DOI: 10.21122/2220-9506-2026-17-2-141-150

Complex Influence of Probe–Sample System Parameters on the Probe Oscillation Mode and Formation of Bistability in Tapping Atomic Force Microscopy

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Received 04.03.2026
Accepted for publication 04.05.2026

Abstract

Engaging the tapping mode in atomic force microscopy does not guarantee intermittent-contact interaction; depending on the parameters combination, the probe–sample system may instead exhibit non-contact (attractive) or mixed behavior. The mixed mode is undesirable due to scanning instability, known as bi-stability of probe oscillations. Aim of this work was to identify influence of probe (spring constant and quality factor), sample (the Young modulus, the Hamaker constant), and scanning (oscillation amplitude of the piezoelectric generator) parameters on the implementation of the bistable interaction mode, as well as to find conditions for achieving stable semi-contact interaction between the tip and sample. Equation of probe tip oscillations was solved taking into account elastic-adhesive contact of the tip and the sample according to the Johnson–Kendall–Roberts model. Dependencies of the dynamic interaction characteristics of the probe and sample on the distance between them were obtained. These dependencies were analyzed to determine if there was switching between repulsive and attractive interaction modes. Results clearly demonstrated which of the dynamic interaction modes was realized with a particular combination of parameters. Conclusions are applicable in practice to choose probes. The following regularities were obtained. The elastic (actually semi-contact, tapping) interaction mode is realized with an increase in the amplitude of piezoelectric generator oscillations, higher values of the spring constant and quality factor of the probe, the Young modulus of the sample material and/or at lower values of the Hamaker constant of the sample. Higher values of the Hamaker constant and/or lower values of other parameters lead to a mixed interaction regime. Having even higher Hamaker constant and further reduction in other parameters, the adhesion mode (non-contact atomic force microscopy) can be realized.

Keywords: atomic force microscopy (AFM), tapping mode, the Young modulus, the Hamaker constant, bi-stability

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Для цитирования:
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Комплексное влияние параметров системы зонд–образец на режим колебаний зонда и формирование биустойчивости в полуконтактной атомно-силовой микроскопии

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Поступила 04.03.2026
Принята к печати 04.05.2026

Включение полуконтактного режима атомно-силовой микроскопии не гарантирует реализации именно прерывисто-контактного взаимодействия: в зависимости от сочетания параметров система зонд–образец может вместо этого проявлять бесконтактное (притягивающее) либо смешанное поведение. Последнее является нежелательным из-за нестабильности сканирования, известной как биустойчивость колебаний зонда. Целью данной работы являлось выявление влияния каждого из комплекса параметров зонда (изгибная жёсткость и добротность), образца (модуль Юнга, постоянная Гамакера) и сканирования (амплитуда колебаний пьезогенератора) на реализацию биустойчивого режима взаимодействия, а также нахождение условий достижения стабильного полуконтактного взаимодействия зонда и образца. Решалось уравнение колебаний острия зонда с учётом упруго-адгезионного контакта зонда и образца по модели Джонсона–Кенделла–Робертса. Получены зависимости характеристик динамического взаимодействия зонда и образца от расстояния между зондом и образцом. Зависимости проанализированы на предмет переключения между отталкивающим и притягивающим режимами взаимодействия. Результаты наглядно демонстрируют, какой из режимов динамического взаимодействия реализуется при том или ином сочетании параметров, и применимы на практике для выбора зондов. Получены закономерности: упругий (собственно полуконтактный) режим взаимодействия реализуется при повышении амплитуды колебаний пьезоэлемента, более высоких значениях жёсткости и добротности зонда, модуля Юнга материала образца и/или при более низких значениях постоянной Гамакера образца. Более высокие значения постоянной Гамакера и/или меньшие значения остальных параметров приводят к смешанному режиму взаимодействия. При еще более высокой постоянной Гамакера и дальнейшем снижении величин других параметров возможна реализация адгезионного режима (бесконтактная атомно-силовой микроскопия).

