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Surface chemical analysis — Atomic force microscopy — Guideline for restoration procedure for atomic force microscopy images dilated by finite probe size

Analyse chimique des surfaces — Microscopie à force atomique — Lignes directrices relatives au mode opératoire de restauration des images de microscopie à force atomique dilatées par la taille finie de la sonde

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Foreword

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 9, *Scanning probe microscopy*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Atomic force microscope (AFM) is a method for imaging surfaces by mechanically scanning their surface contours, in which the deflection of a sharp probe tip sensing the surface forces, mounted on a compliant cantilever, is monitored. AFM belongs to a family of scanning probe microscope (SPM) and is of increasing importance for the characterization of materials surfaces at the nanoscale. Therefore, precise and quantitative measurement of three-dimensional (3D) surface topography at the nanoscale by AFM is highly demanded by researchers and engineers in the various fields of academia and industry. One of the imaging artefacts of AFM topography measurements is caused by the finite size and shape at the apex of an AFM probe used for the scanning. Such a dilation effect due to the probe shape can cause a significant error in the precise analysis of 3D surface morphology. Especially for the critical dimension (CD) analysis of fine devices at the nanoscale, there is a need for probe-shape artefact to be corrected in a reproducible and quantitative way. Thus, the demand for the establishment of an international standard on the guideline for a reliable restoration procedure of dilated AFM images is high.

This document describes a quantitative procedure for the restoration of AFM height images dilated by finite probe size and shape. It includes the quantitative characterization of AFM probe apex in use and the restoration of AFM topography images using the actual probe shape.

Surface chemical analysis — Atomic force microscopy — Guideline for restoration procedure for atomic force microscopy images dilated by finite probe size

1 Scope

This document describes a procedure for the quantitative characterization of the probe tip of an atomic force microscope (AFM) probe and a restoration of AFM topography images dilated by finite probe size. The three-dimensional shape of the probe apex is extracted by image reconstruction using suitable reference materials. This document is applicable to the reconstruction of AFM topography images of solid material surfaces.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11775, *Surface chemical analysis — Scanning-probe microscopy — Determination of cantilever normal spring constants*

ISO 11952, *Surface chemical analysis — Scanning-probe microscopy — Determination of geometric quantities using SPM: Calibration of measuring systems*

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-2 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
dilation
one of the two basic operators of mathematical morphology, whose basic effect on a binary image is to gradually enlarge the boundaries of regions of foreground pixels

Note 1 to entry: The dilation (⊕) of a set *A* by a set *B* is defined as follows:

$$A \oplus B = \bigcup_{b \in B} (A + b)$$

Note 2 to entry: By dilation, areas of foreground pixels grow in size while holes within those regions become smaller.

3.2
erosion
one of the basic operators of mathematical morphology, whose basic effect on a binary image is to erode away the boundaries of regions of foreground pixels

Note 1 to entry: The erosion (⊖) of a set *A* by a set *B* is defined as follows:

$$A - B = \bigcup_{b \in B} (A - b)$$

Note 2 to entry: By erosion, areas of foreground pixels shrink in size, and holes within those areas become larger.

3.3
mathematical morphology
theory and technique for the analysis and processing of geometrical structures, based on set theory, lattice theory, topology, and random functions, which is commonly applied to digital images

Note 1 to entry: See Reference [4]

3.4
probe
structure at or near the end or apex if the cantilever designed to carry the *probe tip* (3.8)

[SOURCE: ISO 18115-2:2021, 5.109]

3.5
probe characterizer
tip characterizer
structure designed to allow extraction of the *probe tip* (3.8) shape from a scan of the characterizer

3.6
probe shape characteristic
PSC
relationship between the probe profile width and the probe profile length for a given probe projected onto a defined plane

[SOURCE: ISO 13095:2014, 3.10]

3.7
probe shape function
PSF
matrix representing a three-dimensional shape of a *probe tip* (3.8) used for AFM imaging

3.8
probe tip
tip
probe apex
structure at the extremity of a *probe* (3.4), the apex of which senses the surface

[SOURCE: ISO 18115-2:2021, 5.120]

