Self-assembly of the deposited graphene-like nanoparticles and possible nanotrack artefacts in AFM studies

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Abstract

Atomic force microscopy (AFM) is widely used for structural characterization of 2D materials. We report here on the appearance of linear pseudo-structures of subnanometer height (‘nanotracks’), observed in AFM images of 2D-nanoparticles of graphene, MoS$_2$, WS$_2$, BN, synthesized by the mechanochemical technique and deposited from dispersions on the mica surface. It is stated that the nanotracks appear as a result of nanoparticle displacement on the surface under the influence of the AFM tip during scanning. The appearance of the nanotracks is caused by a high relative concentration of monolayer nanoparticles in the prepared dispersions; their bulk aggregation; subsequent destruction of the aggregates by the AFM probe with formation of the nanoparticles weakly bound to the substrate. A method is proposed how to distinguish monolithic particles from granulated aggregates, as well as a technique to prevent their displacement during measurements. The possibility is considered of using AFM to develop effective nanolubricants and provide their precise nanoscale deposition on the specified surface areas.

Introduction

Numerous recent studies of atomically thick nanoobjects were stimulated by their unique physical properties. In particular, the works devoted to 2D materials (graphene and its analogs) paid much attention to their attractive mechanical characteristics and high lubricity [1–5], which make it possible to use graphene-like nanoparticles (NPs) as atomically thick lubricants. However, the friction mechanism in them is still not fully understood. The superlubricity mode, which seemingly should take place at the contact of any incommensurable atomically smooth clean plane [6], was actually detected in vacuum conditions for only a few interfaces. Their list was even more limited under ambient conditions and included only the contacts of two atomic planes of graphite or graphene [2, 7–10], as well as some recently discovered interfaces involving the plane of graphite on one side [11, 12]. Therefore, the investigation of NPs weakly bound to the substrate under nonvacuum conditions is of both practical and basic interest.

Atomic force microscopy (AFM) is one of the experimental tools most widely used for structural characterization of graphene-like NPs, which makes it possible to determine their thickness with subnanometer accuracy and study the tribological properties of particles at the nano-level [1, 11, 13]. Unfortunately, multiple artifacts which often appear in AFM images [14], can lead to misinterpretation of the data. For example, under certain conditions, one can get incorrect information about the thickness of graphene [15]. A common problem is the also unintentional displacement of NPs on a surface under the impact of the AFM probe during measurements. This can cause the complete removal or reduction of the number of NPs within the scan area and the accumulation of removed NPs at the raster boundaries [16, 17].
On the other hand, it is possible to purposefully move objects using an AFM probe. So far, such manipulations were carried out mainly with spherical particles with a diameter of several tens of nanometers. In early studies, this was done in the contact AFM mode using the direct pushing technique, where the force of the impact was tried to apply to the center of mass of the particle [18–20]. This was not always possible to realize, which led to a loss of contact with the particle during the pushing.

Later on, the manipulation operations were investigated in the semi-contact AFM mode combined with the lateral pushing technique [17, 21–26]. This made it possible to control the position of the particle at the moments of its interaction with the tip, which simplified the task of precise moving the particle to the given position.

In this work, we report on a new type of AFM artifact associated with sliding of a particle on the surface during scanning caused by a series of lateral impacts of the AFM probe with the particle. We discuss the appearance in images of linear objects of subnanometer height—‘nanotracks’, which were observed in the visualization of various graphene-like NPs deposited from dispersions on the atomically smooth surface of the substrate. The appearance of nanotracks in such systems was promoted by a number of factors leading to a decrease in the friction forces between the NPs and the substrate. Earlier we observed nanotracks in the case of 2D nanoparticles of BN [27]. Here we report on similar effects for 2D-NPs of other materials (graphene, MoS2, WS2). It should be noted that the nanotracks were revealed only in the images of 2D-NPs and were not observed with other nanosized objects observed by us. This paper discusses in detail the nanotracks appearance mechanisms, the methodical cautions associated with performance of AFM studies, as well as suggests some recommendations on the possibilities of application of the revealed effects in tribology. In particular, such problems may arise when the task will be to controllably transfer various nanoobjects or lubricate tiny mechanisms like nanorobots.