Ключевые слова: атомно-силовая микроскопия (АСМ), полуконтактный режим, модуль Юнга, постоянная Гамакера, биустойчивость колебаний зонда

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Introduction

The application area of AFM (atomic force microscopy) tapping mode is significantly broader than static AFM, as the probe is in contact with the sample surface for a small part of its oscillation cycle. Consequently, the probe tip impact on the sample is very gentle, which is especially important for low-modulus ("soft") samples, including biological ones. However, there are a number of cases where scanning artifacts related to tip dynamics occur in dynamic AFM. Since this occurs under specific tip–sample interaction conditions, this problem can be resolved by identifying the combinations of tip, sample, and scanning parameters that trigger this instability.

The dynamic interaction between the probe and the sample is influenced by a complex of parameters: probe (spring constant, quality factor, natural frequencies, tip radius), sample (elastic modulus, surface energy), and scanning (oscillation amplitude of piezoelectric generator, operating frequency of the probe) [1]. The interaction forces between the tip and the sample may vary, but have a common feature – dependence on the position of the tip relative to the sample surface, i.e., on the tip–sample gap.

The majority of studies devoted to the tip motion in the tapping mode of interaction with the sample surface use the Deryagin–Muller–Toporov (DMT) model [2] to define the tip–sample interaction forces. The tip senses the forces at the bottom of the oscillation cycle. The model includes attractive intermolecular van der Waals interactions acting at some small tip–sample distances. During short-term contact between the tip and the sample surface, the Hertzian elastic repulsion force of the deformed sample is taken into account. The advantages of this approach are the relative simplicity of calculations, the inclusion of the sample and tip Young moduli, and the inclusion of the surface energy of the sample using the Hamaker constant.

This led to the discovery of a bi-stability phenomenon – the ability to achieve the same tip oscillation amplitude at two different distances z_{pos} between the cantilever and the sample [3, 4]. It was found that such situations arise due to the competition between the attractive and repulsive tip–sample interactions. For rigid samples, bi-stability arises due to collisions between the probe tip and the sample surface even in the absence of interaction competition [5].

The AFM feedback system maintains a constant amplitude A_1 during scanning by varying the distance z_{pos}, which can become equal to either z_1 or z_2 (Figure 1). The predominance of attractive forces is reflected by the branch of lower probe amplitudes (on the right in Figure 1), while repulsive forces are reflected by the branch of higher amplitudes (on the left). Thus, if the probe oscillation amplitude specified by the operator falls within the region of existence of two amplitude branches, this leads to unexpected jumps between the distances z_1 and z_2, corresponding to the predominance of repulsion and attraction between the probe and sample. As a consequence of this ambiguity, defects appear on the AFM image. Hence, understanding the influence of the tip–sample system parameters on the interaction mode and finding ways to avoid scanning-unfavorable situations are critical.

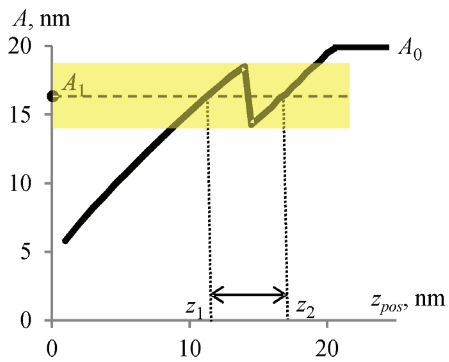


Figure 1 – Dependence of the tip oscillation amplitude on the distance to the sample

The problem of bi-stability and the influence of probe–sample system parameters were particularly actively studied immediately after its discovery. Thus, in [6] it was found that increasing the probe spring constant k leads to the elimination of bi-stability. In [7], numerical modeling showed that the curvature radius of the probe tip R leads to an increase in the van der Waals force in the region of attractive forces and an increase in the elastic repulsive force in the corresponding region of the curve.

It was found in [8] that the contribution of van der Waals forces can be significantly reduced by increasing the excitation amplitude of the probe oscillations, leading to an almost purely repulsive mode. The non-contact (attractive) mode becomes dominant at small excitation amplitudes. It was also shown that an increase in the product HR (the Hamaker constant of the sample and the curvature radius of the tip) leads to the realization of the non-contact mode (with other suitable parameters), while a decrease in the product leads to a repulsive (semi-contact) mode.

