

Use of biaxially oriented polypropylene film for evaluating and cleaning contaminated atomic force microscopy probe tips: An application to blind tip reconstruction

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(Received 28 June 2002; accepted 30 July 2002)

An atomic force microscopy (AFM) image of a surface is basically a convolution of the probe tip geometry and the surface features; it is important to know this tip effect to ensure that an image truly reflects the surface features. We have found that a biaxially oriented polypropylene (BOPP) film is suitable for checking tip performance and for cleaning contaminated tips, thus making it possible to collect images of the *same* area of a BOPP film surface before and after the tip was cleaned. Therefore, the difference between the two different images is solely due to the contamination of the tip. We took advantage of our ability to collect AFM images of the same area using the same tip, in one instance, contaminated and, in the other, after being cleaned. First we used blind reconstruction on the image collected using the contaminated tip. Blind tip reconstruction allows one to extract the geometry of the tip from a given image. Once we had estimated the geometry of the contaminated tip, we used it to simulate the tip effect using the image collected using the cleaned tip. By comparing the simulation result with the image collected using the contaminated tip we showed that the blind reconstruction routine works well. Prior to this, there was no *de facto* method for testing blind reconstruction algorithms. © 2002 American Institute of Physics.

[DOI: 10.1063/1.1510554]

I. INTRODUCTION

An atomic force microscopy (AFM) image of a surface is constructed through the detection of an interaction between the tip apex and the surface features. The interaction, whether it be a contact force, an oscillation amplitude or others, is the feedback signal used to adjust the proximity of the tip and the surface features. Because of this imaging mechanism, an AFM image is, in practice, a convolution of the tip geometry and the surface features. Based on the actual geometry, the tip apex or the surface feature, whichever is sharper, acts as the effective probe. In practice, there could be a large-sized contaminant on the tip apex, making sharper surface features the effective probe. Therefore, images collected using a contaminated or damaged tip can be dominated by the geometry of the AFM tip itself (i.e., self-imaging of the tip) if the surface features are sharper than the tip. Interpretation of such images can easily be misleading if the tip effect is not taken into account. To ensure that the tip is “good” enough for imaging a surface, one needs reference samples that have known surface features, suitable for checking the tip performance. Using dynamic force mode AFM,^{1–4} we have previously demonstrated that a biaxially oriented polypropylene (BOPP) film is suitable for this task.⁵ The BOPP film has the suitable morphological features^{5–10} for evaluating a tip: imaging the nanometer-sized fiberlike network structures is a simple criterion for checking the tip performance.⁵ The BOPP film also has the superior surface

properties of a lower Young’s modulus (1–3 GPa)¹¹ than that of a silicon (132–190 GPa)^{12–14} tip and a low surface energy (~ 30 mJ/m²)^{15–17} (or high hydrophobicity) enabling us to evaluate the tip in a nondestructive manner. Because of these surface properties, the tip will neither be damaged nor contaminated in the process of evaluation. Also intriguing about the use of the BOPP film is that the polymer film can be used to clean a contaminated tip. We demonstrate that contaminants of two different materials can be removed from the tip apex, by simply pushing the contaminated tip several times into the polymer film, up to 50 nm deep.

Considerable effort has been expended to mathematically extract the geometry of the tip based solely on an algorithm derived from a given image.^{18–29} This method is known as blind reconstruction. This methodology is based on an assumption that protrusions in the AFM image represent the self-image of the tip, which is equivalent to the statement that sharper features on the sample surface act as the probe to image the AFM tip. This method has proven useful and successful in estimating tip geometry from an existing image, when appropriate samples were chosen (i.e., some surface features on the sample are sharper than the tip). Once the tip geometry is known, the tip effect may be subtracted from the original image through the mathematical operation of erosion, also known as deconvolution.^{18,23–26} Dilation is another mathematical operation, which adds a tip effect to an existing AFM image by “scanning” the known tip across the “surface” of the image.^{23,24,27,28} This transformation appears useful in simulating tip effect to a given image, because the mechanism of AFM can be regarded as a dilation between

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the tip geometry and surface features of the sample.

