Technical Physics Letters, 2024, Vol. 50, No. 8

13.1;15

Wear of the probe tip depending on the interaction regimes with the sample surface at operating in the amplitude-modulation atomic force microscopy mode

© A.V. Novak[1,2], V.R. Novak[3], A.V. Rumyantsev[1]

[1] National Research University of Electronic Technology, Zelenograd, Moscow, Russia
[2] JSC „Angstrem“, Moscow, Russian Federation
[3] „NT-MDT Spectrum Instruments “, Moscow, Russian Federation
E-mail: novak-andrei@mail.ru

Received March 7, 2024
Revised April 10, 2024
Accepted April 15, 2024

The dependence of probe tip wear on the regimes of force interaction with the sample surface at operating in the amplitude-modulation atomic force microscopy mode has been studied. It was found that in the regime of attractive forces, the wear of the tip is insignificant, in contrast to the regime of repulsive forces. Thus, after 10 scans of a hard, rough surface of polysilicon films with hemispherical grains (HSG-Si) in the attractive regime, the tip radius increased from 3 to 4 nm, and in the repulsive regime from 4 to 20 nm. An estimate was made of the dissipation energy for one oscillation period Edis, which was: 2.1 eV in the attractive regime, and 98 eV in the repulsive regime.

Keywords: amplitude-modulation atomic force microscopy, probe tip wear, attractive force regime, repulsive force regime, dissipation energy.

DOI: 10.61011/TPL.2024.08.58910.19915

Atomic force microscopy (AFM) is based on measuring the force interaction of a probe tip with a sample. A certain degree (depending on the interaction mode) of probe tip wear occurring in the process of scanning the sample surface is inevitable. The amplitude-modulation AFM mode, which is also called the tapping or intermittent contact mode, is used widely in AFM studies [1–4]. In amplitude-modulation AFM, cantilever oscillations are excited at a frequency close to the resonant one with amplitude A_0 of free oscillations falling within the 5–100 nm range [3]. The tip is then brought to the surface of the sample. The approaching probe tip starts interacting with the surface at a certain distance from it, and oscillation amplitude A decreases accordingly. A certain „working“ value of oscillation amplitude A is set and maintained constant in the process of surface scanning by means of a feedback system. Two different regimes of force interaction between the tip and the sample are feasible in this case: attraction and repulsion. According to the definitions introduced in [2], in the attractive regime the force of interaction of the tip with the sample surface, averaged over one period of oscillation, is negative, and in the repulsive regime it is positive. A significant number of studies focused on the problem of tip wear (especially in contact AFM) have already been published [5]. In the case of amplitude-modulation AFM, the influence of such parameters as amplitude A_0 of free oscillations of a probe, working amplitude A, the approach speed, the integral feedback coefficient, the repulsive force magnitude, and the type of the tip coating material on tip wear was examined [6–8]. However, it should be noted that the majority of studies into tip wear were performed in the repulsive force regime and for tip sizes of approximately 20 nm (or more).

In the present study, we examine the wear of the tip of a silicon cantilever probe and estimate the magnitude of force interaction between the tip and the sample surface in the attractive and repulsive regimes of amplitude-modulation AFM. Relatively sharp probes with an initial tip radius smaller than 5 nm were studied.

AFM measurements were carried using a Solver P47 microscope (NT-MDT SI). Silicon NSG10 cantilevers with the same characteristics were used: the resonance frequency was 252 kHz, the force constant was 18 N/m, and the needle tip radius was smaller than 5 nm. The tip wear resulting from a certain number of scans (ten scans 520 × 520 nm in size containing 512 × 512 points) of a fairly hard and rough surface (polysilicon film with hemispherical grains, HSG-Si) was studied. The HSG-Si film was a layer of roughly identical Si grains on the surface of a flat substrate (the average height and the lateral size of grains were 50 and 70 nm) [9,10]. The cantilever tips were examined with a scanning electron microscope (SEM) before and after AFM measurements.

