Some Effects on SPM Based Surface Measurement

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Abstract. The scanning probe microscope (SPM) has been used as a powerful tool for nanotechnology, especially in surface nanometrology. However, there are a lot of false images and modifications during the SPM measurement on the surfaces. This is because of the complex interaction between the SPM tip and the surface. The origin is not only due to the tip material or shape, but also to the structure of the sample. So people are paying much attention to draw true information from the SPM images. In this paper, we present some simulation methods and reconstruction examples for the microstructures and surface roughness based on SPM measurement. For example, in AFM measurement, we consider the effects of tip shape and dimension, also the surface topography distribution in both height and space. Some simulation results are compared with other measurement methods to verify the reliability.

1. Introduction
The SPM family, mainly including scanning tunneling microscope (STM), atomic force microscope (AFM) and scanning near-field optical microscope (SNOM), has been widely used in nanometer or even atomic scale measurements shortly after the first invention of STM in 1981. The ultra-high resolution of SPM attracts researchers in different fields greatly. In precision engineering, more efforts are made to use SPM instruments to meet the increasing demands of metrology for microstructures with high accuracies [1].

For SPM based measurements, there are some important issues we must pay much attention. In this paper, we give a brief review on some fundamental results obtained by our group in field of SPM based surface measurement. We focus on the issue of drawing true information from the SPM images. Topographic structure of surface influences a wide range of its functional properties, so quantitative, accurate evaluation of surface is of great importance [2]. All braches of SPMs cannot get the real surface for the non-ideal imaging conditions. Since the basic operation principle of SPMs is the proximal probe technique, images scanned by SPMs are likely the coupling of a lot of information besides the topography of sample. In some special cases, the topography, which is the main considered information in surface metrology, can be even opposite. Simply speaking, AFM images are combinations of tip geometry and sample topography. The images scanned by STM are more likely to reflect the electrical properties of conductive tips and samples. SNOM signals are greatly influenced by illumination conditions. So it is essential to make surface topography reconstruction for better understanding and characterizing of the scanned images.

We also present some of our previous numerical simulation results on the geometrical factors affecting surface roughness measurements [3]. Parameters of both tip and sample are considered such as tip radius, surface autocorrelation length, standard deviation of original rough surface and its height.
distribution. The rough surface is generated using 2D digital filter and Fourier analysis method with controlled autocorrelation function and height distribution [4]. The AFM image is simulated by mathematical morphology method [5].

2. Image Interpretation and Surface Reconstruction

Images obtained by SPM are rather complex to explain because they provided rich information beside the topography. No SPMs measure the topography alone. As mentioned before, STM yields images related to the electronic energies near the Fermi level of the samples. For AFM image, it is a convolution of sample topography and tip geometry. When measuring with AFM, the sample geometry is dilated with the shape of tips, producing significant distortions in the topography measurements of surfaces. Fig.1 schematically illustrated the case of measuring diameters of particles using AFM tip. The dotted line is scanned particle profile, which is obvious larger than the particle and lead to the over estimation of diameter. In SNOM, there have great differences between the detected signal and the interface topography because the image is influenced by illumination conditions and the optical properties of the samples and also the tips. Further more, the effects of humidity, adhesion etc. on the scanned images should be also taken into account. So surface reconstruction through the corresponding measured images is especially important in analyzing the real sample surface. At least, parts of real sample surface can be recovered by reconstruction.

In AFM, several reconstruction procedures have already been developed in the last few decades. May be the most usual way is to assumed some special geometry of the tip for example spherical, pyramidal, to simplify the reconstruction [6]. D.Keller’s Legendre transformation method is somewhat complex and it is sensitive to the noise [7]. Considering the scanned image is an envelope convolution of the tip and sample surfaces, the sample surface can be reconstructed close to the real one [8]. Based on mathematical morphology, Villarrubia developed an alternative method to reconstruct the sample surface. Mathematical morphology method is not sensitive to the downward noises [5]. M.F.Tabet’s method is by placing the simulated tip at each pixel in the scanned image and removing the areas where the volume of tip and surface overlap, and the remaining image is a closer description of the real surface geometry [9].