In this article, we apply the blind reconstruction method to estimate the geometry of the contaminated tip from images collected on a BOPP film. The advantage of using BOPP film is that images of the *same* area on a BOPP film can be collected with the same tip before and after cleaning. This guarantees that the difference between the two images is solely caused by the contaminant. Therefore, we can estimate the geometry of the contaminated tip from the image it collects and then use the estimated tip geometry to dilate the image collected using the clean tip. We show that this dilation operation transforms the image collected using the clean tip to one resembling the image collected using the contaminated tip. Therefore, such images are necessary and unique to demonstrate how well the blind reconstruction method performs, which is otherwise believed to be difficult to test.²⁸

We also compare the eroded (deconvoluted) image generated by blind reconstruction of the image collected using the estimated contaminated tip geometry with the image collected using the clean tip. Although this erosion operation results in a “sharper” image, it does not necessarily guarantee a “true” image for the sample surface. The reason is simple: when the tip is much larger than the surface features it scans, the resultant image would be dominated by the geometry of the tip. If this is the case, then the information about the surface features would have been lost. Therefore, one should be cautious of the results of erosion (deconvolution). It is clear that a reference sample, such as BOPP film, should be used to check the tip performance so that one can eliminate, or at least reduce, the possible tip effect in an AFM image.

II. MATERIALS AND METHODS

A thermally extruded, biaxially oriented isotactic polypropylene film was used in this study. The BOPP film was produced from a homopolymer resin (molecular weight $M_w = 1.9 \times 10^5$, polydispersity = 6.0). The base resin contains 500–1000 ppm each of an inorganic acid scavenger and a high-molecular-weight phenolic antioxidant. The BOPP film was produced on a tenter frame film line and quenched at 45 °C prior to orientation. The machine direction draw ratio was 5.2:1 and the transverse direction draw ratio was 9:1. The film was converted in a direction identical to the machine direction.

A silicon cantilever with nominal geometric dimensions of 125 μm long, 35 μm wide, and 4.0 μm thick was used in this study. The nominal spring constant of the cantilever was ~ 40 N/m. The tip was ~ 15 μm high with a nominal tip apex radius of 20 nm. The dynamic force mode AFM (TopoMetrix's Explorer) was employed for the experiments. The cantilever was oscillated at its resonant frequency (~ 325 kHz) with an oscillation amplitude of ~ 40 nm in free space. The amplitude of the oscillation decreases as the tip is brought sufficiently close to the surface so that the tip “feels” attractive and repulsive forces. The cantilever was eventually set at a certain distance from the sample surface where the oscillation amplitude was reduced to one half (50 %) of the observed amplitude in free space. The AFM image

obtained at this set point of the amplitude, with a scan rate of 5 $\mu\text{m/s}$, consisted of 500 lines with 500 pixel points per line.

AFM tips were purposely contaminated by scanning a UV/ozone-treated and subsequently water-washed BOPP film. The BOPP was exposed to ozone in the presence of UV light for 15 min and then washed with water for 15 min. There likely remains on the surface oxidized materials, which are represented by nonwater soluble medium molecular weight polymer chains.^{30,31} A tiny amount of this oxidized materials was easily transferred to the tip apex in the course of scanning. During scanning, one could judge the contamination of the tip by observing a sudden change in the image quality. Such a contaminated tip was thus used to image an untreated BOPP film. We found that the tip could be cleaned by pushing it several times into the untreated BOPP film,⁵ making it possible to image the same area on the BOPP film before and after the contaminated tip was cleaned. To demonstrate the efficiency of the cleaning procedure, we collected images of the same area before and after cleaning.

In order to determine whether other contaminants would be removed by this cleaning procedure, we selected a sample of octadecylphosphonic acid (OPA) films prepared on a silicon substrate³² to contaminate the tip. Freshly prepared OPA films on a silicon substrate were found to contaminate AFM tips quite easily. It was found that AFM tips contaminated by OPA are cleaned by pushing the tip into the BOPP film and the cleaning can be immediately confirmed by imaging the BOPP film.

Commercial software (SPIP, Metrology Image ApS, Denmark) was used to estimate the tip geometry using its “tip characterization module,” in which the blind reconstruction algorithm^{24,27} is implemented. The algorithm is based on the fact that features on the sample surface that are sharper than the tip actually act as a probe to image the tip. Although this algorithm does not necessarily result in the “real” geometry of the tip, it does give the upper bound of the tip shape that the surface features can image. The advantage of this method is that it only uses the given image to deduce the tip geometry.

