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Full Length Article

# AFM tip-sample convolution effects for cylinder protrusions

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ABSTRACT

A thorough understanding about the AFM tip geometry dependent artifacts and tip-sample convolution effect is essential for reliable AFM topographic characterization and dimensional metrology. Using rigid sapphire cylinder protrusions (diameter: 2.25 μm, height: 575 nm) as the model system, a systematic and quantitative study about the imaging artifacts of four types of tips—two different pyramidal tips, one tetrahedral tip and one super sharp whisker tip—is carried out through comparing tip geometry dependent variations in AFM topography of cylinders and constructing the rigid tip-cylinder convolution models. We found that the imaging artifacts and the tip-sample convolution effect are critically related to the actual inclination of the working cantilever, the tip geometry, and the obstructive contacts between the working tip's planes/edges and the cylinder. Artifact-free images can only be obtained provided that all planes and edges of the working tip are steeper than the cylinder sidewalls. The findings reported here will contribute to reliable AFM characterization of surface features of micron or hundreds of nanometers in height that are frequently met in semiconductor, biology and materials fields.



## 1. Introduction

Accurate topographic characterization and dimensional metrology of surface features are essential in semiconductor, biology and materials fields, etc. For instance, fabrication of semiconductor devices demands accurately and precisely controlled critical dimensions (e.g. height, width, pitch, sidewall angle, roughness, and so on) of structures [1]. Understanding cellular behaviors such as cell spreading, activation and differentiation requires accurate topographic characterization of characteristic cell features [2,3]. Shape-controlled synthesis of nanocrystals also requires accurate topographic characterization because many physical and chemical properties of nanocrystals are sensitive to crystal size and shape [4,5].

Among various techniques for topographic characterization and dimensional metrology of surface features, optical microscopy (OM), scanning electron microscope (SEM), transmission electron microscope (TEM) and atomic force microscope (AFM) have been routinely used. Although OM is fast and non-destructive, it suffers from low spatial resolution (hundreds of nanometers) [1]. SEM, with nanometer spatial resolution, large view-field depth, and convenient centimeter scale survey capability, has been frequently used for straightforward determination of in-plane dimensions (e.g. lengths, diameters and pitch) of surface features from the top view [6]. However, other important profile details, say the heights and sidewall angles of surface features, can only be obtained through observation of tilted/fractured samples or even sectional samples prepared by the complicated focused ion beam method—sometimes an intimidatingly time-consuming task [7–9]. TEM, a high resolution characterization technique, is capable of nanoscale and atomic-scale structural analyses [10]; however, TEM fails to deliver topographic information. In contrast, AFM is a local probe-scanning technique with nanoscale in-plane resolution and sub-angström vertical resolution; AFM offers outstanding non-destructive topographic characterization and three-dimensional (3D) metrology capabilities [11].

A serious problem of AFM characterization is the frequent occurrence of annoying artifacts [11,12]. An artifact is defined as any fake feature in an image which is not present as the real surface feature. Serious artifacts hinder one to obtain true surface topography and derive reliable dimensional information. Clearly it is essential to recognize artifacts in AFM images. During AFM scanning, the tip-sample convolution effect is caused by the geometrical interactions between the tip and surface features being imaged [12–14]. The tip-sample convolution effect is one of the main causes of AFM

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artifacts owing to the finite sharpness and characteristic geometry of tips [15]. For example, this convolution effect can account for many lateral broadening of surface protrusions [13,16].

The tip sharpness/geometry affects AFM imaging quality depending on the characteristic of sample features. For the flat surface with well separated steps (e.g. step-terrace structures of single-crystal $\alpha$-$Al_2O_3$ [17]) or thin films (e.g. graphene [18]) of only a few nanometers height, the tip sharpness and geometry are not crucial because only the foremost apex of the AFM tip is involved in imaging [12]. On the other hand, for high aspect ratio features of micron and submicron size, e.g. sharp spikes, deep holes, trenches, rods and cylinders, the AFM imaging quality seriously relates with the sharpness and geometry of the tip [12,19]. It has been reported that sharp spikes recorded with blunt tips appear broadened in AFM images [20]. In contrast, deep holes/pits/trenches may appear narrower and shallower because the tip apex fails to touch the steep sidewall and bottom [12]. Many studies have reported that, for rod and cylinder structures with steep sidewalls, tip-induced artifacts appear as the “additional material”, “cliff-like feature” or “shadow” [19,21,22]. For instance, AFM images of rod-shaped bacteria (Escherichia coli K12, height: 800–1000 nm) were consistently found to contain “shadow” artifacts due to the convolution between edges of the pyramidal tip and the bacterium sidewalls [19]. Using carbon nanotube (CNT) tips or super sharp whisker tips, artifacts can be minimized and critical dimension of surface features can be measured more accurately [23,24].

