

Curvature radius analysis for scanning probe microscopy

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Abstract

SFM images are the result of interactions between a sharp tip with a quasi-spherical apex and the surface of a sample. As a consequence, SFM images are highly influenced by the tip geometry especially when the surface features are sharper than the probe. For topographic images, this phenomenon is known as dilation. It can result in an image where the features reflect the tip apex characteristics rather than the sample's surface. Mathematical algorithms are used to show the possibility of computing a curvature image from a topographic image to deduce the radius of the tip. In addition, the interpretation of a surface image obtained with modes other than topography can be achieved by comparing this image to a curvature image. As an example, the Tapping Mode AFM phase image contrast can show similarities or discrepancies with the curvature image contrast, leading to a direct relation between the phase and a topographic or a physical contrast.

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1. Introduction

The development of scanning probe microscopy (SPM) in the 1980s and 1990s has opened new pos-

sibilities for surface investigations concerning the nanometer scale. If the scanning tunneling microscopy (STM) [1] is restricted to conductive surfaces, the scanning force microscopy (SFM) [2] especially with the development of tapping mode AFM [3] and others dynamic modes [4], enables the observation of nearly any kind of surfaces within various environments (vacuum, air, fluids).

One of the constraints with the SPM techniques is the quasi-spherical geometry of the tip apex. An

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unworn tip is usually conical or pyramidal with a spherical apex having 5–50 nm curvature radii [5]. The ideal tip must have both a curvature radius as small as possible and a half angle as low as possible. Very sharp tips such as oxide-sharpened silicon nitride tips, focused ion beam machined silicon tips, etched silicon tips [5–8], and carbon nanotube tips [9,10] have been proposed.

When the tip–surface interaction is limited to a single atom, atomic resolution can be obtained [11]. Nevertheless, in most cases, the tip is not sharp enough to describe the surface features correctly. At this scale, the AFM image is the result of the interactions between the tip and the sample surface, more particularly when the sample slopes are higher than those of the probe. This phenomenon, known as dilation, tends to smooth the surface and equalizes the irregularities [12–15]. In extreme situations, the tip geometry clearly appears on the image [16–18]. This phenomenon known as ‘tip self imaging’ can involve so called double tip artifacts.

Mathematical morphology tools such as erosion can partially correct this ‘tip self imaging’ if the tip geometry is known [12–15]. Several methods were developed to evaluate the tip geometry. The first method consists of imaging a reference surface for which the geometry is perfectly known. If the surface features are sharper than the tip geometry, an image of the tip will be obtained [19–21]. The tip geometry can also be characterized using ‘blind reconstruction’ methods [15,22–25]. The original method developed simultaneously by Williams and Villarubia produces a reconstructed tip, which is the most voluminous geometry that may have imaged the surface [22,23]. Nevertheless, this method fails when the image is noisy [15]. Several solutions were proposed to solve this problem but they are particularly time consuming and can lead to aberrant results (truncated tip) for which it is impossible to compute a radius [15,24–26]. If the geometry of the tip is correctly estimated, the AFM image can be partially reconstructed, producing an image that is closer to reality than the primary one [12,15,22,27]. This is particularly interesting in the field of metrology or for image interpretation.

Furthermore, a significant number of SFM imaging modes were developed to image surface properties [28] by measuring the interaction between the tip and the surface. Different properties such as magnetic, electrostatic, friction and adhesion forces, elasticity, electrical or thermal conductivity can be mapped. For these kinds of imaging, one can quantify the tip–sample interaction but it is much more difficult or even impossible to quantify the surface properties, partially because the tip and the surface interaction geometry are unknown in detail. Additional information is needed. One aim of this paper is to show how some information about the tip geometry can be obtained.

Tapping Mode AFM [3] is one of the most popular SFM imaging modes. In this operating mode, the cantilever is made to oscillate in the neighborhood of the sample surface at a frequency close to its resonant frequency (typically 50–300 kHz) with constant amplitude of a few tens of nanometers. The tip sample interaction causes a decrease of the vibration amplitude as compare to the free amplitude. Usually Tapping Mode AFM is also less damaging for the surface and gives a better quality imaging than the contact mode. This is mainly due to the reduction of the capillary force, shear force, and eventually contact pressure and so visco elastoplastic strain.

Phase contrast imaging could be associated to Tapping Mode AFM. The phase signal is related to the time shift between the oscillation of the cantilever and the oscillation of the driver. It is said that phase imaging can be used to map the change of sample properties. Tapping Mode AFM and phase contrast are difficult to model due to the high non-linearity of both attractive and repulsive forces applied on the tip when the tip oscillates on the vicinity of the surface.

Many numerical or analytical models [29–43] were developed to understand the behavior of the cantilever. These models solve the equation of the cantilever displacement when the tip interacts with highly non-linear gradient forces. In a complementary ways, analytical simulation and energy approach shows the possibility to connect phase signal and energy dissipation [29,44–46]:

$$\sin \varphi = -\frac{A}{A_0} \frac{\omega}{\omega_0} (1 + \eta) \quad (1)$$

where A is the working amplitude, A_0 the free amplitude, ω the working frequency, ω_0 the free resonant frequency and η the ratio between the additional oscillator damping coefficient due to the tip/sample interaction and the oscillator damping coefficient without tip/sample interaction.

