2.5 Ultimate resolution in the contact mode

2.5.1 The effect of elastic deformations

The AFM technique accuracy is limited by elastic deformations which modify a sample topography. One of the effects of this kind, the indentation of large organic molecules surface, results in the measured height decrease by several tens of percent. The same phenomenon can be expected when scanning across tilted, convex or concave surface areas (Fig. 1.1).

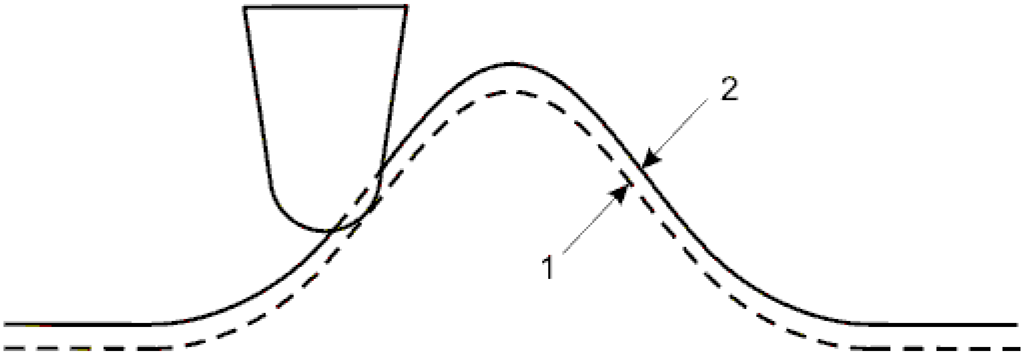

Fig. 1.1. Scan line profile (1) vs. original surface topography (2). Due to elastic deformations the surface convex feature shape is lower and narrower than the original.

The Hertz problem solution allows to estimate the best reliable resolution during measurements. Let us use formula (3) in chapter 2.2.2.2, which we rewrite as:

$$h = \sqrt[3]{\frac{F^2}{K^2 R}}, \tag{1.1}$$

where h – depth of tip and sample mutual penetration, F – applied force, K – tip-sample effective Young's modulus (see formula (1) in chapter 2.2.2.2), $\frac{1}{R} = \frac{1}{r} + \frac{1}{r'}$, where r, r' – tip and sample curvature radii, respectively.

Formulation of the Hertz problem implies that penetration depth h is much less than curvature radius R; this limitation, however, can be neglected in estimations. Let us calculate the minimum characteristic size of the surface feature which is of the order of the deformation under the tip action, i.e. we assume that in (1.1) $h \approx R = \delta_1$. The scale δ_1 can be considered as the resolution limit due to elastic deformations:

$$\delta_1 \approx \sqrt{\frac{F}{K}}. \tag{1.2}$$

This expression approximates both vertical and lateral resolution limit for the small features.

Notice that not only small but larger surface features ($r' \gg r$) can be imaged with shape distortion. If vertical elastic indentation (1.1) for the large features is practically the same as for the small ones ($R \sim r$) and thus can be neglected, the lateral shift of the inclined areas image must be taken into consideration because the shift sign depends on the inclination (Fig. 1.1).

