (4, 5). Note that no friction occurs during the lateral moving since no contact between the probe and the surface is taking place. Data is recorded only when the probe is moving in the normal-to-surface direction. As a whole, "MD mode" operation is free from the abrasion and catching of the probe by complex surfaces and is very similar to the operation of a sewing machine.

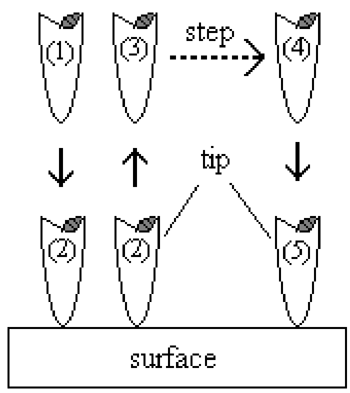

Fig. 1. Path of probe movement at surface scanning by "MD mode" regime.

In fact, the probe trajectory in moving to and back from the surface is more complicated then its schematic presentation in Fig.1 because this trajectory is described by force curve (Fig.2). In "MD mode" the basic information comes from the signal of the cantilever deviation along the normal to the scanning plane [10]. Recording the signal in the delivering of the probe to the surface and back to "no interaction" position gives a force curve – *S(Z)*, i.e. the magnitude of cantilever's bending, *S*, as a function of the coordinate of the surface position, *Z*, that is always treated as known (Fig.2, (b)). Along with physical and chemical properties of the surface, the shape and parameters of the measured force curve are largely dependent of elastic properties of the cantilever (Fig.2, (a)), and geometric properties (size and shape) of the tip of the probe. Therefore, knowing the parameters of the last two, one can obtain not only complex topography but also adhesion force, thickness of adsorbed layer, and other important parameters of the surface [11, 12].

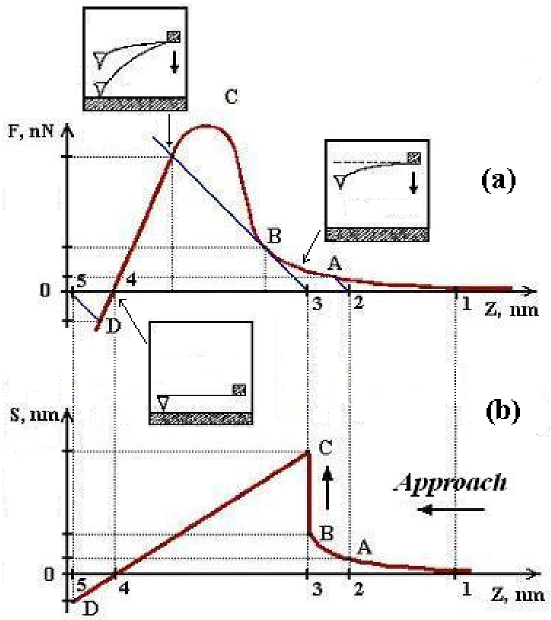

Fig. 2. The process of probe approach to the surface: dependence of Van-der-Waals interactions on the distance up $F_s(Z)$ surface (a); force curve *S(Z)*, there has been shown cantilever positions with the probe on the coordinate of surface position (b).

By measuring the parameters of the *S(Z)* function, say, in an $N\times N$ square of point on the surface under study, we can reconstruct the relief S_{tph}, a map of adsorbed layer distribution over the surface S_{adl}, a map of adhesion forces distribution over the surface S_{adf}, a map of the surface with the constant repulsion force F_{rpf} and a map of the surface with the constant force gradient F_{cfg}. If any other measurement "X" is made on the $N\times N$ points, this would give an additional map of the surface S_x by the parameter "X". The property measured can be either the conductivity in the point "C" or the surface hardness as the slope of the function *S(Z)* in the region of repulsion ($Z<0$). The advantage of this regime is that it does not involve "dragging" of the tip along the surface. This "MD mode" regime also helps overcome the problem of the optical scheme drift because the "zero" of the optical system can be determined each time the tip is beyond the region of the surface force action. A disadvantage of this regime is a relatively long time of information acquisition which varies from 10 to 30 min for a 128×128 frame. In addition, the regime of "MD mode" does not allows working with blunt (R>20 nm) tips.

3. Results and discussion

Below we present experimental results demonstrating the potentialities of the "MD mode" regime and the possibility to use AFM with "MD mode" for the measurement of number properties of the surface.

Fig. 3(a) shows the surface of an aluminium-oxide-based catalyst. The surface is characterized by a strongly developed relief, the difference in *Z* between the lowest and highest point positions corresponds to 30,5 nm. Fig. 3(b) displays the map of adhesion forces taken simultaneously with the relief. It is that the adhesion force radically varies within the frame area. Apparently, the last fact is most likely to be a result of the influence of the capillary forces which are caused by the presence of the adsorbed layer on a porous surface of ceramic catalyst to give, in addition to adhesion forces, above strong variation (ΔZ=1,9 nN) in adhesion force map.