It was shown in [9] that an increase in the amplitude of forced oscillation of the tip away from the surface A_0 together with an increase in the Young modulus E of the sample leads to the implementation of the repulsion mode. Bi-stability arises with a decrease in A_0 and E. An attraction mode is formed at sufficiently low A_0 and E.

It was found in [3] that an increase in the force, which forces the probe to oscillate and depends primarily on the amplitude oscillation of the piezoelectric generator, can lead to the elimination of bistability. The same effect is produced by directly increasing the amplitude of the forced oscillations of the tip away from the surface A_0 [10].

An example of mathematical modeling using the DMT model is presented in [11], which shows that increasing the amplitude of the cantilever oscillations away from the surface A_0 allows to avoid instability of the probe oscillations during scanning a sample with high surface adhesion.

Later, studies appeared that took into account viscoelastic properties of samples. For example, in [12], a viscous force dependent on the sample thickness was added to the DMT model. The authors report that, according to the simulation results, too high the reduced Young modulus or too low a sample viscosity leads to bi-stability.

The studies conducted do not reveal mutual influences between probe–sample system parameters, as only one or two parameters were varied, while the others were fixed. Therefore, although general trends were identified, the conclusions are guaranteed to apply only to the combinations of parameters examined.

A breakthrough was the discovery of an approximate solution to the equation of tip motion during interaction with a sample in general form for both amplitude-modulated AFM (in which the feedback parameter is the probe oscillation amplitude) and frequency-modulated AFM [13]. Unfortunately, using the solution for practical purposes appears difficult.

Thus, the problem of choosing probe and scanning parameters to obtain high-quality AFM images remains relevant. The problem of scanning instability is particularly relates to the research of low-modulus and/or highly adhesive ("sticky") material samples.

The objective of the work was to find conditions for achieving a stable semi-contact interaction between the probe and the sample, taking into account contact adhesion [2].

Mathematical model and simulation

Previously, we proposed and applied model (1) to simulate the process of probe tip motion in intermitted (tapping) mode of interaction with sample. Its advantage is that it takes into account non-contact and contact adhesion. It is based on the Johnson–Kendall–Roberts model for contact between a sphere and a plane. Due to the self-consistent approach to describing the contact of two surfaces, it is known that the applicability range of the Johnson–Kendall–Roberts model is lower modulus and higher adhesive materials than for the DMT model. In addition, model (1) takes into account short-range interatomic repulsive interactions. The coefficients in the Lennard–Jones potential, which is used to introduce noncontact attractive and repulsive interactions, correspond to high adhesive interactions between the tip and the sample.

The equation of probe tip oscillations is:

$$mz'' + \frac{m\omega_0}{Q} z' + k\left(z - z_{pos}\right) = a_{bm} k \sin\left(\omega t\right) + F_{ts}\left(z\right). \qquad (1)$$

Here is the force of probe–sample interaction

$$F_{ts}\left(z\right) = \begin{cases} F_{\mathrm{LJ}}\left(z\right), \ z > z_{F_0} \\ F_{\mathrm{JKR}}\left(z\right), \ z \le z_{F_0} \end{cases},$$

$$F_{\mathrm{LJ}}\left(z\right) = -\frac{HR}{6}\left(\frac{1}{z^2} - \frac{\sigma^6}{4z^8}\right),$$

$$F_{\mathrm{JKR}}\left(\frac{z}{z_{\mathrm{c}}}\right) = \left(\frac{z}{z_{\mathrm{c}}}\left(\frac{F_{\mathrm{JKR}}}{P_{\mathrm{c}}}\right)\right)^{-1},$$