The implementation of a certain interaction regime (attraction or repulsion) depends heavily on the set values of amplitude of free oscillations A_0 and working amplitude A (adjustable during setup), the tip radius, the force constant and frequency of oscillations of the cantilever, and the viscoelastic properties of the sample [2,4,11,12]. The key characteristics allowing one to determine the domain

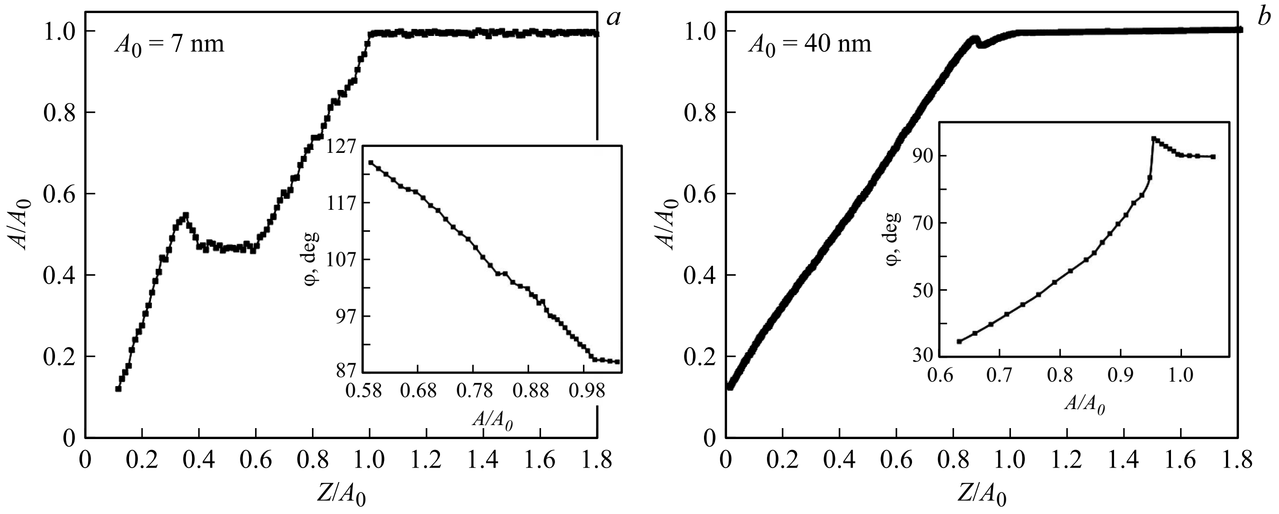

Figure 1. Dependences of probe oscillation amplitude A on distance Z between the tip and the sample normalized to amplitude A_0 of free oscillations and of phase shift φ on ratio A/A_0 (in the insets) measured at $A_0 = 7$ (*a*) and 40 nm (*b*).

of existence of a particular interaction regime are the dependences of amplitude A and phase shift φ of cantilever oscillations (relative to oscillations of the exciting force) on the tip–sample distance measured at certain amplitudes of free oscillations A_0 [2–4]. Notably, the phase shift φ measurement is the most practical and efficient method for identifying the regime of force interaction [2]. According to [2,4,11,13], phase shift φ of oscillations of the probe in the attractive regime is greater than 90°, while φ is lower than 90° in the repulsive regime.

Figure 1 shows the dependences of probe oscillation amplitude A on distance Z between the tip and the sample normalized to amplitude A_0 of free oscillations (A/A_0 on Z/A_0) and of phase shift φ on ratio A/A_0 measured at free oscillation amplitude $A_0 = 7$ (*a*) and 40 nm (*b*). The dependences obtained at $A_0 = 7$ and 40 nm are similar to the typical dependences observed for sufficiently small and large amplitudes of free oscillations A_0 [2,4,11,12]. In the case of small-amplitude oscillations ($A_0 = 7$ nm, Fig. 1, *a*), the attractive regime is manifested first in the $A/A_0(Z/A_0)$ dependence with a reduction in distance Z. As distance Z decreases further, a jump-like transition to the repulsive regime occurs at a certain value ($A/A_0 \sim 0.4-0.6$). The phase shift dependence provides direct confirmation of the fact that the attractive regime is implemented at $A_0 = 7$ nm and $A/A_0 > 0.6$. This dependence reveals that phase shift $\varphi > 90°$, which corresponds, according to [2], to the attractive regime, at A/A_0 ranging from 1 to 0.6. At $A_0 = 40$ nm and larger values of A_0 [2], the transition from the attractive regime to the repulsive one occurs almost immediately when distance Z starts decreasing; in contrast to the case of small A_0 values, this transition is not sharp in the $A/A_0(Z/A_0)$ curve, and the transition in the $\varphi(A/A_0)$ dependence occurs abruptly (at $A/A_0 = 0.95$) as φ decreases from 95 to 85°. Thus, the repulsive regime is implemented at $A_0 = 40$ nm and $A/A_0 < 0.95$. Operating parameters $A_0 = 7$ nm, $A/A_0 = 0.95$ were selected for the attractive regime, and $A_0 = 40$ nm, $A/A_0 = 0.90$ were set to implement the repulsive regime. Two cantilevers were used. Each of them made ten scans of the HSG-Si film surface (the first cantilever was operated in the attractive regime, and the second one was operated in the repulsive regime). SEM images obtained before AFM measurements and after ten scans are shown in Fig. 2, *a* (the first cantilever, attractive regime) and Fig. 2, *b* (the second cantilever, repulsive regime). In the attractive regime, the tip radius increased only slightly (from 3 to 4 nm; i.e., by a factor of 1.3) after ten scans (Fig. 2, *a*). In the repulsive regime (Fig. 2, *b*), the tip radius increased „catastrophically“ from 4 to 20 nm (i.e., by a factor of 5).