The given image can be transformed mathematically by “scanning” a known tip across the image “surface.” This dilation operation simulates the addition of a tip effect to the image. In this article, an image collected using the contaminated tip was used to estimate the geometry of the contaminated tip, which was then used to dilate the image collected using the cleaned tip.

III. RESULTS AND DISCUSSION

Shown in Fig. 1 are images collected using the same tip when it was (a) fresh, (b) contaminated, and (c) cleaned within the *same* area on an untreated BOPP film. The fiber-like network structure of the BOPP is clearly shown in Fig. 1(a). After this, the tip was purposely contaminated by scanning an UV/ozone-treated-water-washed BOPP film, on which there are oxidized materials.^{30,31} When the tip was

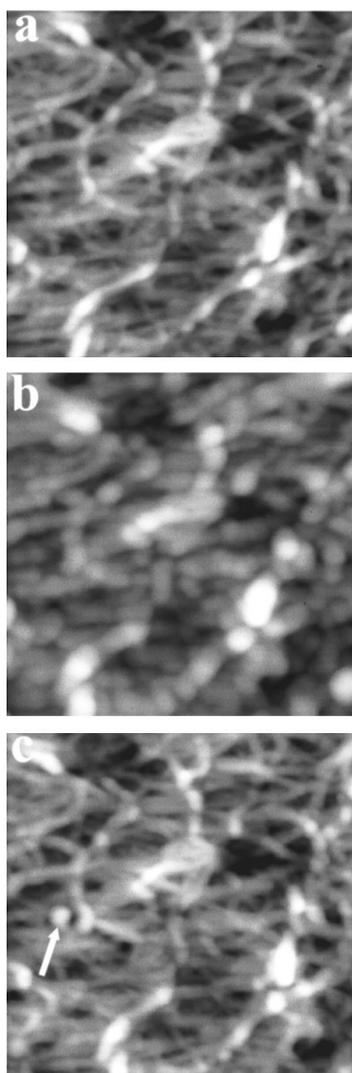


FIG. 1. AFM images of a BOPP film in the same area before (a) and after (b) the tip was contaminated by scanning a treated polymer surface on which there are oxidized materials, as well as (c) after the contaminated tip was cleaned by pushing it into the film. The inset arrow in (c) indicates the deposited contaminant on the film due to the cleaning procedure. The scan area is $1 \mu\text{m}$ square area and the gray scale for the three images is 30 nm.

contaminated, the fibrous features are seen in Fig. 1(b) as dotlike, indicating the image was dominated by the geometry of the contaminated tip itself.

After the contaminated tip was cleaned by pushing it ~ 50 nm into the scan area of the BOPP film for several times, an image reflecting the “true” surface features was obtained once again, as shown in Fig. 1(c). It is clear that the images shown in Figs. 1(a) and 1(c) are similar to each other, indicating the contaminated tip was cleaned. As shown by the arrow in Fig. 1(c), a newly appeared mound indicates that the contaminant was deposited on the film when the contaminated tip was pushed into the film.

It appears that the residual oxidized materials on the UV/ozone-treated-water-washed sample surface were transferred to the tip apex during scanning. We found that this simple method of contaminating an AFM tip is reproducible. The contaminated tip can be immediately checked by scanning an untreated BOPP film. An intriguing aspect of using the BOPP