In a SFM experiments, the amplitude and the frequency are maintained constant, the phase signal is then mostly due to a variation of energy dissipation. The variation of energy dissipation could be due to a change of the sample properties (i.e. adhesion, viscosity or plasticity) or to a change of the tip/sample interaction area due to a change of topography sample visco-elasticity or surface curvature [47,41]. Experimental results confirmed by models, shows that the tilt angles [48], topography [49] or volume properties [50] could be responsible of an energy dissipation and then of a phase shift.

In this paper, a technique to compute the curvature of the image from topographic images is proposed. This treatment can be used for several applications:

- (1) The first application is the measurement of the tip radius. This measurement is limited to images where the dilation phenomenon is present. In this case, the image features are an inverted image of the tip and the measurement of the features curvature gives directly the radius of the tip.
- (2) The second application is the measurement of the radii of curved surface features such as spheres or cylinders. This measurement is limited to data without the dilation phenomenon. Quantification of the radius curvature of nanometer-sized spheres will be shown.
- (3) The last application is the interpretation of non-topographic images. The interaction between the tip and the surface is highly dependent on the surface curvature. Comparisons between curvature images and the corresponding Tapping Mode AFM phase images clearly show whether or not the phase contrast is related to a curvature contrast.

2. Surface curvature

Mathematically, the curvature of a line or a surface can be computed if the second order derivative exists and is continuous [51].

For a line, $z = f(x)$, the curvature ρ , is described by only one parameter: the curvature radius R :

$$\rho = \frac{1}{R} = \frac{z_x'''}{(1 + z_x'^2)^{3/2}} \quad (2)$$

where $z'_x = \frac{\partial z}{\partial x}$ and $z''_x = \frac{\partial^2 z}{\partial x^2}$.

For a surface, the situation is more complex. The curvature needs to be described by two parameters. For one point C of a surface, the normal vector N to the surface is considered. Any plane P which contains the normal vector cuts the surface to form a curved section (Fig. 1). The curvature of the point C in the plane P is described by a curvature radius R , which is equal to:

$$\rho = \frac{1}{R} = \rho_1 \cos^2 \alpha + \rho_2 \sin^2 \alpha = \frac{\cos^2 \alpha}{R_1} + \frac{\sin^2 \alpha}{R_2} \quad (3)$$

R_1 and R_2 are the principal curvature radii. They are defined as the minimum and the maximum values of R . The two planes that contains the curves of radii R_1 and R_2 are perpendicular [52]. α is the angle between the plane P and these

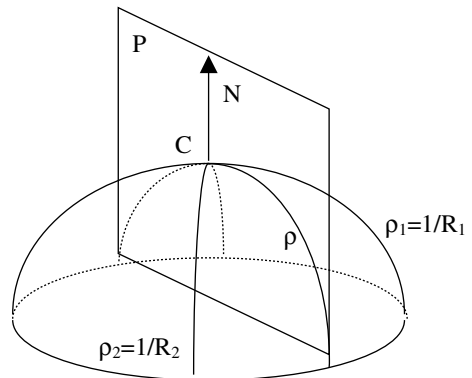


Fig. 1. Curvature of the point C of a surface. We consider any plane which contains the normal vector N to the surface. The intersection between the plane and the surface could be described by a curvature ρ . This curvature ρ is a function of the principal curvature ρ_1 and ρ_2 .