In Fig.2, we present an example of AFM image simulation and surface reconstruction using mathematical morphology algorithms. In mathematical morphology method, the convolution of sample and tip geometries can be written in a compact way as $I=S \oplus P$ [5]. Here symbol $I$ denotes the simulated image and $S$ is the set of the sample surface. $P$ is the reverse of the tip geometry and “$\oplus$” means the dilation algorithm. A two-dimensional grating surface is generated. The size of surface is assumed to be $1 \mu m \times 1 \mu m$ and the height of the surface is 20nm. In this figure, black color means lower height and white color means higher height. The tip used is assumed to be parabolic in the form of

$$z(x, y) = \frac{x^2 + y^2}{2R}$$

where $R$ is the radius of the tip at the apex and in this simulation, the tip radius equals 50nm. From the simulated AFM image, it can be seen that the sharp edges become much more smooth due to tip-sample convolution. And after reconstruction, the reconstructed surface becomes closer to
the original ones. However, there still have some deviations because the existence of regions which cannot be reconstructed. This can be seen clearly from the profiles.

![AFM image simulation and surface reconstruction. (A) Generated grating-like surface, (B) simulated AFM image, (C) reconstructed surface, (D) the profiles of the three images](image)

To verify the importance of the reconstruction, we compared the result of grain size of $\alpha$-Al$_2$O$_3$ determined by XRD with that by AFM [10]. A Rotating Anode X-Ray Diffractometer (Model D/MAX-rA, Rigaku Co. Ltd., Japan) was used with an operating voltage of 40 kV and currency 100 mA. Intensity measurement range was 2000CPS. The range of diffraction angle ($2\theta$) is from $10^\circ$ to $60^\circ$. The background noise of XRD instrument is 0.1°. The wavelength of X-ray is 1.5418Å. The XRD result is shown in Fig.3. According to the Scherrer equation, we got the average diameter of $\alpha$-Al$_2$O$_3$ nanoparticles about 34.0nm.

Then $\alpha$-Al$_2$O$_3$ nanoparticles dispersed on mica surface were measured by AFM quickly after being dried by nitrogen. The AFM result is shown in Fig.4, left image. The average diameter is 37.1nm obtained by the WSxM software (Nanotec Company, Spain).

The tip is a commercial one from Olympus Corp. The average radius of the tip is about 15nm. We use mathematical morphology reconstruction method [5] to eliminate the tip effect. After reconstruction we get the image shown in the right image, Fig.4. The average diameter is about 34.1nm. From above example, the diameter measured by AFM is close to the result of XRD. After considering the tip effect, the diameter is much more closer to the XRD result. From simulation and experiment, we demonstrate the importance of surface reconstruction.

The photon scanning tunneling microscope (PSTM) is sensitive to illumination conditions and to the distance between tip and sample. In many cases, the images do not reproduce the exact surface
Figure 3. The XRD image of $\alpha$-Al$_2$O$_3$ nanoparticles

Figure 4. Left: AFM image of $\alpha$-Al$_2$O$_3$ nanoparticles. Right: Reconstruction result considering the tip effect. The tip is a commercial one from Olympus Corp. with tip radius about 15nm.

topography. Several inverse solutions of the reconstruction of surface profile have already been published in the literatures. By reconstruction procedure, the super-resolution capabilities of the PSTM instrument could be enhanced [11]. We propose a way to reconstruct the surface topography directly through detected signals. Our method is based on perturbation theory and Fourier analysis under the assumption that the interface topography is far smaller than the laser wavelength and the multiple scattering between the sample surface and the detection tip can be ignored [12].

Fig.5 shows one example of PSTM image simulation of a sidestep sample and the corresponding reconstruction result. From Fig.5, the original topography is well reconstructed. The location and height of the small convex is precisely obtained. However, the sample shape is smoothed and additional small peaks emerge due to calculation precision.

3. Simulation on the geometrical factors affecting roughness of AFM images

In AFM measurements, the tip makes contact with the specimen when scanning the tip or sample in a raster way by the piezo-scanner. The location of the tip apex defines the measured height. When scanning, it is not necessarily the apex of AFM tip in contact with the sample. The difference between the real point of contact and the apex causes image distortion as shown in Fig.1. From an intuitionistic
point of view, it is obvious that the measured roughness depends on the tip radius \( R \), standard deviation of the surface height \( \sigma \) and the wavelength \( \lambda \).