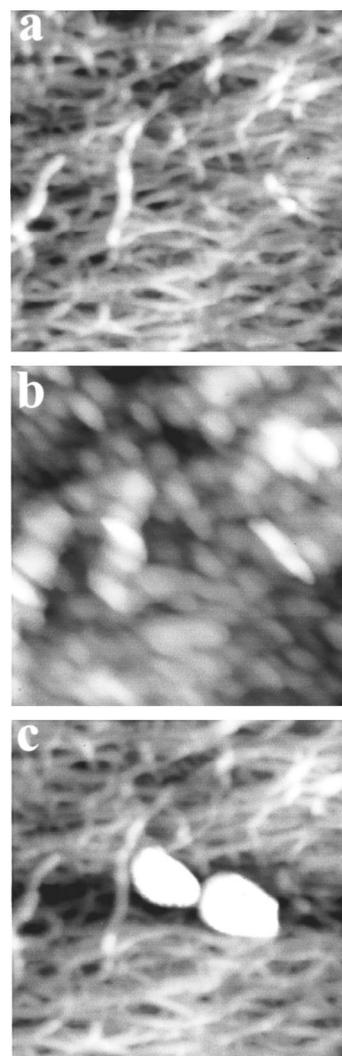


FIG. 2. AFM images of a BOPP film in the same area before (a) and after (b) the tip was contaminated by scanning a sample of OPA prepared on silicon substrate, as well as (c) after the contaminated tip was cleaned by pushing it into the film. Seen in (c) are materials deposited from the contaminated tip after the cleaning procedure. The scan area is $1 \mu\text{m}$ square area and the gray scale for (a), (b), and (c) is 30, 22, and 37 nm, respectively.

film to check tip performance is that the contaminated tip can be cleaned by being simply pushed into the BOPP film. Because the polymer film is flexible and soft compared with the silicon tip, the tip is not damaged. It is also important that the BOPP film be hydrophobic, as this prevents the tip from being contaminated during the evaluation process.

In order to determine if contaminants other than the oxidized materials are also removable, we contaminated the tip with discrete organic moieties. A molecular film of OPA was formed by spin coating its ethanolic solution onto a silicon substrate.³² Such a film was found to contaminate the AFM tip easily, as evidenced by the fact that the AFM image deteriorated rapidly during one scan. The experimental results are shown in Fig. 2. When the tip was fresh, distinct surface features of the BOPP film were imaged as shown in Fig. 2(a). After the tip was contaminated by OPA, rice-like features are seen in Fig. 2(b), indicating that the tip was severely contaminated. The contaminated tip was then pushed ~ 50 nm into the scan area of the BOPP film several times; and the