Figure 3 shows the AFM images of the HSG-Si film after the first and tenth scans in the attractive (*a*) and repulsive (*b*) regimes. It is evident that dark regions, where the tip penetrates to the flat substrate sections lying between Si grains during scanning, are resolved fairly well in both the first and the tenth scans in the attractive regime, while the area of these dark regions decreases significantly in the tenth scan in the repulsive regime. This is a direct indication of blunting of the tip. The key parameters characterizing surface roughness (specifically, relative surface area increase S_{dr}; see ISO 25178−2012) were calculated based on the AFM images. According to [9,10], parameter S_{dr} for the HSG-Si film sample is the one most sensitive to probe sharpness and may be used to estimate the amount of tip wear. Specifically, at tip radii of 4 and 14 nm measured after scanning, the S_{dr} parameter value was 80 and 34%, respectively [9]. In the present case, S_{dr} decreases insignificantly in the attractive regime: from 67% (first scan) to 59% (tenth scan). In the repulsive regime, S_{dr} decreases noticeably and monotonically from 46% (first scan) to 37% (second scan), 32% (third scan), etc. The parameter value corresponding to the tenth scan is 20%.

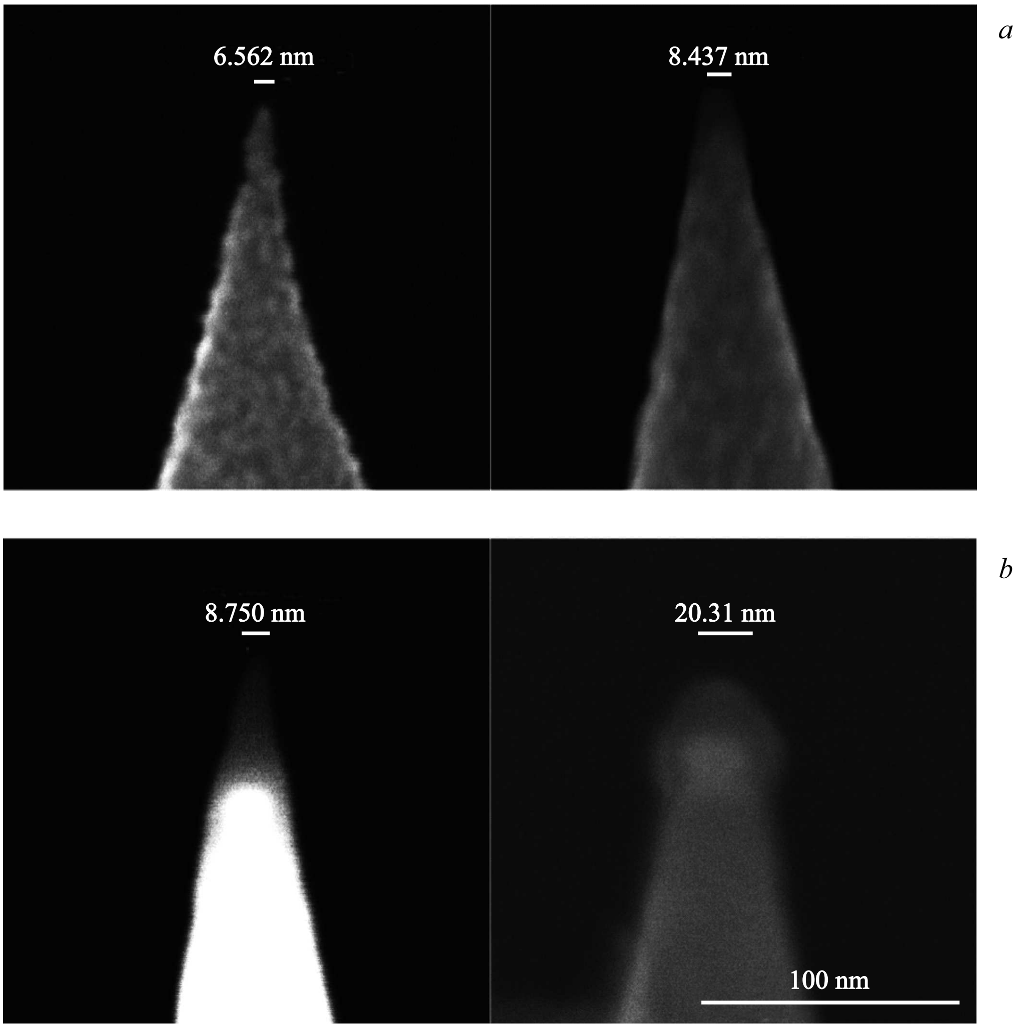

Figure 2. SEM images of two needle tips prior to (left) and after (right) of AFM measurements. *a* — The first cantilever (attractive regime); *b* — the second cantilever (repulsive regime).

This is indicative of a significant increase in the tip radius and demonstrates that wear occurs largely in the process of scanning.