$$\frac{z}{z_{\mathrm{c}}} = \begin{cases} -\left(3\sqrt{\frac{F_{\mathrm{JKR}}}{P_{\mathrm{c}}} + 1} - 1\right)\left[\frac{1}{9}\left(\sqrt{\frac{F_{\mathrm{JKR}}}{P_{\mathrm{c}}} + 1} + 1\right)\right]^{1/3}, \ \frac{z}{z_{\mathrm{c}}} \le 3^{\ 2/3}, \\ \left(3\sqrt{\frac{F_{\mathrm{JKR}}}{P_{\mathrm{c}}} + 1} + 1\right)\left[\frac{1}{9}\left(1 - \sqrt{\frac{F_{\mathrm{JKR}}}{P_{\mathrm{c}}} + 1}\right)\right]^{1/3}, \ 3^{\ 2/3} \le \frac{z}{z_{\mathrm{c}}} \le 1, \end{cases}$$

$$z_c = \frac{1}{3R}\left(\frac{3RP_c}{k_s}\right)^{2/3},$$

$$P_c = \frac{3}{2}\pi R \Delta\gamma, \ \ \Delta\gamma = \frac{H}{16\pi\sigma^2},$$

$$k_{\mathrm{s}} = \frac{4}{3\pi} \cdot \frac{1}{\kappa_1 + \kappa_2};$$

$$\kappa_i = \frac{1 - \nu_i^2}{\pi E_i}, \ \ i = \overline{1, 2}.$$

Initial conditions are $z(0) = z_{pos}$, $z'(0) = 0$.

Here z_{pos} is the position of a cantilever fixing point above a sample surface, nm; m is the mass of the microprobe, kg; z is vertical displacement of the tip probe, nm; t is time, s; ω_0 is the natural angular frequency of the probe, Hz; Q is the quality factor of the cantilever; k is the spring constant of the probe cantilever, N/m; a_{bm} is the oscillation amplitude of the piezoelement, on which the cantilever is fixed, nm; ω is the operating angular frequency of the probe, Hz; F_{ts} is the interaction force between the probe and the sample surface, nN; F_{LJ} is the non-contact interaction force, nN; F_{JKR} is the contact interaction force according to the JKR model, nN; z_{F_0} is a distance, at which the contact and non-contact interaction forces are balanced, nm; H is the Hamaker constant of a sample, 10^{-18} J; R is the radius of probe tip curvature, nm; σ is the interatomic distance, nm; P_c is the maximum force of adhesion, nN; Δγ is the specific surface energy of a sample, J/m^2; k_s is a reduced modulus of elasticity of sample and tip materials, GPa; v_1, v_2 are the Poisson's ratios of tip and sample materials, respectively; E_1, E_2 are the Young moduli of tip and sample materials, respectively, GPa [14, 15].

The equation (1) was solved using methods of mathematical simulation. In the simulation, the distance z_{pos} between the attachment point of the cantilever and a sample was varied to reproduce its changes during process of AFM scanning. It was varied from zero to values close to the free oscillation amplitude of the tip.

In the simulation, the following parameters were varied: Young modulus of the sample material (from 0.01 to 10 GPa); the Hamaker constant characterizing the surface energy of a sample ($0.1–0.3\cdot10^{-18}$ J); the oscillation amplitude of the piezoelectric generator forcing oscillation of the microcantilever (from 0.5 to 2 nm); the spring constant of a probe cantilever (from 0.1 to 100 N/m); the quality factor of the probe cantilever (from 20 to 400) [1, 15]. The radius of curvature of the probe tip was set equal to 10 nm, Young modulus of probe material was 179 GPa (corresponds to silicon) [1, 14, 15]. The frequencies of probe oscillations were set equal to resonant: 38.9; 87; 152.8; 436 kHz for probes with spring constants of 0.1; 1; 5; 100 N/m, respectively.

Data representation structure

For each set (720 in total) of probe–sample interaction parameters, four dependences on the distance z_{pos} were obtained: the amplitude and phase shift of the probe oscillations, the probe–sample interaction force at the bottom of the tip oscillation cycle, and the sample deformation depth. An example for three types of probe–sample interaction is shown in Figure 2.