3.9
reconstruction
estimate of the sample's (or tip's) surface topography determined by removing from the image the effect of the tip's (or sample's) shape and other measurement artefacts

[SOURCE: ISO 18115-2:2021, 5.132]

4 Symbols (and abbreviated terms)

The symbols and abbreviated terms are:

AFM Atomic force microscopy

I A function describing the measured topography image of a sample by AFM; $z(x,y)$

S A function representing the true topography image of a sample; $s(x,y)$

T A function representing the shape by a probe apex of AFM; $t(x,y)$

S_r A function representing the reconstructed topography image of a sample by AFM; $s_r(x,y)$

P A function describing the reflection of the probe shape T through the origin; $p(x,y) = -t(-x,-y)$

P_r A function representing the reconstructed image of the reflected-tip shape; $p_r(x,y)$

R Tip radius

$\oplus$ A symbol representing a dilation operation in mathematical morphology

$\odot$ A symbol representing an erosion operation in mathematical morphology

5 Mathematical morphology modelling

For quantitative morphology imaging of nano-objects, it should be noted that significant distortion in imaging may occur if the surface of a nano-object has large corrugation compared to the size and shape of the probe tip[5]. When the sample surface is relatively flat on the atomistic scale, it is suitable to express the influence of the probe shape on the AFM topography imaging by the convolution integral with the probe shape. On the other hand, when the unevenness or roughness of the sample surface is somewhat larger than the atomic size, it is more appropriate to express the AFM imaging by dilation which is one of the fundamental operators of mathematical morphology, where the location on the top apex of a probe tip approaching closest to or making a point contact with the sample surface is most important. Interaction from any other area which does not make any contact or near-contact with the sample surface is not considered. The operation of dilation is expressed by Formula (1):

$$z(x,y) = \max\{\, s(x',y') - t(x'-x, y'-y) \,\} = \max\{\, s(x',y') + p(x-x', y-y') \,\} \quad (1)$$

Here, $\boldsymbol{I} = \boldsymbol{z}(\boldsymbol{x}, \boldsymbol{y})$ is a function describing the measured image of the top surface of the sample, while $\boldsymbol{S} = \boldsymbol{s}(\boldsymbol{x}, \boldsymbol{y})$ is a function representing the true surface morphology. Meanwhile probe shape function $\boldsymbol{T} = \boldsymbol{t}(\boldsymbol{x}, \boldsymbol{y})$ represents the probe shape describing the surface of the probe tip, where the coordinates of the topmost point of the tip are set as the origin. Finally, $\boldsymbol{P} = \boldsymbol{p}(\boldsymbol{x}, \boldsymbol{y})$ means $-t(-x, -y)$, describing the reflection of the probe shape $\boldsymbol{T}$ through the origin, which may refer to *reflected tip*. The relationship between the sample $\boldsymbol{S}$, tip $\boldsymbol{T}$, image $\boldsymbol{I}$, and the reflected tip $\boldsymbol{P}$ can be written using the dilation operation $\oplus$ as shown in Formula (2):

$$I = S \oplus (-T) = S \oplus P \quad (2)$$

On the contrary, the reconstruction of the actual surface morphology from the measured AFM image and the probe shape function is expressed as an erosion operation ($\ominus$) in the concept of mathematical morphology. The reconstructed surface morphology $\boldsymbol{S}_r = s_r(x, y)$ is described by Formulae (3) and (4):

