Deformation and wear of pyramidal, silicon-nitride AFM tips scanning micrometre-size features in contact mode

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Abstract

An experimental study was carried out, in order to investigate the deformation and wear taking place on pyramidal silicon-nitride AFM tips. The study focuses on the contact mode scanning of silicon features of micrometre-size. First the deformation and the mechanisms of wear of the tip during scanning are discussed. After that the results of an experiment showing both phenomena on a used AFM tip are presented. Both the damaged and the unused tip are shown on AFM and SEM images. Using these images the actual mechanisms of wear are determined. It is shown that adhesive wear, low cycle fatigue and plastic deformation take place on the tip. © 1999 Elsevier Science Ltd. All rights reserved.

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1. Introduction

The atomic force microscope (AFM) was introduced by Binning et al. [1] in 1986. With an AFM high-resolution topographic images of both insulators and conductors can be made. In this type of microscopy the sample to be imaged is brought close to a sensing tip attached to a small cantilever. Interaction forces between sample and tip deflect the cantilever. In the most commonly used measuring mode, the tip–sample distance is kept constant by a feedback loop during the measurements. This loop gives the information to create an image of the surface of the sample.

Those images often carry ambiguity. While imaging features of the order of magnitude of several hundreds of nanometres, the tip geometry has significant influence on the results. Methods for the ‘de-convolution’ of the tip geometry from the images are discussed in many papers [2–4]. For these ‘deconvolution’ methods it is necessary to know the tip geometry. To make the correct changes to the images even the changes of the tip geometry during scanning should be known.

Usually, little explicit attention is paid to the problem of plastic deformation and wear of the tip during scanning. Some authors [5–8] have shown deformation and wear of the AFM tip. They investigated and discussed the deformation and wear for imaging and scratching flat surfaces. This paper is dedicated to deformation and wear taking place while scanning μm-size features. Samples with these features are often used to calibrate an AFM. The scanning is carried out in the contact-mode. First it is calculated whether plastic deformation of the tip can
be expected. Then the mechanisms of wear, which can take place on the tip or the sample during scanning, are discussed. After that the results of experiments showing the effects of wear and deformation on a tip are presented. Finally some conclusions concerning the mechanisms of wear, which take place during scanning, are pointed out.

2. Plastic deformation

Depending on the magnitude of the interaction forces, plastic deformation of the AFM tip can take place. Plastic deformation will occur when the maximum contact stress at the interface is larger than the critical stress of the interacting materials. To estimate whether plastic deformation can be a problem we will compare the critical stress to the maximum contact stress.

The maximum contact stress at the interface can be estimated by [8]:

$$\sigma_{\text{max}} = \left( \frac{6F_c E_t^2}{\pi^2 R^2} \right)^{1/3}$$

where $F_c$ represents the contact force, $R$ the radius of the tip and $E_t$ the combined elastic modulus of the tip and sample material. During the experiments the contact force is in the order of 100 nN. The tips used have a radius of ~100 nm. $E_t$ is given by:

$$E_t = \left[ \frac{(1 - \nu_a^2)}{E_a} + \frac{(1 - \nu_b^2)}{E_b} \right]^{-1}$$

in which $\nu_a$ and $E_a$ represent the Poisson constant and Young’s modulus of the sample material, silicon, and $\nu_b$ and $E_b$ represent the Poisson constant and Young’s modulus of the tip material, silicon-nitride. For this estimation we use the following values:

$\nu_a = 0.33$
$E_a = 110 \times 10^9 \text{ N/m}^2$
$\nu_b = 0.27$
$E_b = 310 \times 10^9 \text{ N/m}^2$
$F_c = 100 \text{ nN}$
$R = 100 \text{ nm}$

The Poisson constants and Young’s modulus’ are adapted from the CRC Materials Science and Engineering Handbook [9]. The force is estimated from force curves and the radius is given by the manufacturer of the tips. This results in a $\sigma_{\text{max}}$ of 2.5 GPa.