The dependencies were analyzed to determine if there is switching between repulsive and attractive interaction modes [1, 15]. The attractive mode corresponds to non-contact dynamic AFM. The repulsive mode is the actually semi-contact (tapping) mode. The interaction mode for a certain set of input parameters (E, H, a_{bm}, Q, k) was considered purely elastic (repulsive) if the curve of the probe oscillation amplitude versus distance z_{pos} is monotonic and the probe–sample interaction force at the bottom point of the probe oscillation cycle is positive over the entire interval $0.05 \le A/A_0 \le 0.95$ (Figure 2*a*, *b*) [1, 15]. Negative values of the probe oscillation phase shift φ relative to the probe oscillation phase far from the sample surface, as well as a non-zero deformation depth of the sample by the probe confirm the predominance of the elastic interaction mode (Figure 2*c*, *d*). The attractive (adhesive) interaction mode corresponds to a monotonic curve of the probe oscillation amplitude and a negative interaction force of the probe with the sample [1, 15]. Other cases are classified as a mixed interaction mode, in which switching occurs between the modes of attraction and repulsion of the probe and sample, accompanied by abrupt changes in the dependence of the probe oscillation amplitude on the distance z_{pos} [1, 15].

The results of such classification according to the modes of probe–sample interaction are summarized in tables containing diagrams (Tables 1–3). The axes of the diagrams show the variable parameters of the sample material: the Young modulus E and the Hamaker constant H. Each table corresponds to the probe quality factor Q: 20, 200, or 400. Each point on the diagrams corresponds to a single simulation run with a unique set of parameter values: E, H, a_{bm}, k, and Q. Each symbol on the diagram represents not just a point, but a data set – four dependencies similar to those shown in Figure 2. The symbols on the diagrams correspond to one of the interaction modes (elastic (tapping), attractive (non-contact), mixed). The main purpose of creating the diagrams is to systematize the voluminous array of simulation data and clearly visualize the parameter combinations that enable a particular AFM operating mode.

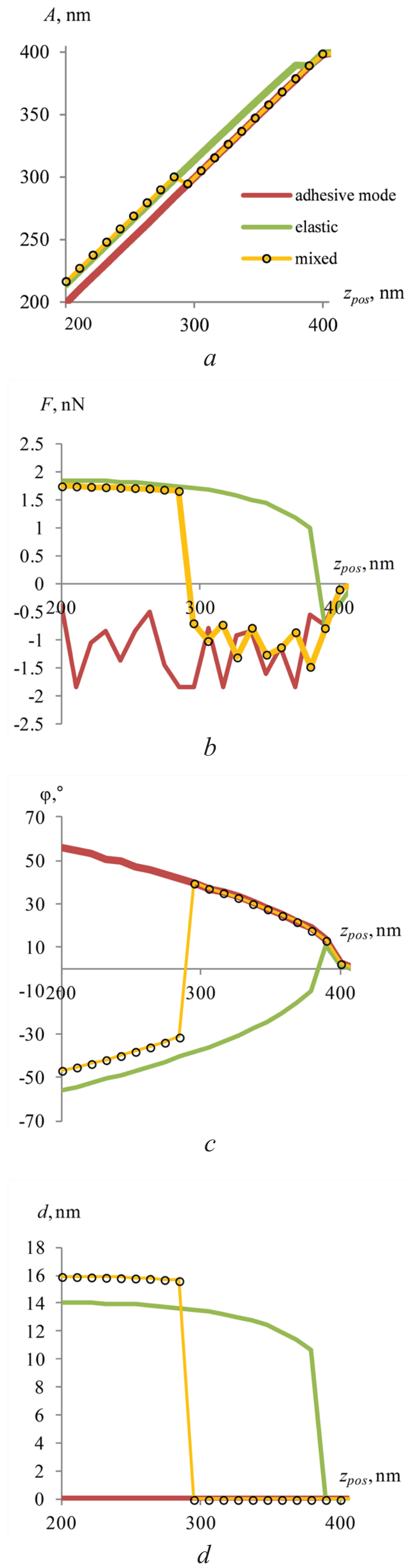


Figure 2 – Dependences of the interaction parameters of the probe and sample on the distance z_{pos} in elastic, adhesive and mixed modes: *a* – the oscillation amplitude of the tip; *b* – the probe–sample interaction force at the bottom point of its oscillation cycle; *c* – phase shift of the tip oscillation; *d* – the maximum deformation depth of the sample

There are cases of probe–sample interaction (marked by the symbol Ŧ in the diagrams) in which multiple chaotic jumps are observed in the interaction curves. In [15], it was established that these are not related to surface adhesion of the sample and were therefore classified as a conditionally elastic mode, which, however, is unfavorable for scanning.