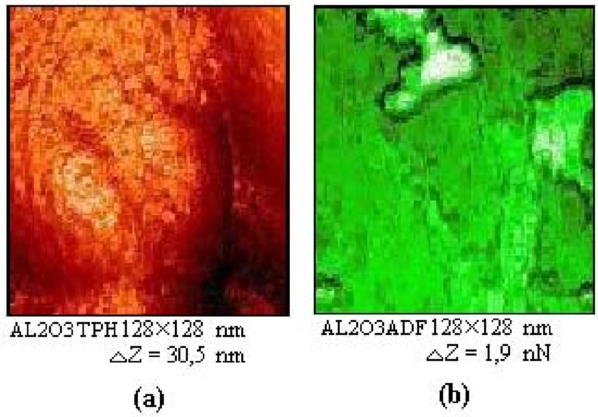

Fig. 3. Topographic image of a surface fragment of AL oxide-base ceramic catalyst S_{tph} (a); map of adhesion forces distribution over the AL_2O_3 surface S_{adf} (b).

The potentialities of the "MD mode" regime in working with soft samples were tested on the surface of a 2×10^6 nm mercury ball. Fig. 4(a) displays a picture of the mercury surface 1000×1000 nm in size taken when of the tip was not in contact with the surface, that is, it was removed immediately after its jump towards the surface. Based on these results, we reconstructed the surface with the constant force gradient F_{cfg} (*dF/dz=4 N/m*). The data in Fig. 4(b) and Fig. 4(c) were obtained upon the repeated scanning of the lower left quadrant of the above surface. However, in this

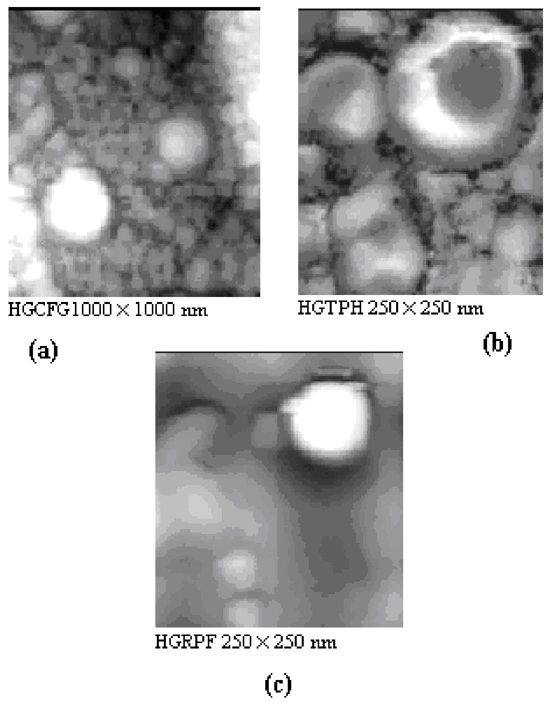

Fig. 4. Map of the surface with the constant force gradient F_{cfg} (a); topographic image of a surface of mercury droplet S_{tph} (b); map of the surface with the constant repulsion force F_{rpf} (c).

case the tip was brought into contact with the surface with the repulsion force $F_{rpf} \approx 10^{-9}$ N. The relief of the surface S_{tph} is displayed in Fig. 4(b) and the map of the constant repulsion force F_{rpf} is displayed in Fig. 4(c). If these two pictures are superimposed, we obtain an exact copy of the lower left quadrant of the Fig. 4(a). This example demonstrates how radically the surface image obtained by the steady level of repulsion forces can differ from that obtained by the constant gradient.

Thus, "MD mode" regime of AFM is integrating in itself both the non-destructiveness of "tapping mode" and "non-contact mode" and high resolution of "contact mode". As shown in this work, "MD mode" regime, which allows simultaneous registration of a number of surface parameters, can be reliable applied to study surfaces with various physical-chemical characteristics.

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ATOM-QÜVVƏ MİKROSKOPİYASI ƏSASINDA SƏTHİ TƏDQİQ ETMƏK ÜÇÜN ALTERNATIV ÜSUL

Atom-qüvvə mikroskopiyası üsuluna əsaslanan yeni "MD mode" tədqiqat rejimi işlənilmişdir. "MD mode" rejimi böyük dəyişkən yüksəkliyi və mürəkkəb relyefi olan qeyri-keçirici səthləri tədqiq etməyə imkan verir. Bu rejim, skaner prosesi əsnasında, zond ilə nümunə arasında yarana bilən idarəolunmaz qarşılıqlı təsiri və artefaktları istisna edir. Atom-qüvvə mikroskopu vasitəsilə səthin bir sıra xüsusiyyətlərinin ölçülməsində "MD mode" rejimindən istifadənin imkanları experimental nəticələrdə nümayiş etdirilmişdir.

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АЛЬТЕРНАТИВНЫЙ ПОДХОД ПРИ ИССЛЕДОВАНИИ ПОВЕРХНОСТИ С ПОМОЩЬЮ АТОМНО-СИЛОВОГО МИКРОСКОПА

Разработан новый режим исследования поверхности "MD mode", основанный на методе атомно-силовой микроскопии. Режим "MD mode" позволяет исследовать любые непроводящие поверхности со сложным рельефом и большими перепадами высот. При этом исключается неконтролируемое взаимодействие зонда с образцом и артефакты, вызываемые процессом сканирования. Приведены экспериментальные результаты, демонстрирующие возможность использования атомно-силового микроскопа с режимом "MD mode" для измерения ряда характеристик поверхности.

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