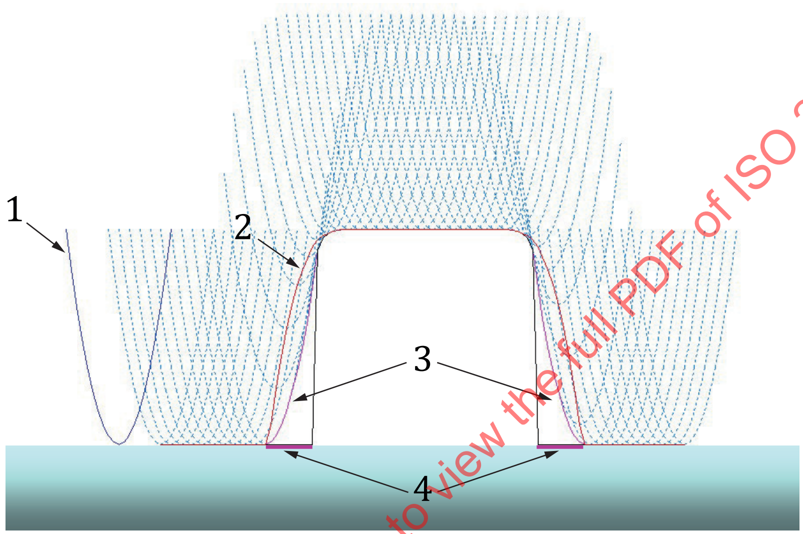
$$s_r(x,y) = \min\{\, z(x',y') - p(x'-x, y'-y) \,\} = \min\{\, z(x',y') + t(x-x', y-y') \,\} \quad (3)$$

$$S_r = I \ominus P \quad (4)$$

It should be noted that $\boldsymbol{S}_r$ is the least upper bound on the actual surface, and not necessarily equal to $\boldsymbol{S}$. Since $\boldsymbol{I} = \boldsymbol{S} \oplus \boldsymbol{P}$,

$$S_r \supseteq S \quad (5)$$

By the processing of the dilation and erosion operations, while scanning the AFM probe of the finite size, the measured and reconstructed AFM images can be expressed as shown in Figure 1. As described by Formula (5), there exist non-reconstructable regions or hole regions where the tip of the probe cannot reach due to its finite size. Therefore, $\boldsymbol{S}_r$ is the best reconstruction because it is the surface of the deepest penetration with the probe tip.



Key

1 tip shape
2 AFM imaged surface
3 reconstructed surface
4 hole (non-reconstructable) region

Figure 1 — Mathematical morphology modelling for AFM topography imaging (dilation processing) and image reconstruction (erosion processing)

6 Procedure of restoration of AFM topography images

6.1 General

This clause describes the general procedure for the recovery of dilated AFM topography images. Firstly, the AFM topography images of the reference nanostructures with given shapes, dispersed on flat substrates, are acquired. Then, the probe shape function of the apex of an AFM probe in use is determined by the numerical calculation. Then, by erosion operation using the probe shape function, the most probable surface morphology shall be extracted from the observed AFM topography image of an unknown actual sample (see examples in Annex A).

6.2 Calibration of measuring systems

Calibration of the measuring systems used for AFM topography imaging shall be carried out using certified standards with proper intervals. For example, the calibration of X-, Y- and Z-axes piezoelectric scanners are primary prerequisite for the precise topography imaging by AFM. The calibration of the lateral scan axes, or X- and Y-axes of the measuring system, shall be done with one-dimensional (1D) or two-dimensional (2D) lateral standards. Calibration of Z-axis of AFM shall be done by using a set of step height standards in accordance with ISO 11952.

The deflection sensitivity and normal spring constant of a cantilever probe used for the measuring system shall be calibrated properly in accordance with ISO 11775.

6.3 Environment requirements

It is recommended that the measurement be performed in controlled and stable conditions with the temperature stable within ±1 °C or better to minimize the drift of the measuring system. It is also recommended to carefully measure the drift rate of the measuring system in accordance with ISO 11039.

6.4 Extraction of probe tip shape using certified reference materials

It is possible to reconstruct the probe tip shape of a force sensor from AFM topography images of certified reference materials called a probe characterizer (or a tip characterizer), whose actual surface morphology, $\boldsymbol{I}$ is well characterized. Using erosion operation, reconstructed reflected-tip shape $\boldsymbol{P}_{\mathbf{r}}$ shall be extracted using Formula (6):

$$P_{\mathrm{r}} = I \ominus S \tag{6}$$

Analogously to the reconstruction of a specimen surface, $\boldsymbol{P}_{\mathbf{r}}$ is the best reconstruction and an outer bound on the probe tip shape. From the known surface morphology of a probe characterizer, the unevenness area whose abruptness is like or larger than that of the probe tip shall be taken as the operation area.