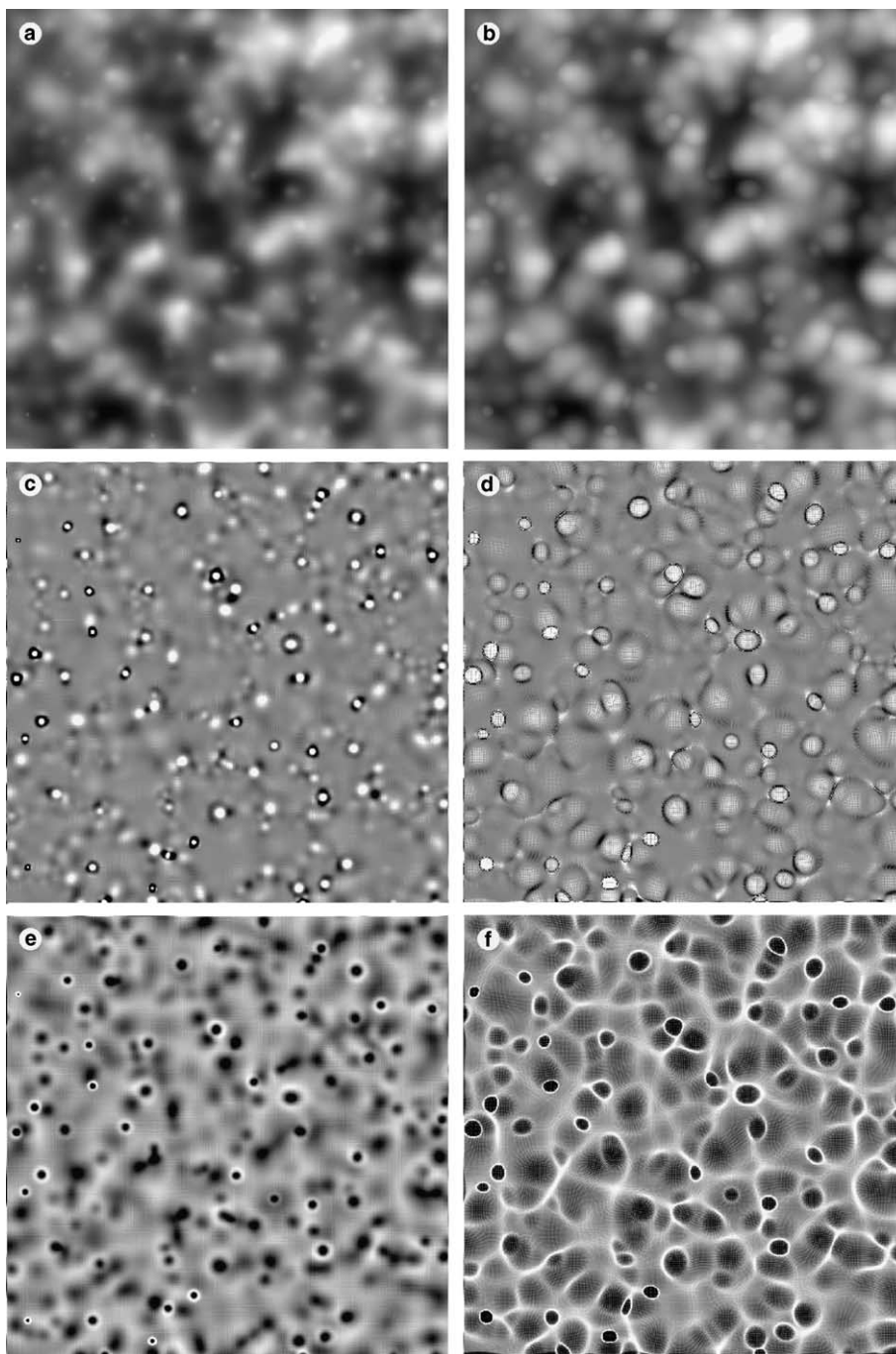


Fig. 2. Signature of the tip during the imaging process. We consider a $1 \mu\text{m}^2$ virtual surface (a), and we present the corresponding SFM image (b), generated by a virtual spherical tip (20 nm in radius). The comparisons between the average (c) and (d) and gaussian curvature (e) and (f) of the two images clearly shows the effect of the dilation on the curvature image. The gray scale are 59 nm, 0.03 nm^{-1} and -0.001 to 0.001 nm^{-2} respectively.

two planes. If the surface is described by the equation $z = f(x, y)$, R_1 and R_2 are the solutions of the 2nd degree equation:

$$(z''_x z''_y - z''_{xy})R^2 + H[2z'_x z'_y z''_{xy} - (1 + z'^2_x)z''_y - (1 + z'^2_y)z''_x]R + H^4 = 0 \quad (4)$$

where $z'_x = \frac{\partial z}{\partial x}$, $z''_x = \frac{\partial^2 z}{\partial x^2}$, $z''_{xy} = \frac{\partial^2 z}{\partial x \partial y}$ and

$$H = \sqrt{1 + z'^2_x + z'^2_y} \quad (5)$$

For any point C, if the two principal radii of curvature have the same sign, the tangent plane to the surface does not cut the surface. The surface is elliptical (or spherical if $R_1 = R_2$). If R_1 and R_2 have different signs, the surface describes a “horse saddle”. The surface is hyperbolic. If R_1 and R_2 are equal to infinity, the surface describes an inflexion. The surface is parabolic.

The curvature is generally described by the average curvature A and the gaussian curvature G :

$$A = \frac{1}{2} \left(\frac{1}{R_1} + \frac{1}{R_2} \right) = \frac{z''_x(1 + z'^2_y) + z''_y(1 + z'^2_x) - 2z'_x z'_y z''_{xy}}{2H^3} \quad (6)$$

$$G = \frac{1}{R_1 R_2} = \frac{z''_x z''_y - z'^2_{xy}}{H^4} \quad (7)$$

These two parameters are useful to describe the shape of the surface at any point. If G is negative, the surface is parabolic. If G is positive, the surface is elliptic: The surface is convex (hill) if A is negative and is concave (valley) if A is positive. It is important to note that the ratio of the average curvature on the gaussian curvature gives directly the average radius of the surface:

$$\frac{A}{G} = \frac{R_2 + R_1}{2} \quad (8)$$

The difference between the average radius and the minimum radius (or the maximum radius) ΔR can as well be computed using the following formula:

$$\Delta R = \sqrt{\frac{A^2}{G^2} - \frac{1}{G}} \quad (9)$$

In the case of topographic images clearly dilated by a quasi-ellipsoidal tip, the pixels for which the surface is convex ($G > 0$ and $A < 0$) can be interpreted as pixels of an image apex (and so to a tip self image). They can give information about the tip radius. For topographic images that are not dilated by the tip, the average and the gaussian curvature can be used to measure the radius of the surface features. For example, the radius of spheres or cylinders adsorbed on a substrate can be computed.

In the case of a non-topographic image, the contrast should be sensitive to the curvature of