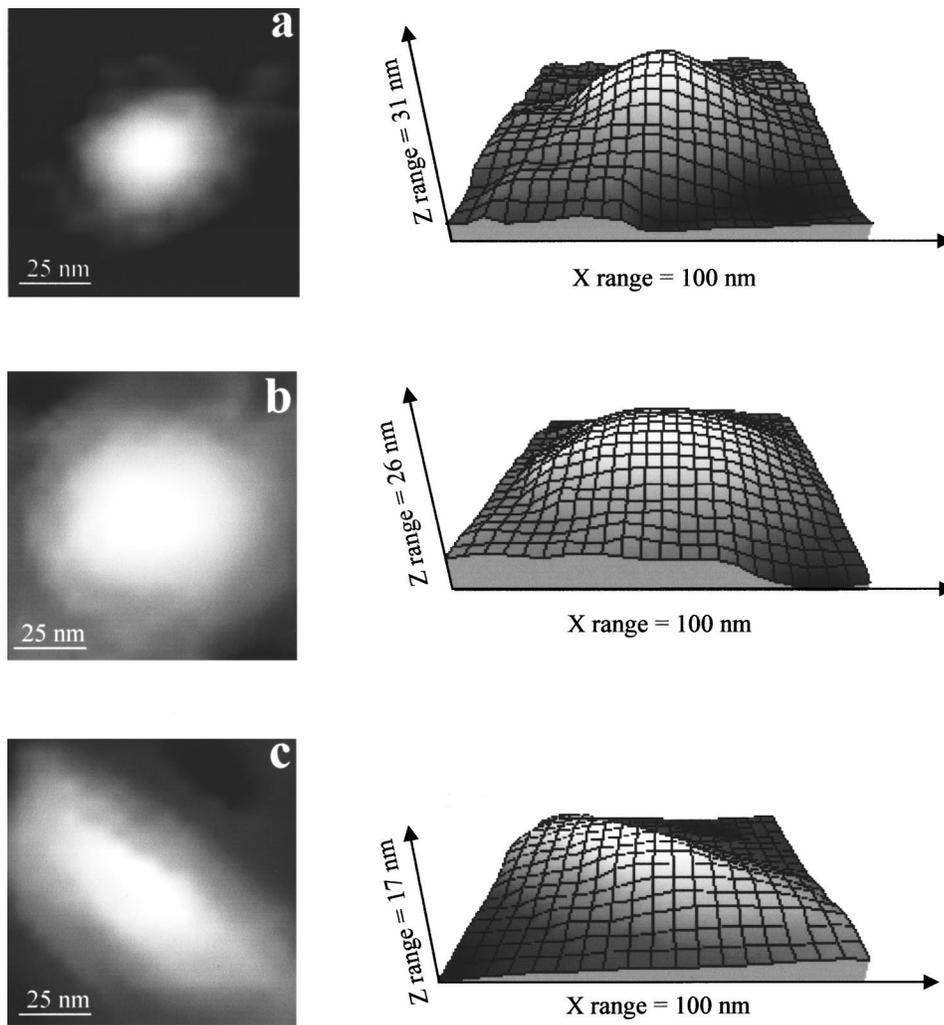


FIG. 3. Tip geometries estimated by blind reconstruction of images shown in Figs. 1(a), 1(b), and 2(b) are shown in (a), (b), and (c), representing a clean tip, an oxidized materials-contaminated tip, and an OPA-contaminated tip, respectively. The gray scales for the left column images are 31, 26, and 17 nm for (a), (b), and (c), respectively. Shown on the right-hand side column are three-dimensional images having the same scale factor for both the lateral and height dimensions.

“true” surface features of the BOPP film appear again after the tip was cleaned, as clearly shown in Fig. 2(c). Because all images in Fig. 2 were collected within the same area of the BOPP film, the newly appeared feature seen in Fig. 2(c) indicates that the cleaning procedure had removed the organic materials from the contaminated tip to the BOPP film.

When the tip is cleaned by pushing it into the BOPP film, the contaminants on the tip apex can be either pushed to the sidewall of the tip or deposited on the BOPP film. Regardless of the cleaning mechanism, the contaminants are removed from the tip apex. In a previous publication we suggested the former mechanism since no deposit was found.⁵ The experimental results shown in Figs. 1(c) and 2(c) show that the contaminants can be deposited on the BOPP film surface.

It is clear that BOPP is suitable for verifying tip performance and cleaning contaminated tips. The fiberlike network structure of the BOPP film is not revealed by a contaminated tip. Using the contaminated tip resulted in an image where the geometry of the contaminant on the tip apex dominates the image. Because the image collected using the contaminated or clean tip of the same area of the BOPP film represents the tip effect or the “true” surface features, respectively, simulations of the tip effect using an estimated tip

geometry would be instructive for the application of the blind reconstruction method.

We first estimate the tip geometry for both the clean and contaminated tips. Because the images collected using the fresh tip and the tip after cleaning show almost the same quality, we will use the image collected using the fresh tip, thereafter called clean tip, as the base image to simulate the tip effect. In practice, one may only have the images collected using the contaminated tip and the cleaned tip to work with. Shown in Figs. 3(a) and 3(b) are both topographic images and three-dimensional representations of the tip geometries for the clean and the contaminated tips estimated from images in Figs. 1(a) and 1(b). These tip geometries were generated through blind reconstruction. We also confirmed that the geometry of the cleaned tip estimated from Fig. 1(c) shows a similar result to that shown in Fig. 3(a). The three-dimensional images have the same scale factor for the height and the lateral dimensions. It is clear that the contaminated tip has a much larger radius than the clean one: The clean one has a radius of ~ 20 nm and the contaminated one ~ 50 nm. Shown in Fig. 3(c) is the tip geometry estimated from Fig. 2(b), where the tip was severely contaminated by OPA. For this type of contaminated tip, it is obvious that the tip cannot be expressed with a radius. It is worth noting that