An example of certified reference materials with known shape is standard nanosphere particles dispersed on flat substrates[6]. The particle size standards are provided with certain certificates of calibration and traceability by photon correlation spectroscopy (PCS), transmission electron microscopy (TEM), or other techniques.

6.5 Estimation of probe tip shape by blind reconstruction

Blind reconstruction is a type of reconstruction based on the theory and technique of mathematical morphology. The use of blind reconstruction methodology allows characterizing the 3D shape of a probe tip used for AFM topography imaging[7]. Blind reconstruction takes advantage of the self-imaging effect that is present in AFM topography imaging on relatively flat surfaces with exceptionally fine protrusions at the nanoscale. All the nanoscale protrusions observed in AFM topography imaging may be regarded as reflected-tip images, each broadened in different ways by the different underlying surface features. The reflected-tip shape $\boldsymbol{P}$ and the image $\boldsymbol{I}$ satisfy Formula (7):

$$(I \ominus P) \oplus P = I \tag{7}$$

The blind reconstruction method is an iterative solution of the above Formula (7) for a set $\boldsymbol{P}_{\mathbf{r}}$, which is an outer bound on the true shape of the reflected-tip. The solution shall be obtained iteratively by Formula (8):

$$P_{i+1} = \bigcap_{x \in I} \left(\left((I - x) \oplus P_i^{'}(x) \right) \cap P_i \right) \tag{8}$$

Here the object P_i' is defined by Formula (9):

$$P_i^{'}(x) = P_i \cap (x - l) \tag{9}$$

The above iteration process shall be continued until its convergence. Then the reconstructed reflected-tip shape P_r shall be extracted as follows:

$$P_r = \lim_{i \to \infty} P_i \tag{10}$$

The obtained shape P_r by blind reconstruction by Formula (10) is an outer bound of the actual shape of the reflected probe tip as shown by the following Formula (11):

$$P_r \supseteq P \tag{11}$$

By applying the blind reconstruction method, the largest shape of the reflected-tip can be extracted from an AFM topographic image, which shall be consistent with the all features included in the image. Therefore, the existence of any measurement artefacts in a topographic image, such as electric noise, mechanical or acoustic vibration, thermal drift and scanner nonlinearity, can be potential sources of errors in the reconstruction process. Especially random spike noise can form sharp features in a topographic image, which may cause significant errors in the reconstructed images.

6.6 Reference materials

There are two types of reference materials for the extraction of probe tip shapes. One is nano-objects with known shapes such as periodic or non-periodic sharp-edged line-space structures with sharp edges, isolated wedges, sharp nano-edges with steep slopes, suspended carbon nanotubes and standard nanosphere particles. The probe tip characterizers with sharp edge-like features shall be used to estimate the line profiles of a probe tip along the specific directions, such as the X-axis or Y-axis. Standard nanospheres shall be used for 3D reconstruction of probe shape. The other is nanostructured material with sharply peaked features which makes it ideal for reverse imaging the tip of an AFM probe in use by the blind reconstruction method.

6.7 Probe shape characteristic and curvature radius

The probe shape characteristic (PSC) and the radius of curvature of an AFM probe apex, *R*, shall be evaluated from the probe shape function measured by the above-mentioned procedure. For a curved apex, the value of *R* equals the radius of the circular arc which best approximates the curve at the topmost point of the probe apex.

6.8 Validity test for topography image restoration

Properly reconstructed topography image using the AFM probe shape function shall conserve the height information. Using the extracted probe shape, the erosion process shall be applied to a pre-processed topography image, so that the reconstructed height image is extracted. Since a properly reconstructed image shall keep the Z-axis values of locally highest and lowest points, the heights of nano-objects shall be conserved after the erosion process. As the validity test of the reconstruction operation, the conservation of height characteristics shall be examined (see example in Annex B).