The critical stress for plastic deformation can be estimated by [10]:

$$\sigma_{\text{max},c} = 0.6 \cdot H$$

where $H$ represents the hardness (expressed in GPa; 1 GPa roughly corresponds to 100 Vickers hardness units). The hardness values of silicon and silicon-nitride are 10 GPa and 20 GPa, respectively. These are macroscopic hardness values. Czernuszka [11] finds that often the near-surface hardness of ceramics is lower than the macroscopic hardness. This means that the given hardness values should be seen as an upper bound. The critical stress for silicon is about 6 GPa and for silicon-nitride is about 12 GPa. The maximum and critical stresses are of the same order of magnitude. Therefore it seems that deformation flattening the tip can take place.

3. Mechanisms of wear

When a surface is scanned by an AFM-tip, there are several mechanisms of wear, which can be due to a damaged surface of the tip afterwards. Possible mechanisms are [12–15]:

- adhesive wear
- abrasive wear
- low cycle fatigue
- tribochemical wear.

In the next subsections these mechanisms will be explained briefly.

3.1. Adhesive wear

The basic view of adhesion is that when two surfaces come into contact they adhere one another, at least at localised sites. As the two surfaces move relative to one another, wear occurs by one surface pulling the material out of the other surface at these sites. This is shown schematically in Fig. 1.

From a macroscopic point of view the apparent contact area will not be the real contact area because
of the roughness of the two surfaces. Because of the small area involved in the AFM tip-surface contact, the surfaces are assumed to be smooth. This implies that the real contact area equals the apparent contact area. In the sketch of Fig. 1 the situation at the end of the contact between the tip and the sample is shown. As the tip and the sample move relative to one another, rupture of material will eventually occur along a certain path. If the rupture occurs along path 1, which is the original interface, no material will be lost from either surface, though some plastic deformation may occur. If on the other hand, the rupture occurs along some other path, for example path 2, the sample can lose material.

It is known that in ambient conditions there is a water layer on the sample. When bringing the tip into contact with the sample this layer is penetrated. There will not be any water left between the tip and the sample. Therefore the contact is considered as a dry contact. The volume of material removal by adhesive-wear can then be estimated from the expression proposed by Archard [16]:

\[ V_a = k \frac{W}{H} L \]

where \( V_a \) represents the volume of the removed material, \( k \) the wear coefficient, \( W \) the friction force, \( H \) the hardness of the softer material and \( L \) the sliding distance.

For the tip-sample contact of the AFM, \( k \) is estimated at 0.5, \( W \) will be in the order of 100 nN, \( H \) of the softer material, silicon, is 10 GPa and \( L \) is calculated from the scan rate of 20 \( \mu \)m/s. During the experiment we are scanning for an hour, so the sliding distance is \( 7.2 \times 10^{-2} \) m. These values give a wear rate of \( 3.5 \times 10^{-19} \) m\(^3\)/h. Taking into account that the volume of the tip involved in measuring \( \mu \)m-size features is in the order of magnitude of \( 10^{-19} \) m\(^3\) also, it can be concluded that this wear rate is rather large. This type of wear will be notified by sample material added to the tip.

3.2. Abrasive wear

A abrasive wear can take place in two different ways: two-body or three-body abrasive wear.

Two-body abrasive wear means that a hard protuberance on a surface produces a groove, scratch or indention on the opposite surface. In the case of the AFM, the tip is usually the hardest material, so this wear could only take place with a protuberance of the tip in the sample. A protuberance on the sample would just deform. This type of wear will therefore not affect the tip. Three-body abrasive wear means that a hard particle trapped between the two bodies produce a groove, scratch or indentation in either of the surfaces of the bodies. This type of wear could take place if a particle becomes trapped between the tip and the sample. This is very unlikely because we are dealing with a very small and changing contact...
area. If this is taking place, it results in a groove or scratch on the surface of the tip.

3.3. Low cycle fatigue

Low cycle fatigue process may result in defect accumulation in one of the contacting surfaces with cracking to follow. This will produce a rough surface. A number of steps leading to the generation of wear particles can be identified. They are:

- transmission of stresses at contact points
- growth of plastic deformation per cycle
- subsurface void and crack nucleation
- crack formation and propagation
- creation of wear particles.