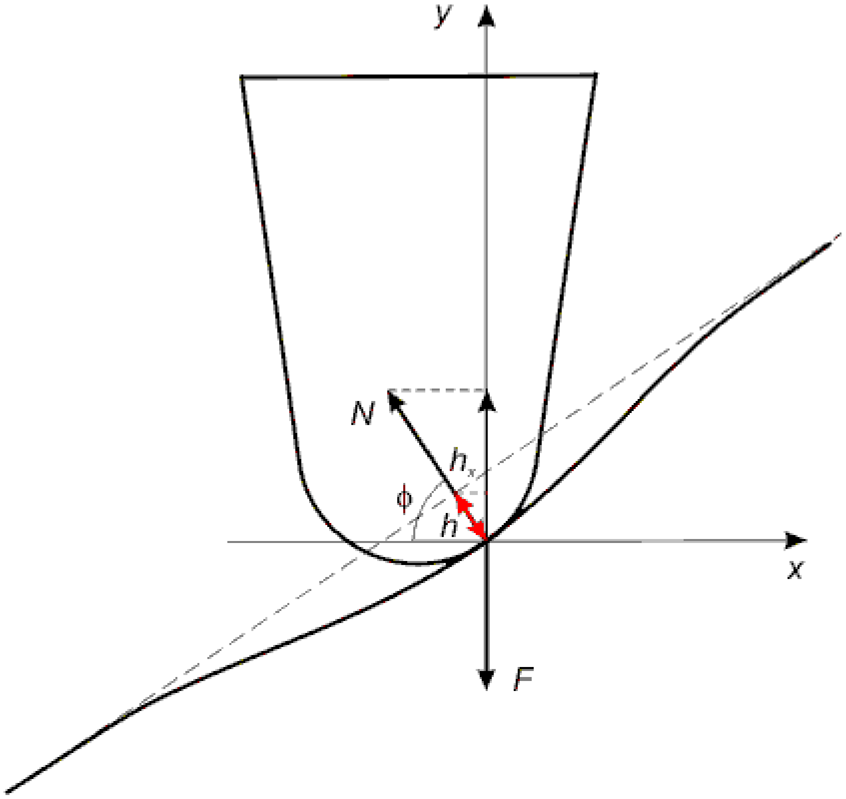

Fig. 1.2. Indentation during scanning of inclined area.

Examine now Fig. 1.2. The vertical pressing force causes the sample normal reaction force, which at the inclined area is given by:

$$N = \frac{F}{\sin\varphi}. \tag{1.3}$$

The arising deformation h is directed normal to the surface. Its horizontal component is:

$$h_x = h\cos\varphi \tag{1.4}$$

This component gives rise to the image distortions when scanning across inclined surface areas and can be taken as the lateral resolution limit due to elastic deformations. Substitute N instead of F in formula (1.1) in accordance with (1.2), use (1.4) and assume for large surface features $R = r$. Then:

$$\delta_2 = \frac{\cos\varphi}{\sqrt[3]{\sin^2\varphi}} \cdot \sqrt[3]{\frac{F^2}{K^2 r}}. \tag{1.5}$$

The obtained expression diverges at $\varphi \to 0°$; however, one should remember that the slope of the contact area can not exceed half the tip cone angle. That's why for the limit upper estimate (1.5) one should take namely this value as φ. For the typical tip cone angle $2\varphi = 22°$, the first multiplier in expression (1.5) second member is approximately equal to 3.0.

As a lateral resolution, one should choose the largest of quantities δ_1 (1.2) and δ_2 (1.5) depending on experimental parameters F, K, r values. If

$$F > 3Kr^2, \tag{1.6}$$

formula (1.5) should be used, otherwise – formula (1.2).

Let us calculate the resolution limit for the silicon cantilever with tip curvature radius $r = 10$ нм and cone angle 22° probing materials with various elastic modules. In table 1.1, the resolution values that satisfy condition (1.6) are marked gray, the other values satisfy condition (1.2)

F, nN K, Pa	0,1	1	10	100
10^8	1.4	6.4	30	140
10^9	0.32	1.4	6.4	30
10^{10}	0.10	0.32	1.4	6.4
10^{11}	0.03	0.10	0.32	1.4
10^{12}	0.01	0.03	0.10	0.32

Table 1.1. Lateral resolution limit, nm.

Summary:

1. Elastic deformation of the surface features by the tip leads to the AFM image distortion, which results in poor resolution.
2. Small features with sizes of the order of the deformation value can not be resolved. For the objects with curvature radius less than the tip radius, the resolution is approximated by $\delta_1 \approx \sqrt{F/K}$.
3. On the other hand, the image resolution is limited by the deflection at the sample inclined areas and is equal to $\delta_2 = 3\sqrt[3]{\frac{F^2}{K^2 r}}$.