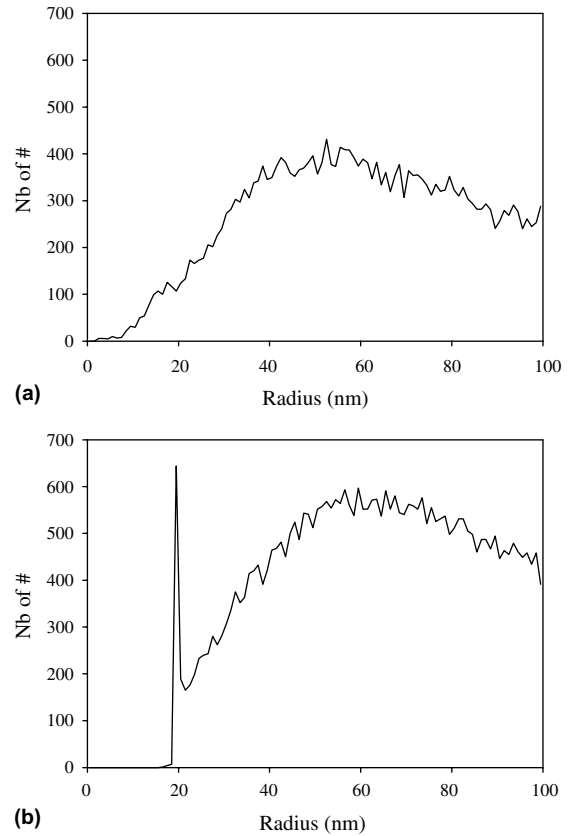


Fig. 3. Histogram of the gaussian radius of the convex pixels of the virtual surface (Fig. 2a) (a) and the corresponding SFM image (Fig. 2b) (b). During the imaging process, the tip is unable to accurately image some parts of the surface. As a consequence, the image reflects the tip geometry rather than the surface. The comparison of the two histograms shows that the surface features which have smaller curvature radii than the tip cannot be mapped accurately. All the features of the surface which have a lower radius than the tip are shifted to the tip radius value.

the tip. For example, if the signal is sensitive to a van der Waals force F_{vdW} or to a capillary force F_{C} force, the signal will depend on the variation of the equivalent radius R^* as shown in the following equations:

$$F_{\text{vdW}} = \frac{HR^*}{6d^2} \quad (10)$$

$$F_{\text{C}} = 4\pi R^* \gamma_{\text{LV}} \quad (11)$$

with

$$\frac{1}{R^*} = \rho^* = \frac{1}{R_{\text{Tip}}} + \frac{1}{R_{\text{Surface}}} = \rho_{\text{Tip}} + \rho_{\text{Surface}} \quad (12)$$

where H is the Hamaker constant, d is the distance between the tip and the surface, γ_{LV} is the surface tension at the liquid air interface.

If we consider that the tip curvature is constant, the variation of the equivalent radius is proportional to the variation of the surface radius:

$$\Delta R^* = \Delta R_{\text{Surface}} \left(\frac{R^*}{R_{\text{Surface}}} \right)^2 \quad (13)$$

If the surface radii are low when compared to the tip radii, the topographic image is highly dilated and does not reflect the sample surface. No relation can be established between the curvature

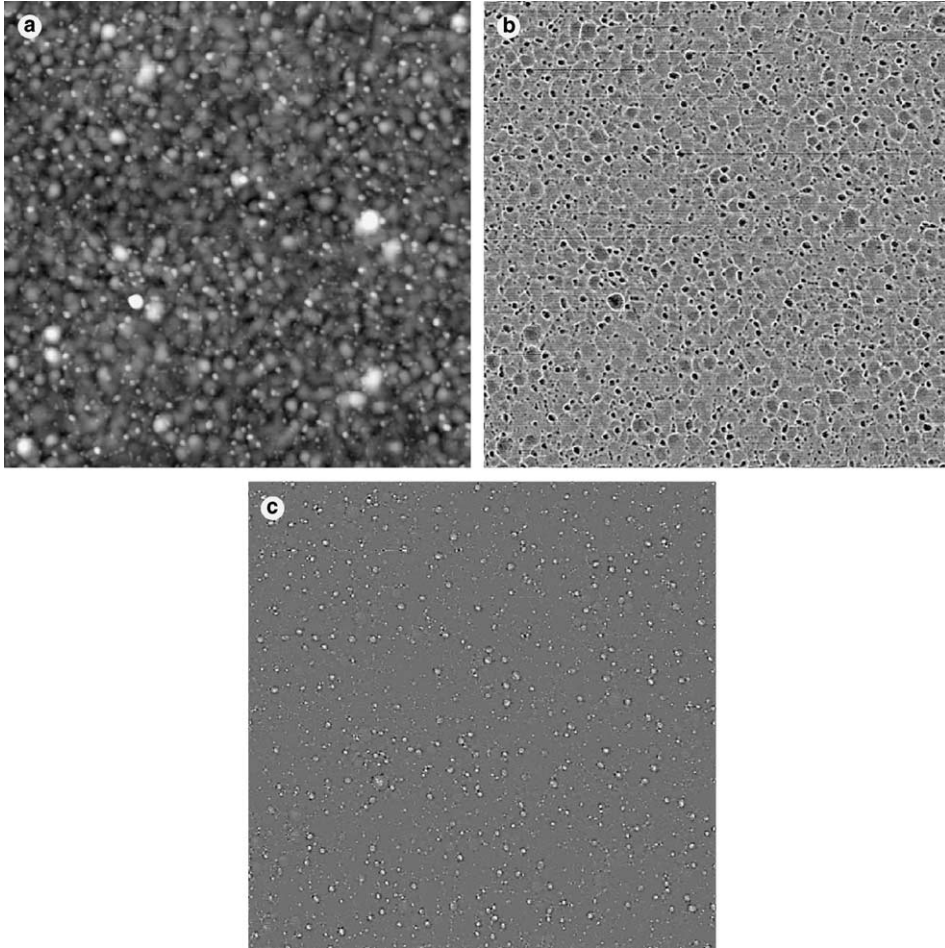


Fig. 4. $1 \mu\text{m}^2$ topographic (a), average curvature image (b) and gaussian curvature image (c) of a gold layer deposited on a glass surface by an evaporation process. The gray scale are 10 nm, -0.1 to 0.1 nm^{-1} and -0.03 to 0.03 nm^{-2} respectively. The two images show distinct regions that are homogeneous. We interpret this as the signature of the tip.

image and the non-topographic image. On the other hand, if the surface radii are large as compare to the tip radii, the topographic image is only slightly dilated and more closely represents the true sample surface. Then, the non-topographic image is sensitive to the variation of the surface curvature radius:

$$\frac{1}{R^*} \approx \frac{1}{R_{\text{Sample}}} \quad (14)$$

In this case, a correlation can be established between the curvature images and the non-topographic images. This is why only a low tip radius should be chosen for accurate phase imaging interpretation.