A sketch is shown in Fig. 2 representing the first, third and last steps. Essential for this type of wear is that a certain load has to be put several times to the same surface. This mechanism will therefore only take place on the tip. The tip we use is made of Si$_3$N$_4$ which is a ceramic. In the literature, several authors described the phenomenon of low cycle fatigue taking place on ceramics [17–19]. They all describe fatigue taking place on a much larger scale than the AFM-scale. Prudence is called for using these results to interpret the AFM results. For example the strain to failure in one loading cycle is a factor of influence on the effects of the fatigue. On the macroscopic scale this strain has a certain value. On the microscopic scale strain will have a certain distribution over the surface, this distribution will have impact on the failure taking place. The existence of this type of wear on the tip of the AFM will be shown by a damaged, rough surface of that tip.

3.4. Tribochemical wear

Tribochemical wear means that soft hydroxides are formed on the sliding surface by the reactions of the surfaces with for example water. This will form a small lubricant film. The creation of this layer will smooth the surface and can introduce hydrodynamic lubrication. This type of wear removes material molecule by molecule instead of the classical removal of the entire particles. This wear mechanism therefore results in an extremely smooth surface. In the AFM situation the silicon sample will get a layer of SiO$_2$ and SiO$_2^{2-}$ on it, because of a reaction with water. This layer will have a thickness of a few nanometres. The tip material, silicon nitride, is almost resistant for water, though it is possible that the sliding and heat production during sliding will cause some reaction of the silicon nitride. This will be shown by a smoothened surface of the tip.

In order to determine which of the mechanisms occur experiments were carried out.

4. Description of equipment and experiments

Before describing the experiment, first the equipment will be pointed out. The AFM used is a Topometrix 2000 TMX (manufactured in 1995). This AFM is placed on a vibration-isolated table mounted with four springs in a frame. The measurements are carried out with pyramidal silicon nitride tips, also of Topometrix (model 1520-00). These tips have a top radius of about 100 nm and a top angle of 90°. The samples measured are from the UltraSharp Silicon grating set TGS02 of the NT-MDT, Moleculair Devices and Tools for NanoTechnology.

The executed experiment consists of three steps. First an image of the tip shape was made, then the

![Fig. 2. Illustration of low cycle fatigue.](image-url)
Fig. 3. Sketch of the dirac-like features on the used sample with the nominal geometry indicated.

Fig. 4. Cross section of an image made on the sample with the dirac-like features.

Fig. 5. Sketch of the trenches on the used sample with the nominal geometry indicated.

Fig. 6. AFM-image of tip on dirac-like feature before the wear experiment.

The tip was subjected to wear and at the end again an image of the tip is made. These steps will be explained in more detail.

1. First an AFM image showing the shape of the tip was obtained. This was done using a sample containing dirac-like features, as sketched in Fig. 3. This sample is one of the samples of the TGS02-set described above. Its geometry was confirmed using SEM. The top radii of the dirac-like features are less than 10 nm, according to the manufacturer. This means that the image will show the tip instead of the sample. This is clearly seen in the sketch of Fig. 4. The only parts of the sample which come into contact with the tip are the tops of the dirac-like features. The dashed line in Fig. 4 shows what the cross section of an image will look like. The height of the features, 0.7 \( \mu \)m, limits the part of the tip to be investigated.

2. During the next step the tip was subjected to wear. A sample was imaged continuously for about an hour. This sample contains square shaped trenches with a height of 512 nm, as shown in Fig. 5. The scanned area was (20×20) \( \mu \)m\(^2\), the scan rate was 20 \( \mu \)m/s. The normal force was about 100 nN.

3. Finally again an AFM image showing the shape of the tip was obtained on the first imaged sample, as described under step 1.

5. Results and discussion

The experiment as described in Section 4 is executed four times, using four tips and different areas on the sample. The results did not show significant differences. The results shown are all of the same experiment.

