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Influence of atomic force microscope tip–sample interaction on the study of scaling behavior

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Images acquired with atomic force microscopy are based on tip–sample interaction. It is shown that using scanning probe techniques for determining scaling parameters of a surface leads to an underestimate of the actual scaling dimension, due to the dilation of tip and surface. How much we underestimate the scaling exponent depends on the shape and aspect ratio of the tip, the actual fractal dimension of the surface, and its lateral–vertical ratio. © *1997 American Institute of Physics.* [S0003-6951(97)03536-5]

Since its introduction in 1968, atomic force microscopy (AFM) has mostly been used in the field of high resolution surface studies. More recently it received increased attention in many other fields for its competence to image microstructural features from nano- to mesoscopic scale. Of great importance in these studies is the actual imaging process. Especially the tip–sample interaction is one of the determining factors. Due to the fact that the tip is finite sized, the obtained image can be distorted, which has been illustrated in several articles in literature.[1,2] Many microscopists use the word convolution to describe this distortion. As we will explain later, this distortion is not a convolution in the mathematical sense, but is better described by the concept of dilation. If one uses AFM to study scaling behavior of fractal surfaces, the question can be raised to what extent the observed scaling exponent (fractal dimension) is influenced by this distorted image of the surface under investigation.

The motive for this investigation stems from a previous study,[3] where the scaling behavior of the surface roughness of highly porous ceramics was analyzed. The pore sizes of SiO_2 and Al_2O_3 were determined by mercury porosimetry and stereo transmission electron microscopy analysis. In these materials a high inner surface area (typically 250 m^2/g) combined with a large porosity (70 vol %) provides a large contact area between catalyst and the material to be converted. Pore diameters in these materials are usually between 7 and 200 nm and can be fine tuned using particular processing.[4] On these fracture surfaces we observed that the roughness exhibited a limited scaling range. Due to the existence of a network of pores however, a very rough fracture surface is created and we encountered dilation problems. This gave rise to the question, to what extent is the observed scaling exponent (fractal dimension) influenced by this distortion.

The method we have used in determining the scaling exponent of the fracture surface is based on the assumption that the surfaces can be described by a superposition of waves of all wavelengths and random phases. The amplitude of this roughness at different wavelengths is given by the power spectrum of the surface. If we assume that the surface height distribution obeys Gaussian statistics, we may extract the fractal dimension using the height correlation function of the surface,

$$S(\Delta x)=\langle |z(x)-z(x+\Delta x)|^2\rangle, \tag{1}$$

where according to Berry[5] the correlation function can be rewritten with the help of Eq. (1) as

$$S(\Delta x)=C\alpha^{2(D-1)}\Delta x^{(4-2D)}. \tag{2}$$

The correlation function is formally equivalent to the structure function $S(\Delta x)$ [Eq. (2)] and can be used to determine the fractal dimension D of the surface. In these equations Δx can be considered to be a roughness ''wavelength'' of the fracture surface. In a plot of log $[S(\Delta x)]$ vs log (Δx), a straight line yields the fractal dimension. Here we call this type of analysis the one-dimensional correlation function analysis, not to be confused with the Fourier profile analysis,[6,7] since here we refer to an analysis in real space. $S(\Delta x)$ is usually termed the height-difference correlation function in order to be distinguished from the height-height correlation function $\langle z(x)z(x+\Delta x)\rangle$.

In our previous study also another method, based on the rms roughness[8–10] of the surface was used. The one-dimensional correlation function method is found to be a rather restricted method, due to its sensitivity for the influence of long wavelengths. The correlation function analysis could only be used for wavelengths up to 1/4 of the data-set size. Both the dimensions found with the rms method and the one-dimensional correlation function method correlate to each other[11] which is to be expected since rms $(L)^2=(1/L)\int_0^L S(x)\,dx$, leading to a rms scaling behavior of rms $(L)\propto L^{2-D}$. The reason for using the one-dimensional correlation function method in this letter is for convenience, since for the determination of the scaling exponent just a single picture has to be simulated and consequently calculation times are reduced considerably.