Under the hypothesis that discrete SPM images are good representations of continuous and derivable functions, the second order derivative and therefore the curvature of the surface can be computed. Eqs. (6) and (7) show that the average curvature and gaussian curvature are very sensitive to the second order derivatives of the surface and to high frequency topography or noise. Thus, it is necessary to have a high quality imaging, with the signal to noise ratio as high as possible and the sampling in the z direction as low as possible.

For a lot of surfaces, the interesting parameter is not the high frequency topography but the gen-

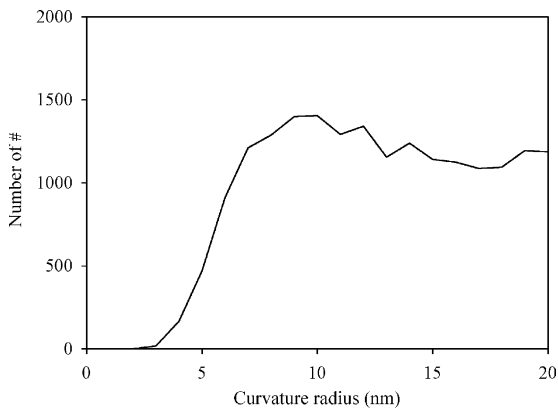


Fig. 5. Histogram of the gaussian radius of the convex pixels of the Fig. 4. The histogram shows a maximum value of 9–10 nm. Due to the dilation phenomenon, this value is attributed to the tip radius.

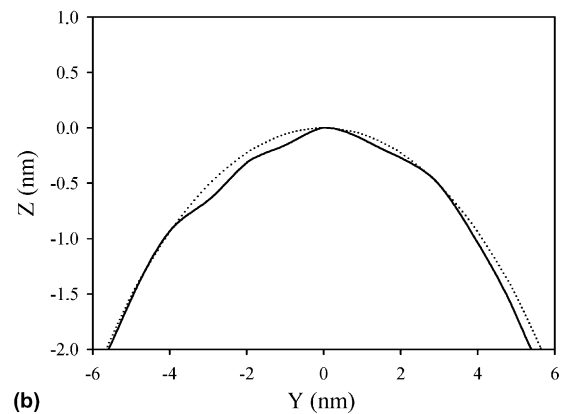
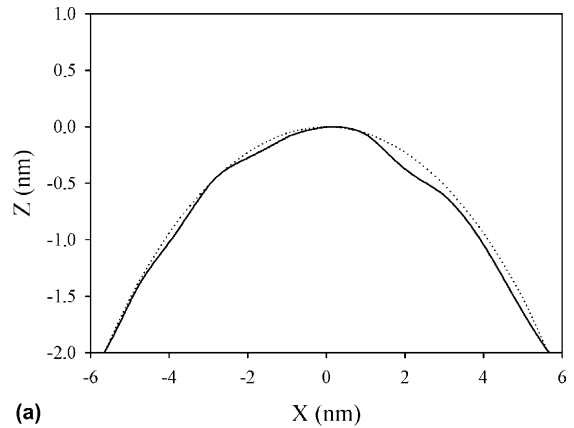


Fig. 6. Profiles in the horizontal and vertical directions of the reconstructed tip using a blind reconstruction method. These two profiles can be fit by 9 nm radius circles, in good agreement with the value given by the curvature analysis method (10 nm).

eral shape of the surface features. In addition, SPM images are generally noisy in the slow scanning direction due to the drift between two following lines. So, it is necessary to remove both the noise and/or the high frequency topography. A couple of solutions can be chosen:

- A low pass or Fourier filter can be applied to the topographic images, the derivatives or the curvature images. Filtering is generally necessary to eliminate the noise and obtain a realistic value of the curvature.
- A second solution is to compute the derivatives using a Kernell filter with a 5–5# or a 7–7#