2.5.2 Effect of the tip curvature radius and cone angle

Despite the ability to reach high spatial resolution, the acquired surface topography image can sometimes not correspond to the real surface features due to the effect of the instrument on the object resulting in the artifacts appearance. These artifacts, as a rule, can be easily taken into consideration while qualitatively interpreting the AFM results; however, some special problems can require quantitative estimation and reconstruction of sample true geometry. During scanning, two major AFM artifacts can appear: "profile broadening" effect due to the tip-sample convolution and "height lowering" effect due to the elastic deformation of studied objects. The last effect is considered in detail in chapter 2.5.1. In this chapter the elementary tip-sample convolution phenomenon is examined for the AFM operating in the contact mode. Different tip and sample feature sizes are considered.

Tip radius R is much less than the feature curvature radius r (R << r)

Fig. 2.1 depicts the studied object and conical tip geometry in case $R \ll r$.

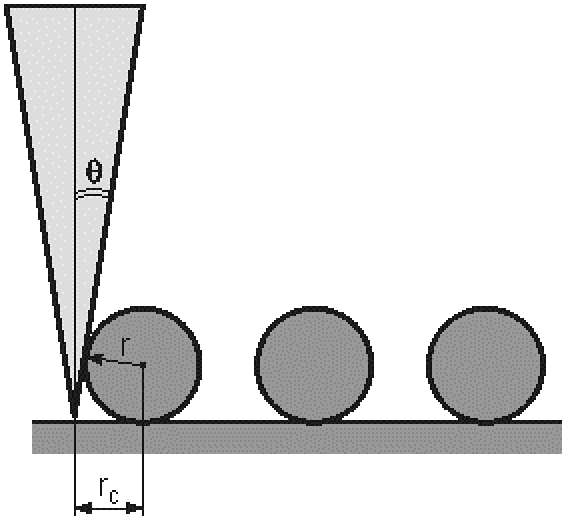

Fig. 2.1. Schematics of the studied object and conical tip in case $R << r$

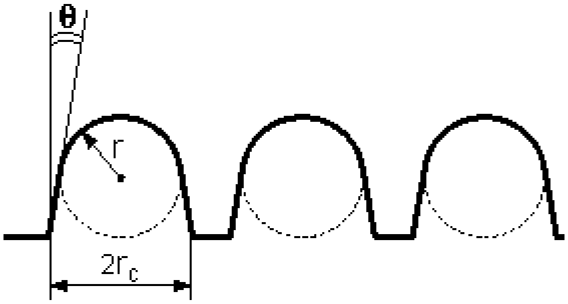

Fig. 2.2. Image profile of objects shown in Fig. 1.

From the case analysis it easily follows that the object lateral width is:

$$r_c = r(\cos\theta + \sqrt{\cos^2\theta + \left(1+\sin\theta\right)\left(-1+\tan\theta/\cos\theta\right) + \tan^2\theta}), \tag{2.1}$$

where θ – the cone half angle. The corresponding surface profile in the contact mode under given conditions is shown in Fig. 2.2. In this case, the object is broadened by $2(r_c - r)$ while its height remained the same - $2r$.

Tip radius R is approximately equal to the feature curvature radius r ($R \approx r$)

The tip and studied object geometry, when $R \approx r$, are shown in Fig. 2.3.

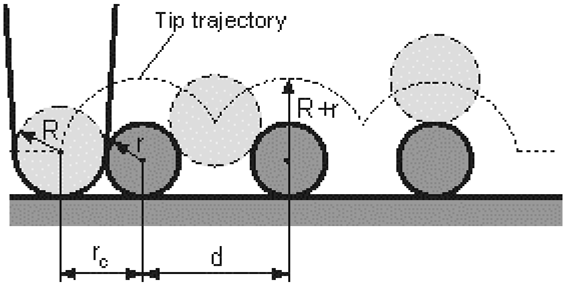

Fig. 2.3. Schematics of the studied object and conical tip in case $R \approx r$. Dotted line represents the tip path.

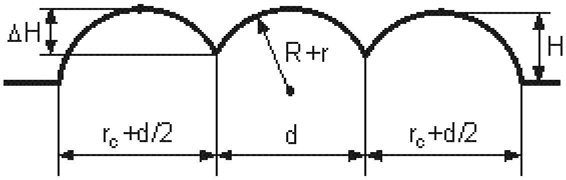

Fig. 2.4. Image profile of objects shown in Fig. 3.