Most real engineering surfaces may not have well-defined structures. They are usually composed of both random and periodic parts. The random nature makes roughness evaluation much more complex. To investigate the factors affecting the roughness measurements, the ideal way is to image a known surface with a known tip and then compare the results. However, this approach is very difficult to achieve experimentally and we choose numerical simulation. By numerical simulation, the surface can be generated efficiently and a better understanding of the geometrical factors affecting surface roughness measurements by scanning proximal probe techniques can be achieved. We adapt the 2D digital filter and Fourier analysis method to simulate the rough surface. By 2D digital filter method, the random surface with prescribed autocorrelation function can be generated. For Gaussian surfaces, we use the autocorrelation function as

\[
R(x, y) = \sigma^2 \exp\{-2.3[(x / \lambda)^2 + (y / \lambda)^2]\} \tag{2}
\]

In the above equation, \( R(x, y) \) is the 2D autocorrelation function of the random surface. \( \sigma \) is the standard deviation of heights, and the correlation distance is \( \lambda \). It is assumed the correlation length of the rough surface in the x direction is the same with that in y direction. The generated rough surfaces by 2D digital filter method are used as the original sample surfaces to simulate the scanned AFM images. The generated random surface with controlled autocorrelation function and the simulated AFM images are then evaluated by roughness parameters [3]. Here we only present the results concerning root-mean-square roughness, \( R_q \). The advantage of \( R_q \) is not only its simplicity, but also the statistical significance. Since \( R_q \) is the standard deviation of the height, it describes the spread of the height distribution about the mean value.
The dependence of geometrical properties of the original surface and tip radius on AFM image roughness measurement is presented in Fig.6. The roughness values for the scanned images are normalized to the roughness of the original images. For each of the Gaussian rough surface generated, a monotonous decay of the measured roughness with increasing tip radius is found. The reduction in the vertical roughness is caused by the AFM tip not being able to trace the surface to the bottom of the regions between the peaks [13]. The monotone character implies by proper interpolation procedure, the real roughness could be drawn accurately for normally distributed surface. From Fig.6 (A), as the correlation length increases, the deviation of roughness becomes more and more smaller at certain tip radius. AFM causes littler distortion on the surface with long correlation length. However, the decrease in roughness also depends on the standard deviation of the original surface heights as illustrated in Fig.6 (B). The effect of the standard deviation on roughness evaluation has almost the same trend as correlation length for normally distributed random surface. At small standard deviation, the roughness decreases smoothly with the gradual increase of tip radius. If the standard deviation is relative large, the roughness decreases quickly with the increasing tip radius. In case of the same tip radius and the same correlation length, the surface with high standard deviation causes larger distortion of the roughness. Also the measured roughness decreases monotonously with the increasing tip radius at certain standard deviation of the original surface. Fig.6(C) represents the dependence of RMS roughness $R_q$ on the correlation length at different tip radius. Here the standard deviation of all
the rough surfaces equals 5nm. For the rough surface with Gaussian distribution, when the correlation length is smaller than the tip radius, the measured roughness error is large, and the \( R_q \) value decreases rapidly as the correlation length increases. When the correlation length is several times of the tip radius, the increase of correlation length causes little effect on the measured surface and the real surface roughness value is produced faithfully. To investigate the effect of distribution on roughness evaluation, random surfaces with relative larger skewness are also generated. Another surface with negative skewness is also generated. In order to highlight the influence of skewness, the other parameters are the same as those of the surface with positive skewness. As shown in Fig.6 (D), the effect of tip radius on the surface with negative skewness is the same with that on a normally distributed one. However, for the surface with positive skewness, it tends to be a first increase and then reduce of \( R_q \) as tip radius increases. Unfortunately, it seems there is no simple analytical relation for such curves.

For a random surface, if its peaks are sharp and narrow while the valleys are wide and smooth, the surface tends to have positive skewness. The opposite case generates the surface with negative skewness. When the tip is sufficiently narrow and is able to probe the low spots between surface peaks with reasonable accuracy, \( R_q \) increases with increasing tip width. At the peak position, the measured image reflects more likely the geometry of the tip for the surface with negative skewness as shown in the figure. It is the tip-shaped depression in the image that causes the first increase of the measured roughness. As the tip radius continues to increase, \( R_q \) decreases with increasing tip radius under the conditions that the tip is unable to probe the valley between two peaks [13]. While for a random surface with negative skewness, the valleys are more difficult to image but the smooth peaks can be imaged accurately due to the finite width of tip. In imaging surface with negative skewness, the tip is unable to distinguish the valleys. It erases the structure that has smaller size than the tip radius. The tip feels the specimen surface as a smooth one without the sharp valleys. Thus the roughness of AFM image decreases as the tip radius increases. Detailed results are referred to reference (3).

4. Summary
SPMs are a key to the nanoworld [14]. They have played an important role in field of nanoscience and nanotechnology. No doubt, they will be more and more universally applied in the future. In this article, we present some results on applying SPMs in field of surface measurements obtained in our group. When analyzing images scanned by scanning probe microscopy, lots of aspects should be taken into account due to the complex nature of SPMs measurement. Then main aim of our experiments and simulation is to get a better understanding of surface measurements based on SPM.

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