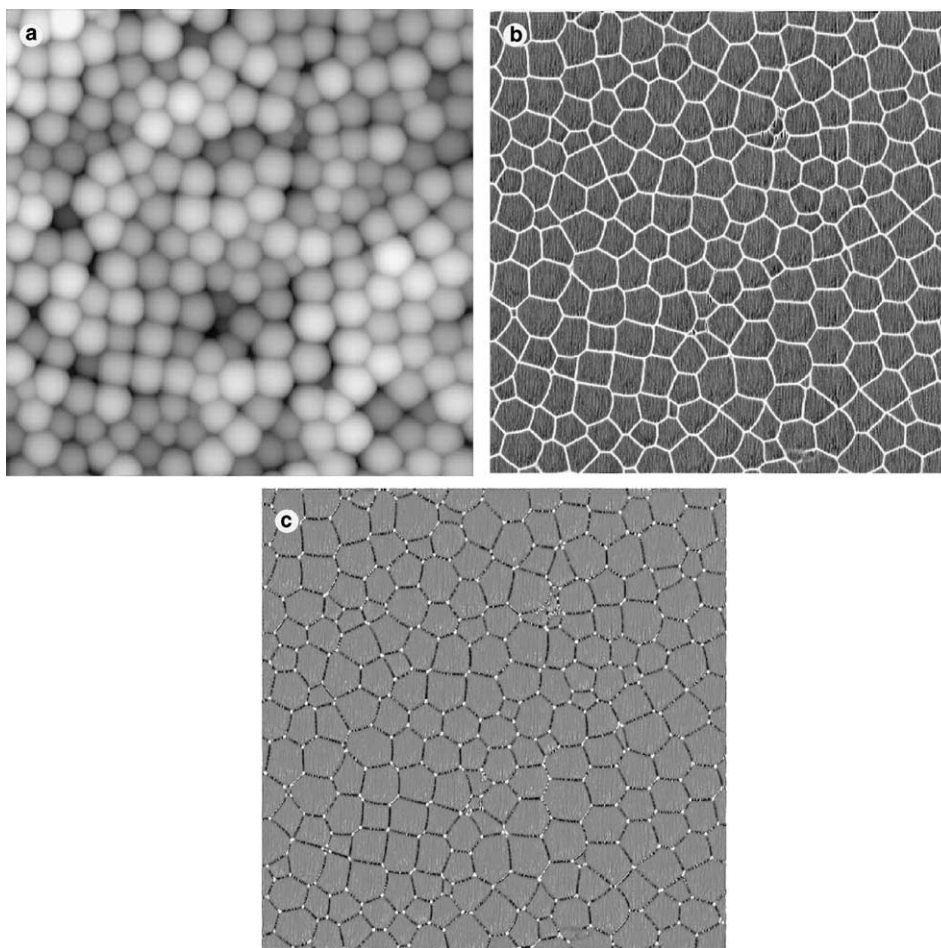


Fig. 7. $1\ \mu\text{m}^2$ topographic (a), average curvature image (b) and gaussian curvature image (c) of glass spheres deposited on a glass surface. The gray scale are 68 nm, -0.1 to $0.1\ \text{nm}^{-1}$ and -0.01 to $0.01\ \text{nm}^{-2}$ respectively.

matrix instead of a 3–3# matrix.¹ The derivatives computed are less sensitive to noise but tend to smooth the value of the curvature.

¹ The derivatives are computed using a Kernell filter. It is the sum of all the members of a matrix product of the Kernell filter and of a matrix of the same size extracted from the image. To decrease the influence of the noise, the derivatives are computed with a 3–3#, 5–5# or 7–7# Kernell filter in order to average the derivatives on a bigger area. For example, the 3–3#, 5–5# and 7–7# Kernell filters for the first derivatives in the x direction are respectively $\frac{\Delta z}{2\Delta x} \begin{vmatrix} -1 & 0 & 1 \end{vmatrix}$, $\frac{\Delta z}{8\Delta x} \begin{vmatrix} -1 & -2 & 0 & 2 & 1 \end{vmatrix}$ and $\frac{\Delta z}{36\Delta x} \begin{vmatrix} -2 & -3 & -6 & 0 & 6 & 3 & 2 \end{vmatrix}$.

3. Application examples

3.1. Tip and surface curvature measurement

Fig. 2a shows a virtual surface. This surface was generated by the superposition of gaussian like features of various widths and heights. If this surface is mapped by a perfect spherical with a radius of 20 nm radius, the surface will be dilated. The resulting image is presented as Fig. 2b. From the two images, we can compute an average and a gaussian curvature images (Fig. 2c–f). The sharpest features of the surface are dilated. Their image reflects the tip geometry rather than the surface

geometry. These features have the same curvature as the tip and appear in white in the average curvature image and in black in the gaussian curvature image. From these two curvature images it is possible to extract a gaussian radius R_G and an average radius R_A of the surface:

$$R_A = \frac{1}{A} \quad (15)$$

$$R_G = \frac{\sqrt[4]{G^2}}{G} \quad (16)$$

Fig. 3 presents the gaussian radius (R_G) histogram of the convex pixels (pixels describing a hill: $G > 0$ and $A < 0$) of the virtual surface and its corresponding image. The image average radius histogram shows clearly that the image is dominated by the tip radius. R_A histogram gives a similar result (not shown). The surface convex features that have smaller radii than the tip are dilated and therefore become images of the tip. It is not possible to have convex features with a lower radius than the tip in the image of the surface. Thus, the histogram of the average or the gaussian radius is truncated at the value of the probe radius.

Experimentally, the presence of noise and the fact that the probe is not perfectly spherical are limiting factors of the method. The signature of the tip is not so obvious on the histogram of the curvature radius of a real image. Fig. 4 presents topographic, gaussian and average curvature images of a gold layer deposited on a glass surface using an evaporation process. The topographic images have been acquired using the Tapping Mode AFM imaging and a silicon cantilever (TESP, nominal radius 10–20 nm). The computed curvature images show homogeneous regions especially over the sharpest features. We interpret this as evidence that the image is dilated by the tip and that the curvature images reflect the curvature of the tip rather than the surface curvature.

Fig. 5 shows clearly that the gaussian radius histogram of the convex pixels has a peak at 9–10 nm which we can attribute to the tip radius. There is an excellent agreement between this value and the profile of the reconstructed tip using a modified ‘partial tip reconstruction’ [22]. Fig. 6 shows the profile in the horizontal and vertical directions

of the reconstructed tip. The two profiles can be fit by 9 nm circles.