Experimental

Synthesis
All used dispersions of 2D particles were prepared by the method including preliminary nanostructuring of the corresponding bulk material by the solventless mechanochemical treatment in the presence of the chemically inert agent (NaCl) and subsequent ultrasonic disintegration of the nanostructured material in an organic solvent. The details of the synthesis and characterization of the prepared samples of 2D particles of graphene, MoS2, WS2, BN are described in [27–30].

AFM studies
AFM studies were performed using a ‘Solver Pro M’ system (NT-MDT). The samples were obtained by placing a drop of the dispersion with NPs on a freshly prepared atomic-smooth surface of muscovite mica (SPI supplies, V-1 grade). AFM studies were carried out after complete evaporation of the solvent. The concentration of the dispersions was chosen in such a way that a sufficient number of spatially separated NPs were located on the surface within the scanning areas.

The measurements were carried out in the amplitude modulation tapping mode [31] using ‘RTESPA’ type probes (Bruker, 300 kHz, 40 N m−1). The free oscillations’ amplitude of the cantilever (A0) was ~100 nm. The interaction force between the vibrating tip and the surface (load) was set by the ‘setpoint’ parameter (i.e., the amplitude ratio $A/A_0$), where $A$ is the oscillation amplitude which was maintained at a constant level by the feedback circuit during the measurements. Setting the smaller setpoint value (SP) increases the load.

The scales of the AFM images were chosen in such a way that the NPs be clearly discernible in them. To attain this, the distance between the adjacent points (lines) of the raster must be at least several times smaller than the apparent lateral size of NPs. Due to this, these distances were chosen as follows: 2–40 nm (figures 1(a), 2(b), (d); 40 nm; figures 1(b), 3(a)–(c); 5 nm; figure 1(c); 2 nm; figure 2(a); 22.5 nm; figures 2(c), (e), (f); 10 nm). In the shown AFM images, the fast-scanning direction was horizontal (x-axis), and the slow-scanning direction (SSD or y-axis) was from bottom to top.

Results and discussion

In our experiments, 2D-NPs were used whose lateral sizes usually did not exceed 50 nm, and the characteristic thickness was 0.6–2.5 nm, which corresponds to the thickness of one or several atomic layers (figure 1).

However, the deposited layers often contained also larger objects several nanometers or more in height, which we call here ‘multi-nanometer particles’ (MNP, plural—MNPs). Their emergence depends on many factors including the conditions of particles’ deposition and even on the choice of scanning area. The reasons for appearance of the MNPs will be discussed below.
We have found a relationship between the nanotracks appearance and the presence of MNPs on the surface. The nanotracks, as a rule, were not observed in the absence of MNPs (figure 1). Moreover, the nanotrack started in most cases from the MNP (e.g. labels A in figure 2) and then extended to the upper line of the raster. If the nanotrack ended in the middle of the raster, then the MNP was also observed at its endpoint (e.g. labels B in figures 2(e), (f)). The height of nanotracks usually did not exceed 0.5 nm, but in some cases it could reach 3 nm (see Z-profiles in figure 2).

It was stated that the number of nanotracks sharply decreased when changing some scanning parameters that reduce the AFM probe load on the surface. In particular, this was attained by increasing the SP parameter (see Experimental). To evaluate the effect of the probe load on the stability of AFM images, three consecutive scans were taken at different SPs over the same surface area (figure 3). The first (figure 3(a)) and the third (figure 3(c)) scans were obtained at a minimum load (SP ≈ 0.82), at which the device still retained its sensitivity to the surface topography. It turned out that nanotracks were absent in these images. The second scan (figure 3(b)) was recorded at a higher load (SP ≈ 0.65). This value is close to that used in taking the images shown in figure 2 (SP ≈ 0.5–0.75). The presence of nanotracks in figure 3(b) and their absence in figures 3(a) and (c) clearly confirms that the nanotracks are actually tip-induced pseudo-structures whose appearance is associated with the increasing probe load on the surface.