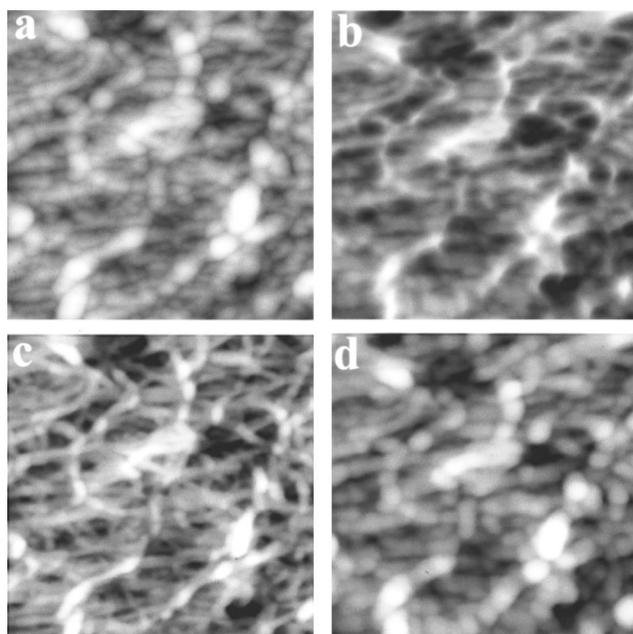


FIG. 4. Dilation operation on Fig. 1(a) using the tip geometry in Fig. 3(b) results in the image shown in (a). Erosion operation (deconvolution) on Fig. 1(b) using the tip geometry in Fig. 3(b) results in the image shown in (b). For comparative purposes, images shown in (c) and (d) repeat Figs. 1(a) and 1(b), respectively.

due to the limited height of the BOPP film surface features, only the top of the tip can be estimated. Therefore, the flat outlying areas seen in Fig. 3, especially for the clean tip shown in Fig. 3(a), should be regarded as artifacts.

Comparison of tip geometry from the blind reconstruction method and from scanning electron microscopy (SEM) images has been made.²⁸ In this article we provide a simpler way to test blind reconstruction: comparison of AFM images collected in the same area of the BOPP by clean and contaminated tips. If the estimation of the contaminated tip geometry is reasonable, then one expects to be able to use the estimated tip geometry to dilate the image collected using the clean tip to obtain an image resembling one collected using the contaminated tip. Conversely, we can also determine if the deconvolution works by eroding the image collected with the contaminated tip using the estimated tip geometry to see whether the result resembles the image collected using the clean tip.

The results for dilation and erosion (deconvolution) from Figs. 1(a) and 1(b), respectively, are shown in Figs. 4(a) and 4(b). For comparative purposes, Figs. 4(c) and 4(d) repeat Figs. 1(a) and 1(b), respectively. Figure 4(a) is obtained by dilating Fig. 4(c) with the tip geometry shown in Fig. 3(b), which has a radius of ~ 50 nm and can be considered being slightly contaminated. Figure 4(a) shows that the dilation operation adds the tip effect to the image in Fig. 4(c), in a way analogous to that as if one would obtain an image by “scanning” the contaminated tip across the “surface” represented by Fig. 4(c). One can see that Fig. 4(a) resembles Fig. 4(d), which is the image collected using the contaminated tip. This simulation suggests that the estimation of the tip geometry from Fig. 4(d) is reasonably good. It is clear that with the help of the blind reconstruction method, one can use

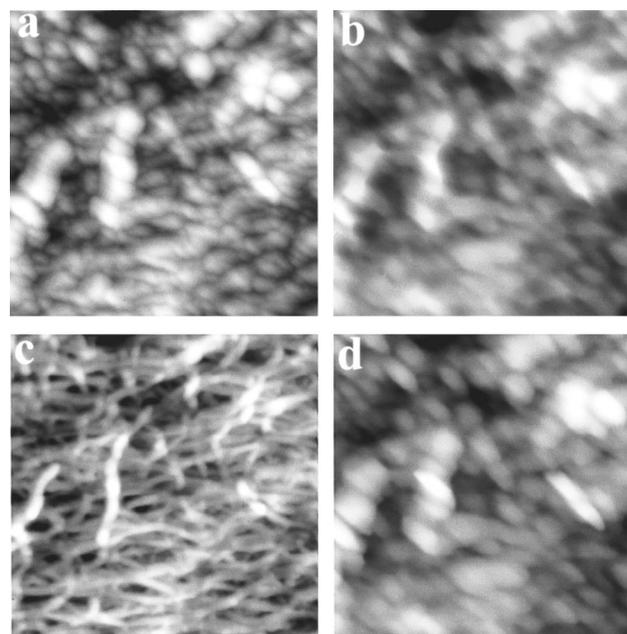


FIG. 5. Dilation operation on Fig. 2(a) using the tip geometry in Fig. 3(c) results in the image shown in (a). Erosion operation (deconvolution) on Fig. 2(b) using the tip geometry in Fig. 3(c) results in the image shown in (b). For comparative purposes, images shown in (c) and (d) repeat Figs. 2(a) and 2(b), respectively.

the BOPP film not only to check the tip performance and to clean a contaminated tip, but also to access the geometry of the contaminated tip.