The measurement of phase shift φ of probe oscillations relative to the exciting force allows one to make a quantitative assessment of the force interaction magnitude (namely, the dissipation energy lost due to interaction of the tip with the sample). Analytical expressions relating the dissipation energy caused by the tip–sample interaction to phase shift φ have been obtained earlier in [1,3]. The relations given in [1,3,13] yield the following expression for energy E_{dis} dissipated in one period of oscillations (in the case when the oscillation frequency matches the resonant one):

$$E_{dis} = \frac{\pi k A A_0}{Q}\left(\sin\varphi - \frac{A}{A_0}\right), \tag{1}$$

where A is the tip oscillation amplitude, A_0 is the free oscillation amplitude, k is the cantilever rigidity, and Q is the quality factor. In the present case, the dissipation energy in one period of oscillations is $E_{dis} = 2.1$ eV in the attractive regime ($A_0 = 7$ nm, $A/A_0 = 0.95$) and $E_{dis} = 98$ eV in the repulsive regime ($A_0 = 40$ nm, $A/A_0 = 0.90$).

Thus, it was demonstrated that the use of a sufficiently small amplitude of probe oscillations, which ensures operation in the attractive regime, allows for a significant freduction of probe tip wear (compared to the repulsive regime). A sufficiently high resolution of the resulting AFM image is achieved in this case. Thus, after ten scans of a fairly hard and rough surface in the attraction regime (at $A_0 = 7$ nm, $A/A_0 = 0.95$ and the value of dissipation energy for the oscillation period $E_{dis} \sim 2$ eV), the tip radius increased insignificantly (from 3 to 4 nm), and in the repulsive regime (at$A_0 = 40$ nm, $A/A_0 = 0.90$ and the value $E_{dis} \sim 98$ eV), the tip radius increased from 4 to 20 nm.

Conflict of interest

The authors declare that they have no conflict of interest.