As mentioned before, dilation of tip and surface plays an important role in scanning probe microscopy on rough surfaces. In Fig. 1(a) the problem is explained. Due to the interaction with the tip, surface details will dilate, loosing all original information steeper than the tip itself. Although in literature it is often suggested that this is just a convolution problem, this is clearly not the case. If we observe two signals convoluting with each other, the result will be the prod-

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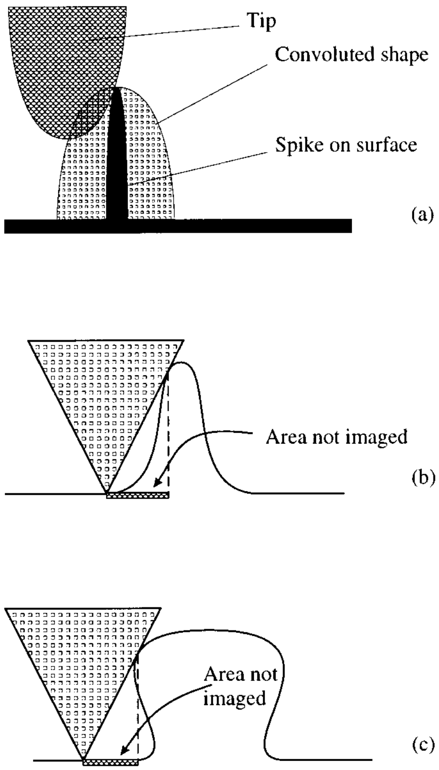

FIG. 1. Dilation of tip and surface (a), resulting in loss of information on single (b) and not single (c), valued surfaces.

uct of both signals. If we now know one of the original surfaces, we can reconstruct the original structure. This process is actually a Fourier convolution, which is a reversible, linear process. In the actual image forming process however, information is lost. This loss of information comes from the fact that the (dilating) process decouples the lateral and vertical interaction in between the tip and the surface. This means that interaction is assumed to take place at the apex of the tip, although it often takes place at the side of the tip. If the surface is single valued, that is for each (x,y) co-ordinate there is a unique z value, steepness of the surface is the limiting factor. When looking at Fig. 1(b) it is clear that the shaded area is distorted in the acquired image, due to the fact that there are two contact points between tip and surface. If the tip is steeper than the surface, the tip will have only one contact point with the surface and the actual surface will be imaged. If the surface is not single valued [Fig. 1(c)] loss of data will also occur, since only the envelope of the surface is imaged.

Afterwards we can only conclude if the surface was steeper than the tip, not how much steeper (provided we know the shape of the tip). Some parts of the surface can be reconstructed using etching algorithms,[12] but this is only the case if the tip touches the surface at only one contact point at a time. The shaded areas as indicated in the Fig. 1(b) and 1(c) cannot be reconstructed. In general, only very limited areas can be reconstructed.

In order to study how exactly this tip–surface interaction will influence our fractal analysis, several model calculations were done. For these model calculations fractal surfaces were generated using a Brownian motion type algorithm.[13]

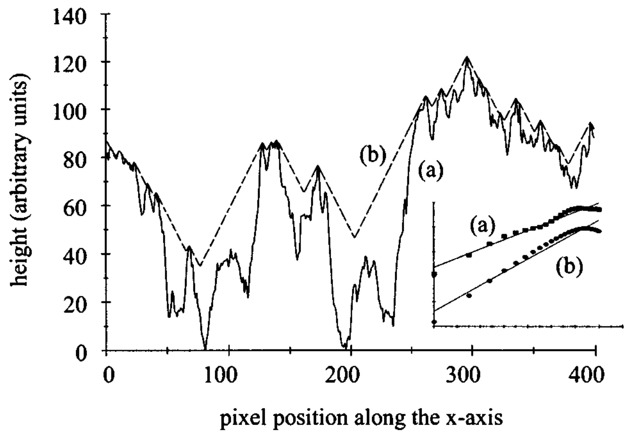

FIG. 2. Height profile of the fractal surface (a) before and (b) after dilation. The inset shows the one-dimensional correlation function analysis, plotting log $[S(\Delta x)]$ against log (Δx). This results in (a) $D=1.51$ before dilation and (b) $D=1.34$ after dilation.

This algorithm enabled us to generate surfaces with fractal dimensions varying from 1 to 1.5. In many cases[14–17] fractured surfaces show static scaling exponents in the range between 0.2 and 0.8, i.e., D between 1.2 and 1.5. These artificial surfaces were analyzed before and after being dilated[18] with a tip with known aspect ratio. Figure 2 shows us a line profile of such a generated surface. The surfaces were dilated with several different tips with different aspect ratios. The aspect ratio can be calculated from the top angle of the tip: $2\tan(\text{top angle}/2)$. The standard, pyramidal shaped tip we used in our experiments has an aspect ratio of 1.15, the conical shaped single crystal silicon NanoProbe (which we also have used in some experiments) has an aspect ratio of 0.35.