Discussion

The elastic (i. e., semi-contact, tapping) mode of probe–sample interaction is realized when a cantilever with a higher spring constant *k* and quality factor *Q* is selected, the sample exhibits a higher Young modulus *E*, the oscillation amplitude of the piezoelectric generator a_{bm} is increased, and/or the sample has a lower Hamaker constant *H* [1]. A mixed regime arises in which attraction and repulsion alternate, if the Hamaker constant *H* is higher while the other parameters are the same or lower [1]. A further increase in *H* together with a reduction of the remaining parameters leads to the adhesive (non-contact AFM) mode.

Higher values of the Hamaker constant, with other parameters of the probe–sample interaction being fixed, make a greater contribution to the adhesive component of the interaction force and lead to an expansion and shift of the bi-stability region of the probe oscillations (Figure 3).

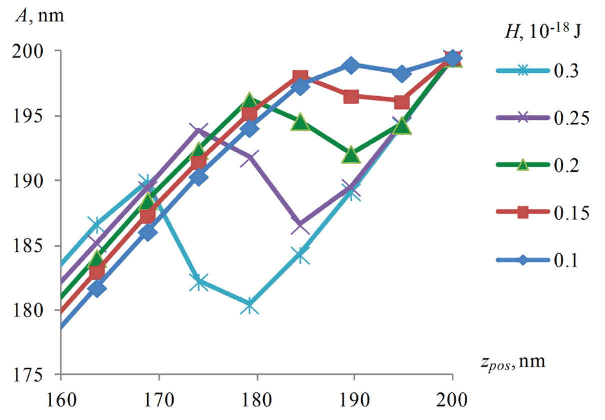
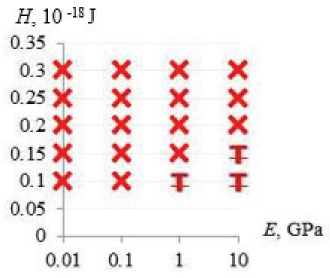
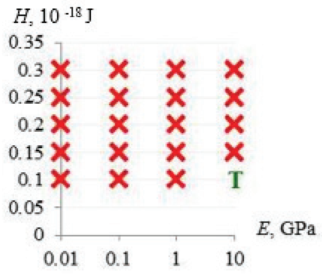
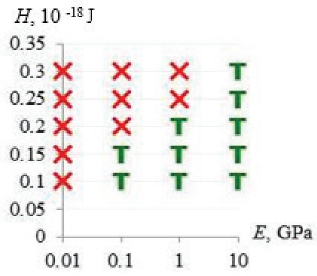
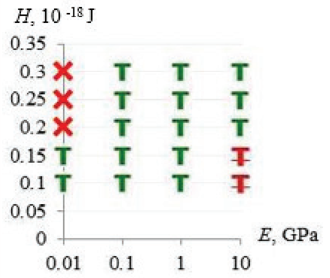
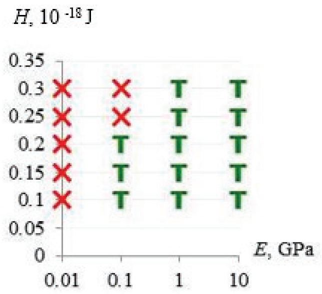
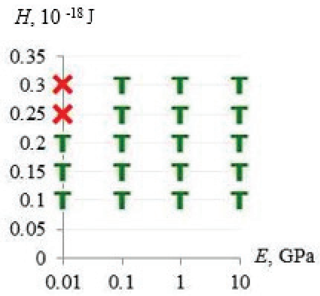


Figure 3 – Dependences of the probe tip oscillation amplitude on the distance to the sample z_{pos} with varying the Hamaker constant of the sample

During the simulation, the curvature radius of the tip was not varied, but some conclusions about its influence on the interaction mode can be obtained directly from the expressions of the mathematical model (1).