The main advantage of the present method (Curvature Analysis method, hereafter called CA method) as compare to the Blind Reconstruction methods (hereafter called BR methods) is the computing time required. The CA method can be carried-out in a few seconds whereas BR methods require a long computing time (few minutes to few hours) making them not practical. However, the CA method does not give a reconstructed tip but an average tip radius of the probe. This method can be used as a test to control the tip quality, especially in automated SFM, to detect the dilation’s influence on the image, to estimate the tip radius when quantitative measurement are required, and to validate a reconstructed tip.

Fig. 7 shows topographic, gaussian and average curvature images of glass spheres (Clariant, France) adsorbed on a glass surface. The theoretical radius of the spheres is 35 nm. The histogram of the gaussian radius deduced from the gaussian curvature image is centered on 42 nm (Fig. 8). This radius size is different from the theoretical value but in good agreement with the average distance between the center of two spheres in contact (80 nm) as it could be measured on a profile. Unlike the previous case, the topographic image is only slightly dilated and the histogram reflects the surface curvature instead of the tip curvature.

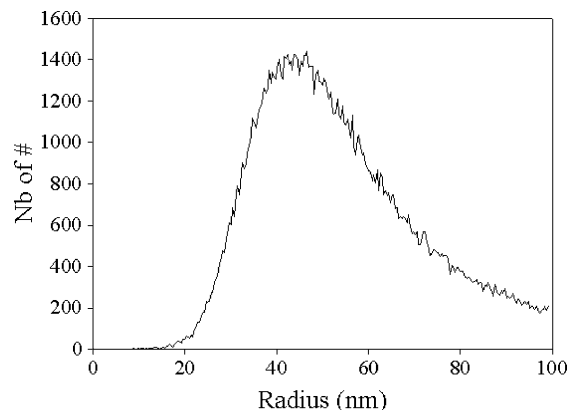


Fig. 8. Histogram of the gaussian radius of the convex pixels of Fig. 5. The histogram presents a maximum for a value of 42 nm. This value is attributed to the average sphere radius.

This method can therefore be used to characterize the curvature of objects such as nanotubes and proteins if their radii of curvature are large enough compared to the tip radius to prevent the dilation effect from occurring.

3.2. Phase signal and curvature correlation

When imaging with the Tapping Mode AFM, there are two cases when the topographic images and the phase contrast images can be directly related.

- (1) In the case of light tapping (ratio set point close to 1), there is a direct relation between the phase signal and the amplitude signal. It can be explained by a servo loop time delay, which does not maintain a constant vibration amplitude for the cantilever (Eq. (1)).
- (2) In the case of lower ratio set point, and according to the literature [29,45,46], the phase signal is sensitive to energy dissipation. If the material properties do not change, variations in the phase signal can be interpreted as variations of the tip/sample interac-

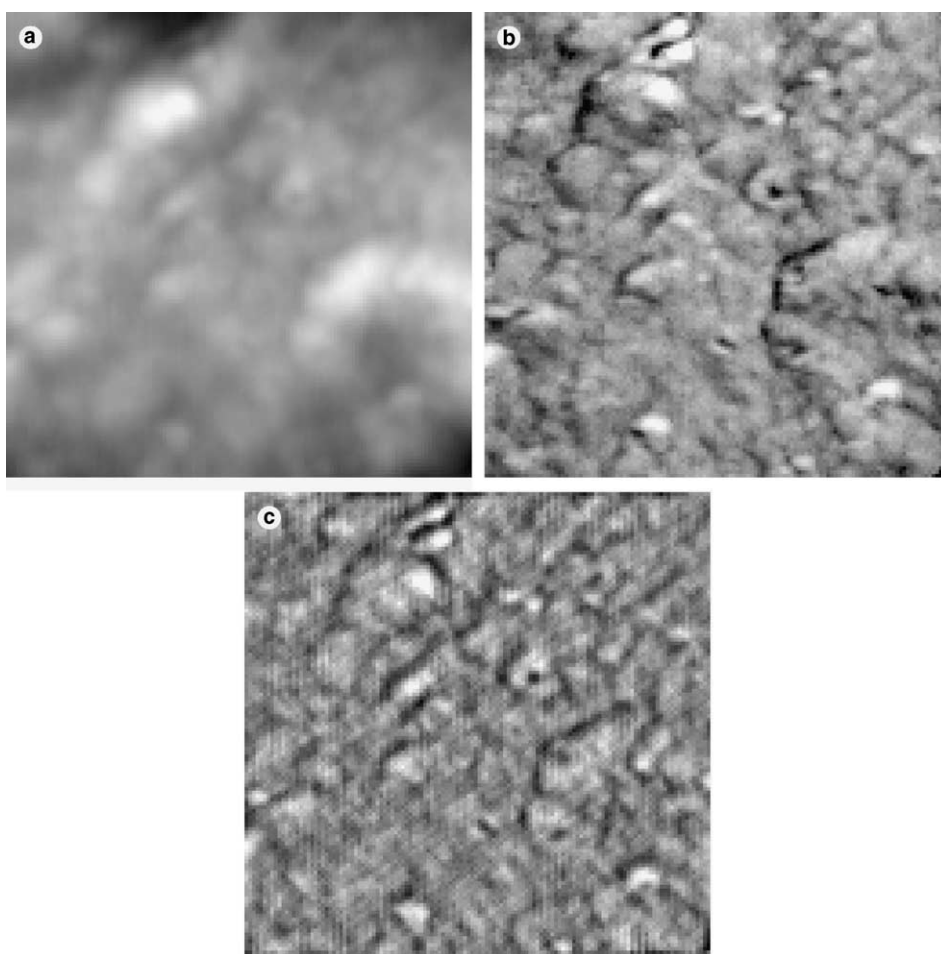


Fig. 9. $0.25 \mu\text{m}^2$ topographic (a), phase image (b) and average curvature image (c) of a polystyrene surface. The gray scale are 19 nm, 22° and 0.02 to -0.02 nm^{-1} respectively. An excellent correlation between the last two images is clearly noticeable.

tions due to variations of the surface sample curvature. Then, if the sample curvature radius is small compared to the tip curvature radius, the curvature and the phase images will present some similarities.