In this case, the tip move across the object surface can be approximated by the sphere of radius R move along the sphere of radius r surface, i.e. the tip describes arc of radius R+r. Elementary calculation gives in this case for the object lateral dimension

$$r_c = 2\sqrt{Rr}, \tag{2.2}$$

and for the relative height of the object

$$H = r\left[1 - \sqrt{1 - \frac{r_c^2}{\left(R+r\right)^2}}\right]. \tag{2.3}$$

If minimum distance between features $d - 2r$ is less than the tip diameter: $2R > d - 2r$ (see Fig. 2.4), then, during the tip passing between them, it will penetrate as deep as:

$$\Delta H = r\left[1 - \sqrt{1 - \frac{(d/2)^2}{(R+r)^2}}\right] \qquad (2.4)$$

Quantities H and ΔH are shown in Fig. 4 where the surface image profile is depicted for the given conditions taking into consideration the tip-sample convolution. In this case, the object broadening value is ($r_c - d/2$). Moreover, the tip finite size does not allow it to penetrate into narrow cavities on the sample surface resulting in their depth and width decrease.

Lateral resolution of an AFM

The resolution criteria in the normal direction ΔZ can be the minimum Z-coordinate change during scanning which can be detected at a given noise level. Resolution depends much on scan parameters (speed, scan size, parameters of the feedback circuit proportional and integration sections) as well as on the sample elastic properties. Normally, the vertical resolution is several tenths of an angstrom.

There is no unambiguous procedure of the microscope lateral resolution determination. The simplest way to estimate it is as follows.

Let R be the probing tip curvature radius and r – resolvable surface feature radius (Fig. 2.5). Then lateral resolution is connected with the vertical resolution limit ΔZ. The resolution criteria is the ability to detect the difference in the tip vertical coordinate over features and between them.

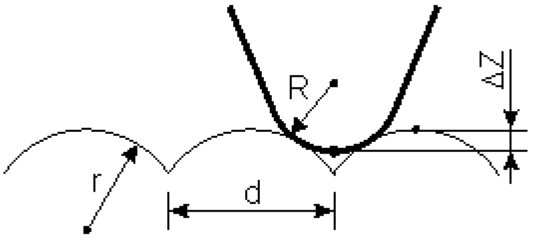

Fig. 2.5. On defining the lateral resolution: ΔZ — vertical resolution limit, d — desired lateral resolution limit, R and r — curvature radii of tip and resolved objects

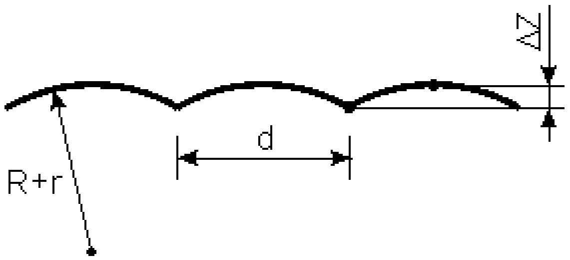

Fig. 2.6. Expected result of AFM topography study of surface shown in Fig. 5.

The geometrical analysis (Fig. 2.5) allows to obtain the expression for the minimum separation between resolved asperities when the AFM image "dip" can still be detected (i.e. equal to the limit ΔZ):

$$d \simeq \sqrt{8(R+r)\Delta Z}. \qquad (2.5)$$

Because the best spatial resolution must be the invariant characteristic of the instrument (independent on the studied object), it should be defined, e.g. from the condition of two point objects ($r = 0$) detection. Then formula (2.5) is written as:

$$d = \sqrt{8R\Delta Z}, \qquad (2.6)$$

relating lateral resolution limit d, vertical resolution limit ΔZ and tip curvature radius R.

Summary:

1. To reconstruct the sample true geometry one should solve the inverse problem of tip-sample convolution.
2. If $R << r$, the feature size in the image is defined by formula (2.1).
3. If $R \approx r$, the feature size in the image is defined by expressions (2.2)-(2.4).
4. The best lateral resolution in the contact mode depends on the vertical resolution limit (quoted AFM characteristic) and tip curvature radius (2.6).