Table 1

Modes of probe–sample interaction at Q = 20

a_{bm}, nm k, N/m	0.5	1	2
0.1	MIXED MODE		
1			
5			TAPPING MODE
100	TAPPING MODE		

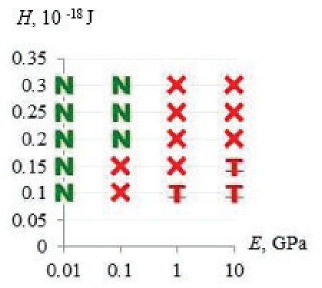
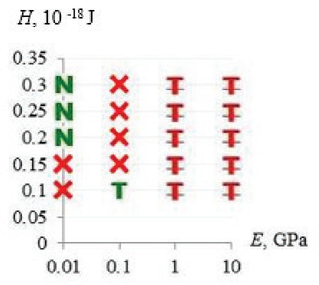
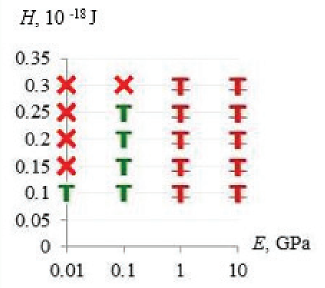
T – tapping mode, favorable for scanning;
X – mixed mode, not recommended for scanning;
Ŧ – unfavorable tapping mode.

The curvature radius and the Hamaker constant factors are included in the formula for the non-contact interaction force F_{LJ} to the first power, exactly the same as in the expression for the maximum adhesive force P_c. In the expression for the rupture point of tip–sample contact z_c the Hamaker constant is present to the 2/3 power, and the tip radius is present to the 1/3 power. Thus, changing the tip radius affects the tip–sample system dynamics similarly to changing the Hamaker constant: smaller tip radii are required to achieve an elastic tip–sample interaction mode.

The regularities observed here are in good agreement with data from earlier studies, including [3, 6–11], devoted to the influence of certain parameters of the probe–sample system on the formation of a bistable probe–sample interaction.

Table 2

Modes of probe–sample interaction at Q = 200

<table>
<tr><td>a_{bm}, nm
k, N/m</td><td>0.5</td><td>1</td><td>2</td></tr>
<tr><td>0.1</td><td>
</td><td>
</td><td>
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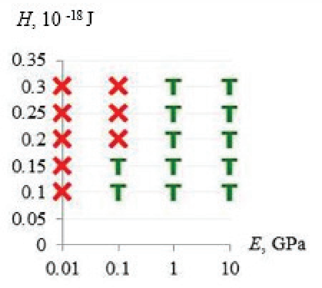
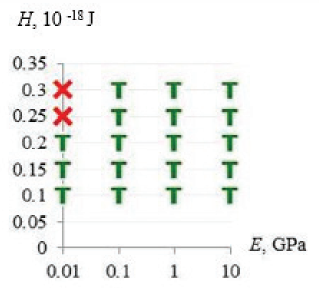
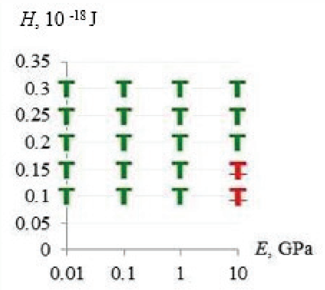
<tr><td>1</td><td>
</td><td>
</td><td>
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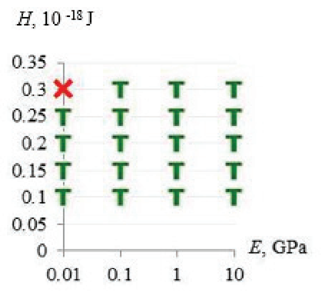
<tr><td>5</td><td>
</td><td colspan="2">TAPPING MODE</td></tr>
<tr><td>100</td><td colspan="3">TAPPING MODE</td></tr>
</table>

T – tapping mode, favorable for scanning;

N – non-contact mode;

X – mixed mode, not recommended for scanning;

Ŧ – unfavorable tapping mode.