Representative images of the shape of a tip made before and after the tip was subjected to wear are shown in Figs. 6 and 7.
Fig. 7. AFM-image of tip on dirac-like feature after the wear experiment.

When comparing these two images two differences are noticed. Firstly the top of the tip is flattened and secondly a part of the pyramid appears to be removed. This is confirmed by a vertical cross section of both Figs. 6 and 7 which is presented in Fig. 8. In this figure both effects (tip flattening and material removal) are indicated. In order to get a different view the damaged tip is investigated under an SEM. Before depicting the tip in the SEM a layer of a few nanometres of gold is applied to the tip. The resulting SEM image is shown in Fig. 9. For comparison, an SEM image of an unused tip is shown in Fig. 10.

The first peculiarity to notice on the SEM image of the damaged tip is the extra material. This is not shown in the AFM images, because it is taking place at the end of the part of the tip involved in the experiment. This appears to be adhesive wear, where material of the sample is moved towards the tip. From the SEM image we estimate that the extra material added to the tip is in the order of \(10^{-10}\) m. (This crude estimation is done by considering the added material at the right side of the tip to be a rod of 100 nm by 100 nm with a length of 1 \(\mu\)m.) This is 10 times less than the volume calculated at the theoretical explanation about adhesive wear, Section 3.1. On one hand, the sliding distance we used in the Archard relation in Section 3.1 can cause a part of this difference, on the other hand the uncertainty in
estimation of a volume out of 2-D SEM data cannot be much better than one order of magnitude. In the way we calculated this length we assume that the end of the tip is always in contact with the sample. In reality when the tip moves down in the trench the contact point is moving on the tip. This is also happening while moving out of the trench. This means that the sliding distance is actually smaller.
and therefore that the adhesion volume is smaller. The difference in volume can also be due to the fact that we used the model for dry contact for the calculation in Section 3.1. In reality we do not have a complete dry contact, but there will be some water left in the contact area which counteracts the adhesion.

Besides this it is observed that the top of the tip is flattened. This is not surprising, recalling that the maximum contact stress is in the same order of magnitude as the critical stress of the tip.

In the AFM image it can be observed that from one side of the pyramid material is removed. In the SEM image this part of the tip is showing many irregularities. This can be explained by the presence of low cycle fatigue during scanning. The damaged part of the tip, is the part of the tip that comes at first into contact with the sample. Every time that a feature is approached a load of about 100 nN is exerted to that part of the tip.

From the AFM images, the volume loss $\Delta V$ of the tip can be estimated by subtracting the volumes of the tip before and after the wear experiment. This volume appears to be $3 \times 10^{-20} \text{ m}^3$.

6. Conclusions

The impact of wear and deformation on the tip of an AFM is investigated for a scan rate of 20 $\mu\text{m}/\text{s}$. The conclusions of our investigations will be valid for scanning $\mu\text{m}$-size structures at a scan rate of the order of some tens of micrometres per second. According to the results of the executed experiment it may be concluded that at least three wear and deformation phenomena take place on the tip during scanning.

- Adhesive wear at the part of the tip that gets in contact with the features of the sample. This is shown by the added material to the tip.
- Plastic deformation of the top of the tip, because the contact pressure approaches the critical stress. The resulting flattening of the top of the tip is shown.
- Low cycle fatigue of the part of the tip which comes first into contact with the features of the sample. This is shown by the irregular surface of the tip at that part of the tip.

Abrasive wear is not taking place on the tip, which was already expected because silicon is much softer than silicon nitride. About the existence of tribochemical wear a conclusion cannot be drawn. From the resulting images it cannot be seen whether the surface has become smoother after the experiment or not.

According to these results it may be concluded that the influence of wear during image with a scan rate of the order of some tens of micrometres per second is significant. Therefore it is necessary that after scanning for about 1 h, with the same tip, either the wear of the tip should definitely be taken into account while analysing the images or the tip should be replaced.

For a lower scan rate the wear of the tip will change in two different ways. On one side it could happen that the adhesive wear will increase because the contact with the features of the sample will appear for a longer time. On the other hand the wear caused by the low cycle fatigue will decrease.

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References