Annex A
(informative)

Example studies

A.1 General procedures

The general procedure for the recovery of a dilated AFM image using a probe shape function is shown in Figure A.1. The X-, Y-, and Z-axes of the AFM measuring system are properly calibrated using certified standards with an appropriate calibration interval. The AFM measurement is started when the thermal drift is regarded as negligibly small. Normally waiting for more than one hour is recommended after the sample set to the AFM system. The measurement mode can be contact, intermittent contact, or noncontact mode. Input signals to be recorded are Z-axis height (topography) and error signal. The scan parameters such as setpoint, gain, and scan rate are optimized to minimize the error signal and noise. Monitoring of the forward (trace) and backward (retrace) scan lines of both topography and error signals is useful for the optimization of scan parameters. Firstly, an AFM topography image of the reference material with a given nanostructured feature, or a tip-characterizer, is acquired. Then an AFM topography image of a target sample is acquired. Both raw AFM height images are processed by proper plain correction, and/or noise reduction if necessary. Then, the probe shape function for the AFM probe in use is extracted by the blind reconstruction method. By erosion operation using the probe shape function, the most probable surface morphology can be extracted from the pre-processed AFM topography image of an unknown actual sample. See Figure A.1.

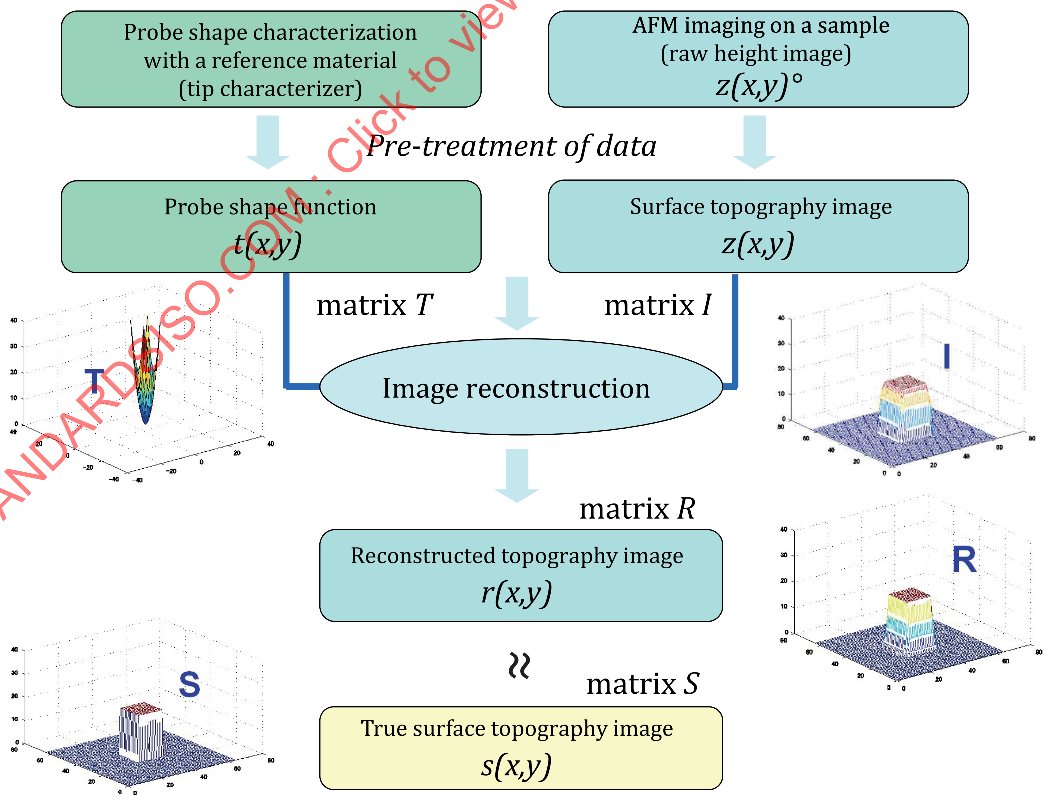
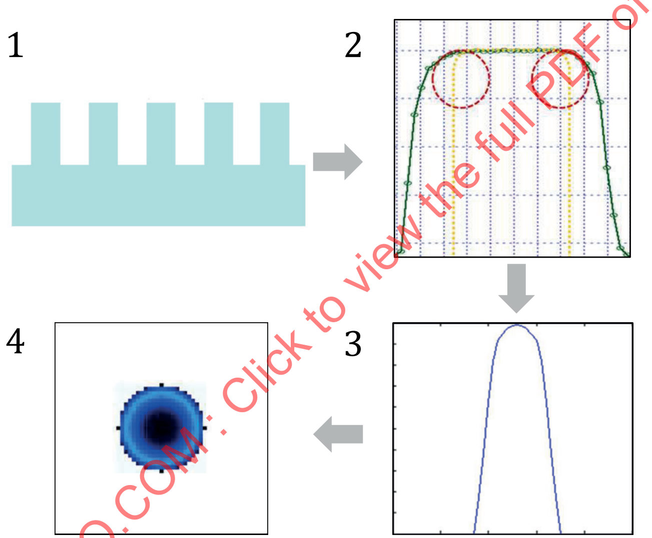