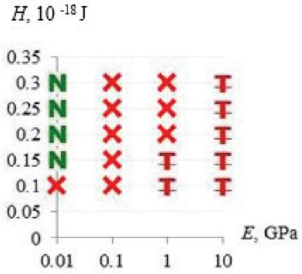
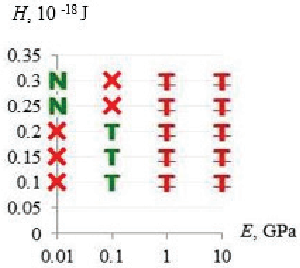
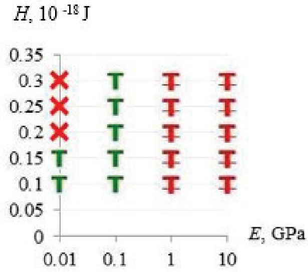
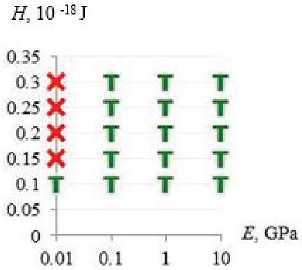
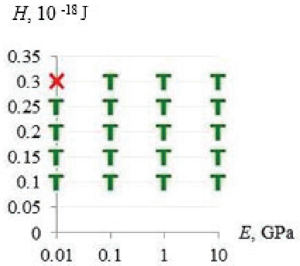
Based on the general patterns obtained, it seems advisable to take the steps to avoid scanning artefacts in dynamic mode: increase the piezoelement excitation amplitude; if that was insufficient, use the probe with a higher spring constant [1]. Using probes with the maximum spring constant at the outset is not advisable, as this increases the sample deformation depth and introduces height errors into the AFM images [1].

Examples of the results' practical use

Example 1. Let the quality factor and the probe spring constant be Q = 350, k = 3 N/m; the sample is a "hard" polymer (the Young modulus of the order of 10 GPa). According to Table 3 (for Q = 400) we find that for a probe spring constant of both 1 and 5 N/m and E = 10 GPa, a tapping mode favorable for scanning is realized.

Table 3

Modes of probe–sample interaction at *Q* = 400

<table>
<tr><td>a_{bm}, nm
k, N/m</td><td>0.5</td><td>1</td><td>2</td></tr>
<tr><td>0.1</td><td></td><td></td><td></td></tr>
<tr><td>1</td><td></td><td></td><td>TAPPING MODE</td></tr>
<tr><td>5</td><td colspan="3">TAPPING MODE</td></tr>
<tr><td>100</td><td colspan="3">TAPPING MODE</td></tr>
</table>

T – tapping mode, favorable for scanning;
N – non-contact mode;
X – mixed mode, not recommended for scanning;
Ŧ – unfavorable tapping mode.

Example 2. Let the quality factor and the probe spring constant be $Q = 150$, $k = 1.5$ N/m; the sample is a low-modulus material: E is about 0.01–0.1 GPa. According to Table 2 (for $Q = 200$), we find that for a probe spring constant of 1 N/m and the given Young modulus, either a tapping mode or a mixed interaction mode can be realized, depending on the oscillation amplitude of the piezoelectric generator. In this case, if bi-stability occurs, it is recommended to increase the piezoelectric generator amplitude. If no improvement occurs, increase the set-point parameter or choose a probe with a higher spring constant, for example, near 5 N/m.

Conclusion

Patterns have obtained for realizing elastic, adhesive, and mixed modes of dynamic probe–sample interaction, depending on combinations of their parameter values. Recommendations for the practical application of these results are provided.

Acknowledgments

The work was carried out within the framework of task 3.03.3 of the State Program for Scientific Research "Convergence-2025" for 2021–2025.

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