The rather high reproducibility of the images recorded at the minimum load proves that in this case the AFM probe only slightly affects the integrity and location of the objects on the mica surface. On the contrary, the AFM method becomes destructive with increasing load. An example of the scan depicted in figure 3(b), which we call ‘manipulating scan’, shows that even one-time scanning of the surface caused a noticeable resizing of some MNPs and appearance of new objects. Actually, the comparison of figures 3(a) and (c) reveals that several new objects appeared along the line, whose y-coordinate corresponds to the last line of the manipulating scan. The positions of these objects in figure 3(c) match well the positions of the ends of the nanotracks in figure 3(b) (see labels 2′, 3′, 4′ in figures 3(b), (c)). MNP #2 in figure 3(b) is the starting point of the nanotrack with the height of ∼0.17 nm (figure 3(d)), which extends to point 2′ located on the last line of the raster. After completion of the manipulating scan, a new point-like object 2′ with the height of ∼0.35 nm is revealed in this position (figure 3(c)), and comparison of figures 3(a) and (c) shows that the height of MNP #2 decreased from ∼2.7 nm to ∼0.7 nm. These facts point out that some part was splitted off the MNP during the manipulating scan and then was moved aside. Thus, we can conclude that the nanotrack is actually not a real object, but reflects the process of moving (trajectory) of some object on the surface of the substrate. Let us consider in more detail the mechanism of splitting and movement of the deposited 2D-NPs with the AFM probe.

We suggest that aggregates of weakly bound 2D-NPs are primarily subjected to splitting, because the destruction of monolithic particles requires much more efforts and seems unlikely. Indeed, after manipulation with the AFM probe, the height of monolayer or few-layer NPs did not change, but, with a sufficiently strong load, they could be entirely detached from their places. Earlier, we carried out structural AFM studies of the intermediate materials used to prepare the NPs of graphene and WS₂ (see [32] for details). These materials were characterized by a high content of “large” NPs with a height of several nanometers or more. On the AFM images

![Figure 1](image1.png)

**Figure 1.** AFM images of graphene, MoS₂, WS₂ nanoparticles deposited from alcohol or DMF. The distance values near the arrows indicate the height of the objects. Image sizes are 10 × 10 μm² (a), 3.3 × 3.3 μm² (b), 1.5 × 1.5 μm² (c).
of such NPs obtained at loads similar to the manipulating scan, nanotacks were absent. It appears that in this case we deal with sufficiently strong NPs whose integrity is provided by the Van der Waals bonds preserved at the intermediate synthesis stage. In most our experiments, however, MNPs are not monolithic particles, but rather aggregates of particles with irregular size and orientation of its constituent ‘2D-nanograins’ (a sandwich-like structure). Then, if the atomic planes of neighboring nanograins of the aggregate are azimuthally disoriented relative to each other, such MNP could be quite easily split by mutual displacement of these nanograins along the contact plane. It is known that the azimuthal disorientation of atomic planes leads to a significant decrease in the friction force between them. This effect, called ‘superlubricity’, was described for graphite planes in [2, 7, 8].