Figure A.1 — Restoration procedure for AFM topography images dilated by probe tip shape

A.2 Type of reference materials

The reference materials for probe tip characterization and extraction of probe shape functions contain either of two kinds of nanoscale structures. One is nano-objects with artificially designed structures such as periodic or non-periodic sharply-edged line-space structures, which are used to measure the line profiles of a probe tip along specific directions, such as the X- or Y-axes. The other is the nanostructured flat surfaces with sharply peaked nanostructures, which are used for reversely imaging the tip of AFM probes using the blind reconstruction method.

The reference materials of the first category consist of nanometer-scale lines and spaces structures with constant pitches. A typical example is shown in Figure A.2 (image 1) which is commercially available (e.g. AS100P-D, NTT-AT, Japan). Different pitches ranging from ~100 nm to ~500 nm, and line widths ranging from ~50 nm to ~250 nm fabricated on a flat Si (110) substrate using an anisotropic etching technique are available. Each line-space nanostructure has a rectangular cross-sectional profile with sharp edges. The sidewalls are vertical (90°) to the substrate plane. A typical line profile from an AFM topography image of the line-and-space nanostructure is shown in Figure A.2 (image 2). The line profile of a probe tip projected along the specific direction can be extracted. It is possible to extract the approximated 3-D probe shape function by assuming coaxial symmetry as shown in image 3 of Figure A.2. By line-profiling along several different directions of the AFM probe tip, more precise extraction of 3D probe shape function can be realized (see image 4 of Figure A.2).



Key

1 line-and-space reference material

2 AFM height profile along X-axis

3 extraction of probe line profile

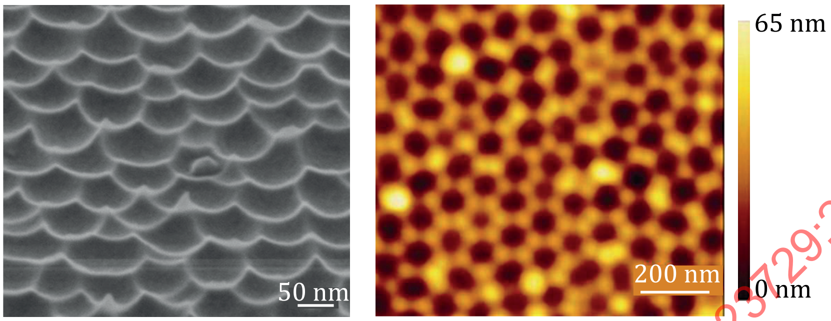
4 extraction of probe shape function

Figure A.2 — Typical reference material consisting of nanometer-scale lines and spaces structures with constant pitches

To evaluate the shape of a probe tip with a nanoscale curvature radius, sharply-peaked nanostructures fabricated on a very flat substrate are used. It is desirable that the sharp peaks or spikes with a nanoscale curvature radius are dispersed uniformly on the flat surface with appropriate separations. One such reference material is a thin film of porous alumina (aluminium oxide) consisting of hexagonal-like hollow cells with sharp spikes, formed on an aluminium substrate by anodic oxidation, which is commercially available. The typical curvature radius of the nano-spikes formed at the intersections of the partitions is approximately 2 nm.