Fig. 9 presents the topographic image, the gaussian curvature image and the corresponding phase image of a polystyrene sample. An excellent correlation is clearly noticeable between the curvature image and the phase image. In this case, the phase contrast must be interpreted as a variation

of the sample curvature and not as a variation of the local sample properties.

Fig. 10 presents the topographic image, the gaussian curvature image and the corresponding phase image of a ‘polypropylene choc’ sample. This material is heterogeneous and contains inclusions of an elastomeric compound. The phase image clearly shows a high contrast corresponding to the polypropylene (white) and the elastomere (black). On the homogeneous part of the sample, the phase contrast and the curvature contrast are correlated, but the high contrast between the two

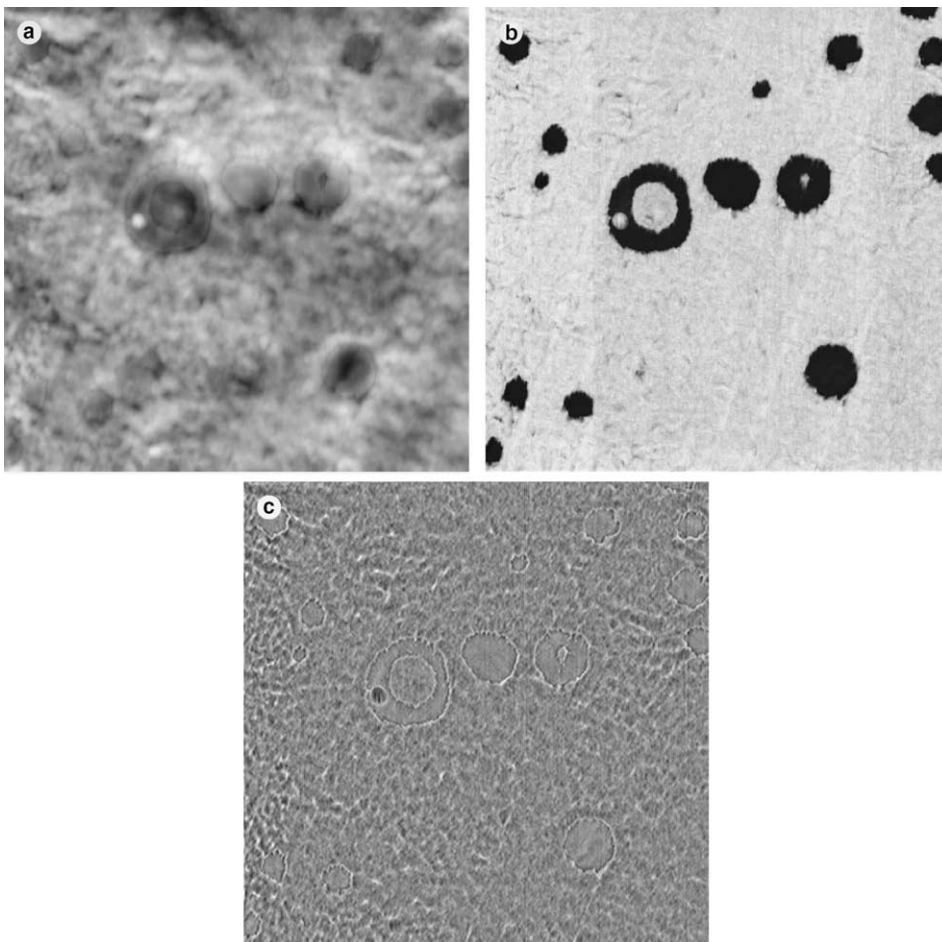


Fig. 10. $4\ \mu\text{m}^2$ topographic (a), phase image (b) and average curvature image (c) of a polypropylene choc surface. The gray scale are 29 nm, 59° and -0.05 to $0.05\ \text{nm}^{-1}$ respectively.

materials cannot be explained by a change of the sample curvature. In this case, as expected, we should conclude that the phase contrast reflects a change of sample properties.

4. Conclusion

The analysis of the curvature of topographic images is of great help to interpret SFM data. It can be used to detect the dilation effect and then to deduce the radius of the tip. This information is necessary for quantitative measurements of the physical properties of a surface. When no dilation effect exists, the analysis gives quantitative measurements of the curvature of spherical objects such as spheres, ellipsoids or tubes. In addition, a curvature image is useful to interpret the origin of the contrast seen in phase images obtained using Tapping Mode imaging. If the two images are similar, we deduce that the contrast is not due to a change of sample properties but due to the curvature of the sample. If the two images cannot be correlated, the interpretation is more difficult. The contrast can be attributed to a heterogeneous sample, to a signal not sensitive to the reduced curvature or to a dilation effect. Therefore, before any images interpretation, it is of major importance to investigate the role that the tip and sample curvature played on the contrast of SPM images, using the present method and the conditions required.

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