Figure 4(b) is obtained by eroding the image in Fig. 4(d) collected using the contaminated tip with the estimated tip geometry shown in Fig. 3(b). The erosion operation tries to subtract the tip effect from the image. One can see that Fig. 4(b) becomes sharper than Fig. 4(d) in terms of reflecting the “true” surface features of the BOPP film, especially for the thicker fibers. However, for the thinner fibers, the erosion operation does not transform them to resemble those shown in Fig. 4(c), which is the image collected using the clean tip.

When a tip is severely contaminated, the dilation operation also appears to work well to add the tip effect to an image. Shown in Figs. 5(a) and 5(b) are dilated and eroded images from Figs. 5(c) and 5(d), respectively, using the OPA-contaminated tip geometry estimated from Fig. 5(d), as shown in Fig. 3(c). For comparative purposes, Figs. 5(c) and 5(d) repeat Figs. 2(a) and 2(b), respectively. By comparing Figs. 5(a) and 5(d), one can see that the dilation operation works reasonably good in adding the tip effect to Fig. 5(c). Figure 5(a) appears to have more detailed features than Fig. 5(d), which can be explained by the fact that the former is a transformation of Fig. 5(c) which has finer structures, while Fig. 5(d) was collected using the contaminated tip.

The erosion operation results in an image, Fig. 5(b), which is not even similar to Fig. 5(c), but basically resembles the image in Fig. 5(d) with finer “surface features.” This complete failure of the erosion operation to restore the “true” surface features for Fig. 5(d) reflects the fact that the tip effect dominates the image and the information about the “true” surface features has been lost.

From the results described above, one can see that the

blind reconstruction method is reliable, because one can add tip [Figs. 3(b) or 3(c)] effect to an image [Figs. 4(c) or 5(c)] by dilating it [Figs. 4(a) or 5(a)] to resemble the image [Figs. 4(d) or 5(d)] collected using the respective contaminated tip. In other words, from the image collected using the clean tip one can get, by the dilation operation, an image resembling the image collected using the contaminated tip. It is clearly demonstrated in Figs. 4 and 5 that the dilation operation actually mimics the AFM imaging mechanism. When the AFM tip scans a surface, the sharper of the two acts as the probe to image the other. Therefore, the dilation operation, adding the tip effect to an existing image, works well to the extent that the surface features in the image are sharper than the tip. The results shown in Figs. 4 and 5 are a proof of the effectiveness of the blind reconstruction method for estimating the tip geometry.

Because an AFM image is a convolution of the surface features and the tip geometry, if neither of them is known, there is no way to know, on an unknown sample, if the image is dominated by the surface features or the tip effect. When the tip is much sharper than the surface features, it will collect an image reflecting the “true” surface features. This is the reason why a reference sample is essential to check the tip performance. It is important to note that a tip could be easily contaminated or damaged depending on the chemical and mechanical properties of the sample surface.³³ Using electron microscopes one can evaluate the outlines of the tip shape from specific directions,^{28,33} but it is difficult, if not impossible, to capture the three-dimensional geometry of the tip. In combination with blind reconstruction, using BOPP film to check the tip performance provides a simple and effective protocol to test the estimation of the tip geometry of a contaminated tip. One can do this by comparing the image collected using the contaminated tip with the image generated by dilating the image collected using the clean tip.

On the other hand, when the tip is much larger than the surface features, using such a tip to scan the surface will result in an image that is merely a reflection of the geometry of the tip apex itself. In this case, it is evident that the information about the surface features is physically lost. Therefore, the erosion operation will not lead to the recovery of the “true” surface features, though the mathematic operation may result in an image which is likely closer to the “true” surface features. The degree of the recovery by the erosion operation is dependent on how severely the tip is contaminated. One can imagine that different surface features can have similar images if a large tip is used: they are dominated by the tip effect.

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