A scanning helium ion microscope (SHIM) image of sharply-peaked porous alumina nanostructures, and an AFM topography image observed with a normal AFM probe of which typical curvature radius is 8 nm are shown in Figure A.3. Since the typical curvature radius of the nanoscale spikes is as small as 2 nm, AFM topographical features at the spikes taken with a normal probe represent the inversed image of the probe tip. It should be noted that the imaging mode of AFM can be intermittent-contact mode, non-contact mode, or contact mode with small forces, to avoid possible breaking-off of the spikes. Observation by hard cantilevers in contact mode with large forces may break off the nano-spikes.

1 2



Key

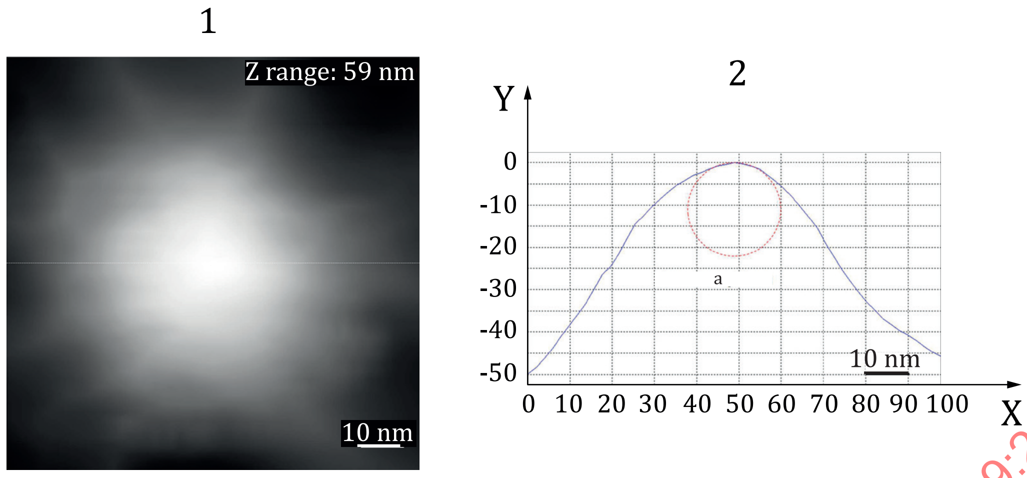
1 a high-resolution SHIM image observed with a 14-degrees tilt angle

2 an AFM topography image observed with a normal AFM probe

Figure A.3 — Typical reference material of a nanostructured flat surface with sharply-peaked porous alumina nanostructures

A.3 Extraction of probe tip shape by blind reconstruction

The shape of a probe tip in use shall be estimated by using blind reconstruction methodology. Several data analysis software for the extraction of probe tip shape by blind reconstruction are available commercially or non-commercially. Firstly, the raw topography image data are pre-processed by proper plain correction and/or noise reduction if necessary. A typical image of the reflected probe tip shape extracted by blind reconstruction is shown in Figure A.4, image 1. Proper choice of pixel size for the extraction of probe tip shape is important. It should be selected so that the properly reconstructed region of a probe tip shape can be covered by the pixel size. Line-profiles along certain directions including the topmost apex of a probe can be extracted from the probe shape function. A typical line profile along X-axis direction is shown in Figure A.4, image 2.