To consider the scanning-induced separation process of a sandwich-type aggregate, we use a simplified model in which the aggregate consists of two circular particles and the tip of the AFM probe has the shape of a hemisphere (figure 4). When the AFM probe moves along the line, its tip touches the upper particle of the aggregate and can displace it relative to the lower one. If the distance between the lines of the raster is several times smaller than the diameter of the particle, then the first touch always occurs at the point of the raster whose

Figure 2. AFM images of graphene, MoS₂, BN, WS₂ nanoparticles deposited from DMF or ethanol. Below the images are presented their Z-profiles along the dashed lines marked on the images. The distance values near the arrows indicate the height of the objects. The meaning of symbols A–E is disclosed in the main text. Image sizes are 5 × 5 μm² ((a), (c), (d), (e)), 10 × 10 μm² ((b), (f)).
\( y \)-coordinate is lower than that of the center of the particle. Therefore, the component of the force \( F_y \) acting on the aggregate along the \( y \)-axis is directed upwards of the raster. It should displace the overlying NP in the slow scanning direction (SSD), i.e. towards the upper boundary of the raster. The action will repeat on the next line of the raster. Thus, during the scanning, the aggregate undergoes a series of lateral pushes, leading to the sliding of its upper grain relative to the lower one, until they are completely separated. For granulated aggregates, it is rather difficult to predict the position of the grain boundaries along which their exfoliation will preferably occur. This is influenced by the frictional properties of the interfaces, which, in turn, depend on the relative azimuthal orientation of the grains, but also by the positions of the touch points of the probe with the aggregate on each of the raster lines, determined by geometry of the aggregate and the tip.

After detachment of an NP (or a group of NPs) from the aggregate, it begins to move along the substrate under the effect of successive lateral pushes. The interaction between the probe and the NP while pushing generates a short-term jump in the AFM topographic signal, which leads to appearance of one or more high brightness pixels on the image line. This integrally looks out as a solid nanotrack.

There are several works related to the study of lateral pushing of nanoobjects using an AFM probe. In [11, 33–36], particle displacement due to lateral pushing was used to determine the friction forces at the nanoscale. Other papers [17, 21–26] were devoted to controlled displacement of nanoobjects on the surface (spherical Au...
particles were used with diameters of 5–70 nm). In these works, as in our case, the displacement process was accompanied by the appearance of nanotacks in AFM images, which made it possible to analyze the influence of various factors on the particle trajectory. We will try to compare these results with some nanotracks observed in our experiments. However, it should be remembered that we dealt with nanoparticles whose thickness was 1–2 orders of magnitude smaller than in the mentioned studies, and the contact was the interface of two different atomic planes—mica and nanoparticles deposited on it.

Let us consider the sequence of motions of the probe along the directions of fast and slow scanning (x and y) during performing a raster. As shown in the inset of figure 4(a), the interline probe movement along the y-axis is carried out before forward x-axis scanning, and the probe almost repeats its forward scanning path during backward x-axis scanning (the actual line may slightly differ from the programmed line due to creep of the AFM piezoceramic scanner).

The probe forces the NP out of the scan line during the forward scanning (figure 4(b)), which weakens or completely prevents interaction between the particle and tip during the backward scanning. This was also found in experiments with Au nanospheres [23], in which motion paths observed in the forward topography AFM image were absent in the backward one. In our experiments, the forward x-axis scanning direction corresponded to the probe moving from left to right, which explains the deviation of the majority of the nanotacks in figures 2 and 3(b) from the SSD to the right.

Almost straight-line shape of the motion paths of most NPs points out that the frictional characteristics of the NP–substrate interface did not undergo significant changes during the movement [23]. Under these conditions, most NPs freely passed over the scanning area and were ultimately located along the last line of the manipulating scan (e.g. 2', 3', 4' in figure 3(c)). However, rather rare, the nanotacks ended in the middle of a raster (e.g. labels B in figures 2(e), (f) and nanotrack 1→1' in figure 3(b)). That could be caused by a change in the sliding conditions, for example, when there is an island of impurities with a high adhesive ability or another stopping defect on the substrate surface. When an NP is fixed above such a stopper, its movement will be interrupted. Then the tip will leap over the particle in subsequent lines of the raster and the nanotrack will not be continued. The rectilinear motion of an NP during surface scanning can also be disturbed when some trajectories intersect. This means that displacing NPs meet in the intersection point. In such cases, one of the particles blocks the movement of the others (see nanotacks 3a→c→3' in figure 3(b)), or several NPs can be combined into a new aggregate which will continue to move as a whole (see nanotrack 4→4' in figure 3(b)).