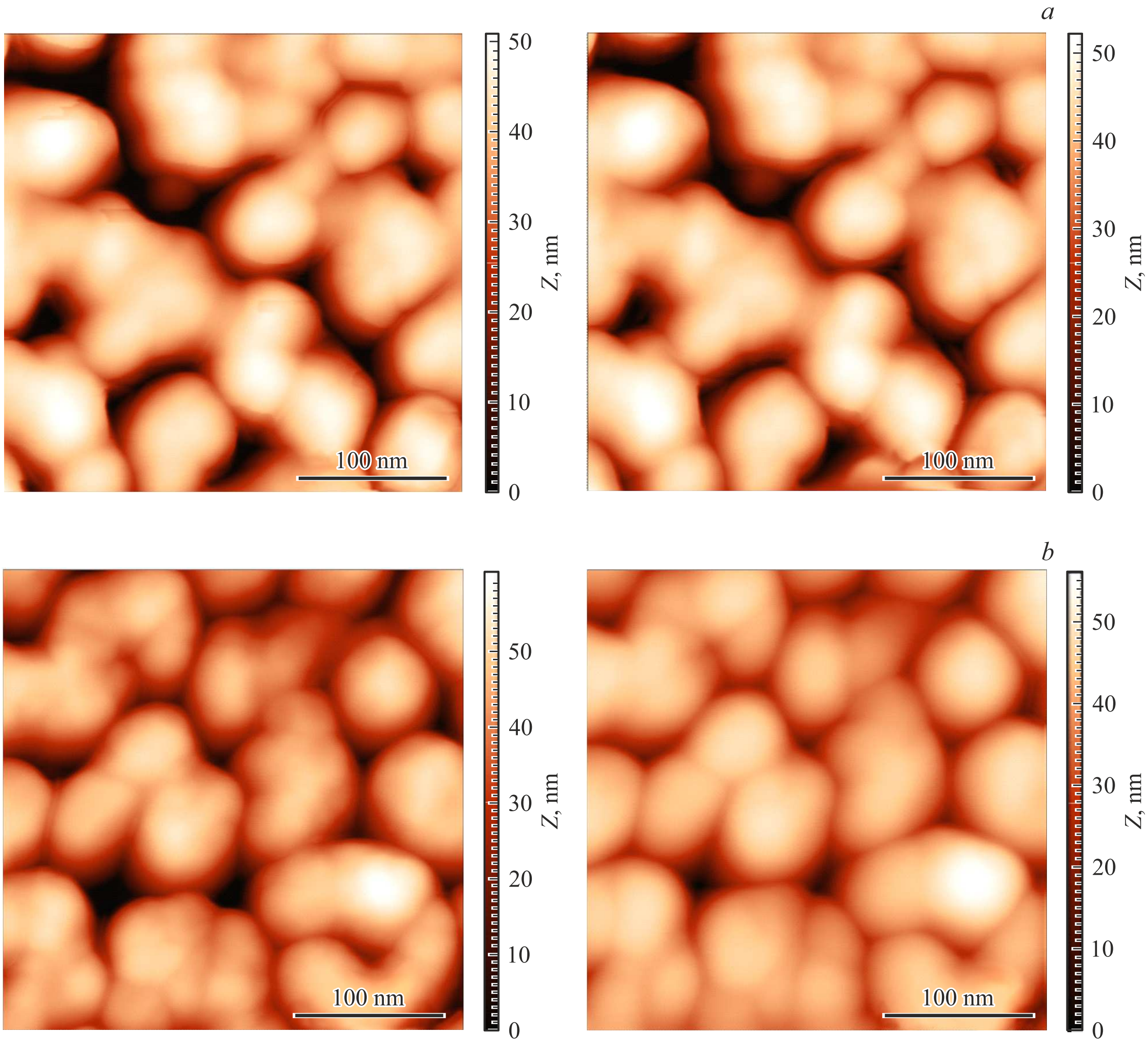

Figure 3. AFM images of the HSG-Si film after the first (left) and tenth (right) scans in the attractive (*a*) and repulsive (*b*) regimes.

References

[1] J.P. Cleveland, B. Anczykowski, A.E. Schmid, V.B. Elings, Appl. Phys. Lett., **72**, 2613 (1998). DOI: 10.1063/1.121434

[2] R. García, A. San Paulo, Phys. Rev. B, **60**, 4961 (1999). DOI: 10.1103/PhysRevB.60.4961

[3] A. San Paulo, R. García, Phys. Rev. B, **64**, 193411 (2001). DOI: 10.1103/PhysRevB.64.193411

[4] R. García, R. Pérez, Surf. Sci. Rep., **47**, 197 (2002). DOI: 10.1016/S0167-5729(02)00077-8

[5] S. Huang, Y. Tian, T. Wang, Sensors, **23**, 4084 (2023). DOI: 10.3390/s23084084

[6] C. Su, L. Huang, K. Kjoller, K. Babcock, Ultramicroscopy, **97**, 135 (2003). DOI: 10.1016/S0304-3991(03)00038-X

[7] V. Vahdat, D.S. Grierson, K.T. Turner, R.W. Carpick, ACS Nano, **7**, 3221 (2013). DOI: 10.1021/nn403435z

[8] B. Xue, Y. Yan, Z. Hu, X. Zhao, Scanning, **36**, 263 (2014). DOI: 10.1002/sca.21099

[9] A.V. Novak, V.R. Novak, A.V. Rumyantsev, Izv. Vyssh. Uchebn. Zaved., Elektron., **26** (3-4), 234 (2021). DOI: 10.24151/1561-5405-2021-26-3-4-234-245

[10] A.V. Novak, V.R. Novak, J. Surf. Investig., **10**, 949 (2016). DOI: 10.1134/S1027451016050104.

[11] S. Santos, L. Guang, T. Souier, K. Gadelrab, M. Chiesa, T.P. Thomson, Rev. Sci. Instrum., **83**, 043707 (2012). DOI: 10.1063/1.4704376

[12] A.G. Temiryazev, A.V. Krayev, M.P. Temiryazeva, Beilstein J. Nanotechnol., **12**, 1226 (2021). DOI: 10.3762/bjnano.12.90

[13] N.F. Martínez, R. García, Nanotechnology, **17**, S167 (2006). DOI: 10.1088/0957-4484/17/7/S11

Translated by D.Safin