As we can see from Fig. 3, the analyzed fractal dimension D after dilation depends on both the initial fractal dimension of the surface under investigation and the aspect ratio of the tip used. The dilation process will lead to an underestimate of the actual scaling behavior of the surface. Intuitively, one would expect the effect of the aspect ratio to decrease with decreasing fractal dimension, which is indeed

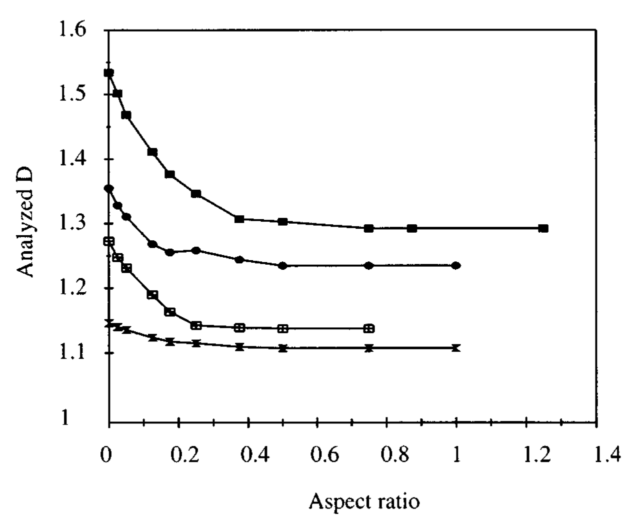

FIG. 3. The analyzed fractal dimension D from a simulated surface as a function of the aspect ratio of the tip. The intersection of the curves with the vertical axis gives the initial fractal dimension of the surface.

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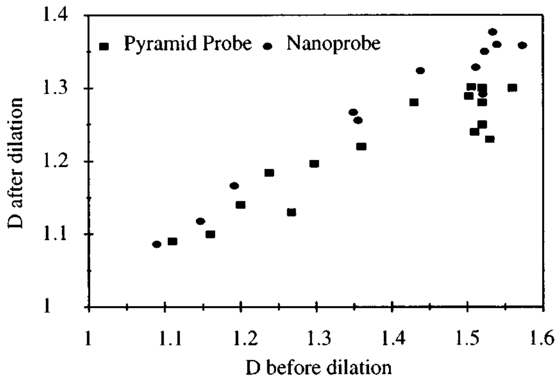

FIG. 4. The fractal dimension before and after dilation of tip and surface.

the case, except for the ''open squared'' experiment in Fig. 3. It shows a larger effect of the aspect ratio than expected. We expect this to stem from statistical fluctuations, since only one surface was generated for each curve.

The curves of the analyzed D versus the aspect ratio show a saturated behavior, which we expect to stem from the fact that we have only generated fractal surfaces with a limited scaling range. Large wavelengths, which will only be distorted by tips with large aspect ratios, are not present in the generated data sets. We can also translate this figure to a graph from which we can estimate the initial fractal dimension of the surface for the two tips we have used in the real experiments (Fig. 4).

One has to note that these generated surfaces have a fixed lateral–vertical ratio, which is important for the exact dilation behavior. At first glance this may sound strange for a fractal surface, since any part of the surface is in the statistical sense equal to the other parts. But the artificial surfaces have a limited resolution of 400 pixels. By adjusting the scansize of this picture, the distance between pixels and hence the slope of the curve is altered. All generated pictures have the same lateral–vertical ratio of 1.6, which is defined as the ratio of the scansize over the maximal height within the picture.

Another point to be noted is that the generated fractal surfaces are pure two-dimensional surfaces. Each line of the generated AFM image is equal to all other lines. This simplifies the dilation problem, since there is no ''line-to-line'' dilation. This also implies that in these model calculations differently shaped tips with the same aspect ratio yield the same results. In a two-dimensional representation both the pyramidal and conical shaped tips are the same. For the three-dimensional case we expect that, when compared to a conical shaped tip with the same aspect ratio, the often used pyramidal shaped tip will yield a larger distortion of the surface due to the stronger line-to-line dilation behavior, leading to an even larger underestimate of the fractal dimension.

Tip effects play an important role in the determination of the fractal dimension. The fractal dimension of a surface determined with a scanning probe technique will always lead to an underestimate of the actual scaling dimension, due to the dilation of tip and surface. How much we underestimate the scaling exponent depends on the aspect ratio of the tip, the shape of the tip, the actual fractal dimension of the surface, as well as on the lateral–vertical ratio of the surface itself. In general, the aspect ratio of the tip proves to be the limiting factor in the imaging process. If fractal surfaces are to be imaged with an acceptable amount of distortion, tips with aspect ratios of around 0.05 would have to be developed. Such a small top angle would result in a very fragile tip and would therefore not be suitable for use on rough surfaces.

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