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We expected that the scanning parameters would affect the direction of motion of the particles. Actually, Rao et al [22] found that the angle of the particle’s trajectory relative to the SSD can be precisely adjusted by changing the distance between the lines of the raster. However, the direction of the nanotacks remained unchanged in our experiments. Moreover, in some cases, it did not change even after reversion of the forward x-axis scanning direction (i.e., from right to left), although the momentum received by the particle from the tip should then shift it to the left. This suggests the anisotropy of the friction within the 2D-NP—substrate pair and existence of the directions of ‘easy’ sliding determined by the relief of the substrate. Thus, the energy obtained upon the impact...
with the tip causes a displacement of the particle in one of the preferred directions in which its detachment and movement occur more likely. This can explain why some nanotracks in figures 2 and 3 deviate to the left (e.g. label C in figure 2(f) and nanotracks 1→1′, 3c→3′ in figure 3(b)). When the movement of the 2D-NP in the right-up direction is obstructed, effective interaction with the tip can occur during backward x-axis scanning, leading to the pushing of the particle in the left-up direction. In addition, if the azimuthal orientation of the particle changes during one of the impacts with the tip, this can sharply change the nanotrack, switching it to another direction of ‘easy’ sliding (see labels D in figures 2(c), (f) and nanotracks 1→1′, 3c→3′ in figure 3(b)). The origins of the ‘easy’ sliding directions may be associated with the anisotropy of atomic structure of the mica surface [38] as well as with possible presence of ripples on it. Specificity of a favourable structure matching at the friction contact between the 2D-NP and mica surface can also be essential in this case.

The shape and size of the nanotracks make it possible to distinguish them from real linear nanoobjects, such as nanotubes, nanoribbons, or NPs assembled in a row [39]. Once again, it should be stressed that the nanotracks completely disappear in the image at a rather weak load of the probe on the surface.

For consideration of the factors that can affect the emergence of NPs aggregates, we suggest that aggregates are formed as a result of stacking of 2D-NPs directly in the dispersion or when it dries after precipitation on the substrate. The intensity of aggregation can be influenced by various factors, e.g., synthesis conditions and storage duration of the dispersion, presence of impurities, etc. Additional factors that affect the processes of aggregates’ self-assembly are the substrate hydrophobicity, mobility of the particles, the rate and uniformity of the solvent evaporation. Their combinations can lead to the growth of aggregates of a highly complex form. One such example, a fractal superstructure of MoS2 2D-NPs, is shown in figure 5.

Taking into account the tendency of 2D-NPs to self-assembly, it is important to be able to distinguish a monolithic particle from a sandwich-type aggregate of 2D-NPs. Based on the obtained data, we propose a route for solving this task using AFM. It includes three steps:

1) Adjustment of an optimal load at which there are no displacements of the NPs during scanning. The criteria for this are the absence of the nanotracks in AFM images as well as stable particle positions in the first and subsequent scans. One of the possible ways to achieve this goal was described above: increase the SP up to the maximum possible value at which the device still retains its sensitivity to the surface topography. Of course, this will impair the image resolution and may result in loss of sensitivity during scanning. Besides, it is not always possible to achieve a complete absence of the nanotracks by increasing the SP. In such cases, to further reduce the load, the free oscillations amplitude of the cantilever should be reduced or a probe with a lower cantilever stiffness should be used.

2) Carrying out a series of scans over a new surface area with a gradual decrease of the SP until the nanotracks appear. If the brightness of the starting point is higher than the average brightness of the nanotrack, this means that the aggregate of NPs has been splitted up (e.g. labels A in figure 2). At the same load, the monolithic NPs usually remain at their initial places (this matter will be discussed further). However, a
weakly adhered monolithic NP can also be displaced by the probe from the place where it was after deposition. In this case, the brightness of the starting point will not significantly differ from the average brightness of the nanotrack (e.g. label E in figure 2(d)).