Key

1 a reflected probe tip shape

2 a line-profile along x-axis

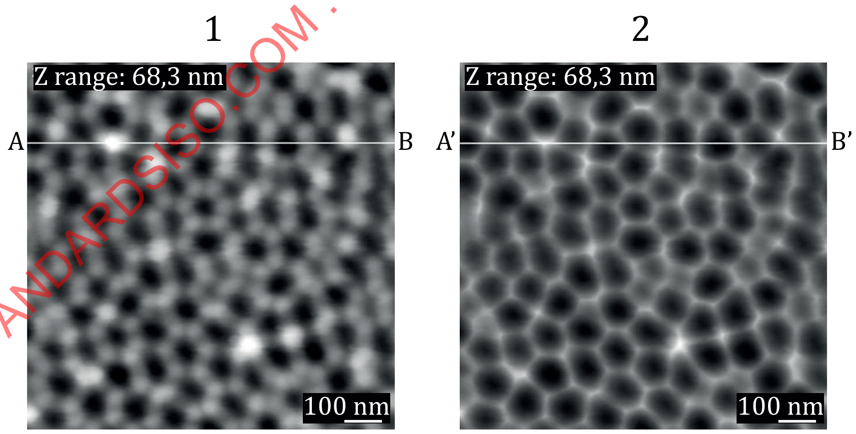
3 Z-axis, in nm

4 X-axis, in nm

Figure A.4 — Typical example of the extraction of probe shape by blind reconstruction

A.4 Reconstruction of height image

Using the extracted probe shape function, imaging artefacts caused by the tip geometry can be removed. After proper pre-processing and extraction of the probe shape, the erosion process is applied to the AFM height image, so that the reconstructed height image can be extracted. A typical example of the image reconstruction process applied on an AFM height imaging of a porous alumina surface is shown in Figure A.5.



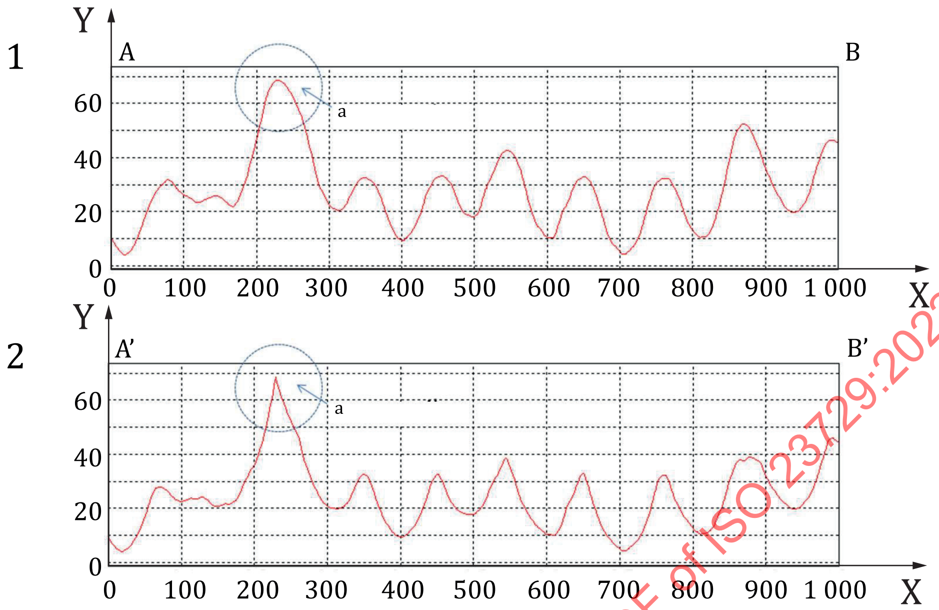
Key

1 an AFM height image (raw data)

2 a reconstructed image after erosion

Figure A.5 — Typical example of the reconstruction of an AFM height image on porous alumina

Properly reconstructed images conserve the height information. For example, in the case of porous alumina, heights of nano-spikes should be conserved after erosion. The validity of the reconstruction can be examined as follows: i) identify a local topmost point of a single-peaked nano-spike in raw and reconstructed images; ii) extract the line-profiles including the topmost points of nano-spikes for the raw and reconstructed images as shown in Figure A.6; iii) confirm that the heights of the nanospike before and after erosion is the same.



Key

X X-axis, in nm

Y height, in nm

1 height image

2 reconstructed image

A start point of the line profile shown in image 1 of Figure A.5

B end point of the line profile shown in image 1 of Figure A.5

A’ start point of the line profile shown in image 2 of Figure A.5

B’ end point of the line profile shown in image 2 of Figure A.5

[a] Nano spike.

NOTE The height of the topmost point of the same nanospike in raw (1) and reconstructed (2) images is conserved.

Figure A.6 — Horizontal line profiles including local topmost point of a nanospike