For many conventional and low-cost AFM probe tips, it will be challenging to use to their full capacity, for accurately imaging surface features with high aspect ratio or steep sidewalls, and to choose appropriate tips for the specific imaging task even they are sometimes susceptible to various artifacts [25–28]. However, there are much room left for systematic and quantitative studies about the tip geometry dependent artifacts and tip-sample convolution effect. To achieve this goal, it is essential to construct an appropriate model sample system with carefully specified dimensions and geometries. In this way, tip geometry dependent artifacts and the tip-sample convolution effect can be demonstrated and quantified. In this regard, rigid cylinder protrusions, are advantageous over other geometries like trench [29] or rod [19,30] as the model surface features because cylinders, enjoying both steep sidewalls and circular symmetry, are well suitable to investigate tip geometry dependent artifacts—especially for asymmetrical tips with multiple planes and edges.

In this paper, using rigid sapphire cylinder protrusions as the model system, we carried out a systemic and quantitative investigation about AFM tip geometry dependent artifacts and tip-cylinder convolution effect of four types of tips (two different pyramidal tips, one tetrahedral tip and one super sharp whisker tip) through comparing tip geometry dependent variations in AFM topography of cylinders and constructing the rigid tip-cylinder convolution models. The findings reported in this paper will contribute to gaining more insights into tip geometry dependent artifacts and demystifying the tip-sample convolution effect, as well as improving AFM characterization efficiency.

## 2. Experimental section

### 2.1. Fabrication of sapphire cylinders

Cylinder patterned sapphire substrate (CYPSS) wafers (diameter: 2-inch) were kindly donated by GAPSS OE Technology Co., Ltd (Xuzhou, China). The cylinder arrays were fabricated following standard photolithography process and inductively coupled plasma ($BCl_3/H_2$) etching on the flat *c*-plane (0001) sapphire wafer. Cylinder protrusions were arranged regularly with hexagonal symmetry with the pitch of 3.2 $\mu$m. The CYPSS samples with the size of 10 mm × 10 mm × 0.4 mm for SEM and AFM characterizations were cut from 2-inch wafers. In one piece of the sample, there are millions of cylinder protrusions.

### 2.2. SEM characterization

The morphology of the CYPSS sample, cylinder protrusions and AFM tips was characterized with a field emission SEM (Zeiss Supra 55). The acceleration voltage was set to as low as 3 kV to effectively reduce the charging effect, thus facilitating direct SEM observation without sputtering a conductive layer as required for the routine SEM observation of insulating samples. The working distance was ∼8 mm. All SEM images were recorded with the secondary electron detector.

### 2.3. AFM characterization and analysis

AFM images were acquired using a commercial AFM system (Bruker MultiMode 8) with a calibrated J scanner at ambient conditions (25 °C, 35–40% RH). AFM images were collected with 512 × 512 pixels/frame. Four types of probes were selected to represent a variety of tip geometries commonly used by researchers: PNP-TR probe (NanoWorld AG, Switzerland) with the pyramidal tip of low aspect ratio, PPP-NCHR probe (NanoWorld AG, Switzerland) with the quadrilateral pyramid tip of high aspect ratio, 4XC-NN probe (MikroMasch, Bulgaria) with the tetrahedral tip with steep edges, as well as NSC probe (NT-MDT, Russia) with super sharp “whisker” tip. Each AFM tip was characterized prior to usage. At least two probes of each type were tested to ensure reproducibility. The geometries of four types of probe tips were extracted from SEM images at multiple viewing orientations. Table 1 summarizes the mechanical properties, materials and tip geometries of four types of probes.

Analyses of AFM images were performed with the NanoScope Analysis software. Note that for the height and angle analyses of cylinders in AFM images, the baseline height was leveled against the flat base plane of the substrate. All AFM images were only subjected to the primary first order plane fit correction to remove sample tilt so that potential artifacts induced by other image processing steps were avoided as much as possible.

## 3. Results and discussion

### 3.1. SEM characterization of cylinder protrusions

The morphology and dimensions of sapphire cylinder protrusions are carefully obtained through SEM multiple-view observations for detecting/comparing AFM imaging artifacts of cylinders. Top-view SEM image (Fig. 1a) of the cleaned cylinder patterned sapphire substrate (CYPSS) shows that cylinder protrusions are arranged regularly with hexagonal symmetry with the pitch of 3.13 ± 0.04 $\mu$m. The cylinder is of good circular symmetry and the base diameter is 2.25 ± 0.03 $\mu$m. Then, the fractured CYPSS is tilted vertically for deriving accurate profile dimensions of cylinders. Side-view SEM image (Fig. 1b) reveals clearly that the line profile of the sidewall of cylinders: the profile (red line) of upper zone (named as $CY_0$-zone) is convex with slant angles ranged from 30° to 78°; in contrast, the profile (green line) of lower zone (named as $CY_1$-zone) is straight with the slant angle of 78°. The mean height of cylinders is determined to be 575 ± 4 nm.

### 3.2. AFM tip type I. Pyramidal tip of low aspect ratio

As outlined in the introduction, using conventional pyramidal tips, AFM imaging of cells with steep sidewalls is susceptible to