3) If a monolayer NP remains at the initial place of the separated aggregate, then it may be undistinguishable against the background of the nanotrack. Therefore, after obtaining the image with the nanotacks, it is desirable to restore the optimal load and make a comparative scan.

A question may arise why only the upper NP (or a group of NPs) of the aggregate is displaced, and not the entire aggregate. In our opinion, it occurs because the friction forces between the atomic planes of the NPs are weaker than between the lower aggregate plane and the substrate surface. Actually, structural superlubricity was observed between graphite flakes assembled under ambient conditions [10]. However, another question is: why do some NPs (or groups of NPs), splitted off the aggregate, move along the surface under the influence of a probe, while most other NPs under the same load remain at their initial places? We assume that not only NPs, but also some impurities that could produce highly adhesive areas (stoppers) on the surface are deposited from the dispersions. During evaporation of the solvent, NPs and their aggregates present in the liquid medium could migrate over the surface until they are fixed above one of these areas. It can not be excluded that the residual solvent (wetting) layer between the substrate and the particle can also act as a stopper (a glue). That is why the adhesion forces of NPs deposited directly from the dispersion can appear higher than those of NPs that were splitted off the aggregates by the probe on the dry substrate. Note that the amplitude modulation tapping mode of AFM makes it possible to determine the energy loss of the probe during manipulations by measuring the phase shift of the cantilever oscillations relative to the exciting periodic signal [17, 33]. Such experiments not carried out in this work and may be the subject of further research.

The similarity of the observed phenomena for various materials (graphene, MoS2, BN, WS2) allows us to conclude that the mechanisms of self-assembly, surface fixation, and sliding of graphene-like NPs are similar. The common factors in our experiments are the relatively small lateral size (<50 nm) and the graphene-like structure of the used NPs, which, in our opinion, exerts the decisive effect on their mobility and self-assembly. The self-assembled NP aggregates can be used as sources of particles of atomic thickness weakly bonded to a substrate under ambient conditions, and an AFM probe serves as a tool to split up and move them. In particular, the technique of lateral pushing with an AFM probe can be conveniently applied for precise application of lubricant or for targeted delivery of NPs to predetermined surface areas, since it allows one to visualise the particle’s trajectory in real time and control its direction by adjusting the scanning parameters.

We assume that the mechanism of the emergence of nanotacks suggested here could also occur in other microscopic methods, where the image is formed in the process of scanning surfaces with various microprobes. A microprobe acting on a surface can be, e.g., a focused electron beam in SEM or an exciting laser in a Raman microscope.

**Conclusions**

Using the mica substrate as an example, we demonstrated a new route for producing 2D-nanoparticles weakly bonded to a substrate under ambient conditions, which is based on the mechanical separation of the deposited self-assembled nano-aggregates that are formed in dispersions. The AFM technique was applied to visualize nanoparticles, to rearrange them using the AFM probe as a mechanical tool and to controllably move them on the substrate. Previously, similar manipulations were described by other authors for spherical particles having diameters of tens of nanometers. In contrast, we demonstrated the possibility of creating and moving, by lateral pushing, several new objects of subnanometer thickness on mica surface: flakes of graphene, MoS2, WS2 and BN. Their motion was observed by the appearance of clearly distinguishable nanotacks. It is shown that the nanotrack is an artifact that displays the particle trajectory under the influence of a series of lateral pushes from the tip of the AFM probe during scanning. In order to avoid unintentional displacement of 2D-NPs during AFM measurements and misinterpretation of their results, special attention should be paid to minimize the interaction force between the probe and the surface. A technique was proposed that allows one to distinguish a thick monolithic particle from the nano-aggregate by analyzing the nanotrack image. These results confirm that the AFM method is an efficient tool for both splitting nano-aggregates and the targeted delivery of the NPs to predetermined surface areas.
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Data availability statement

Any data that support the findings of this study are included within the article.

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