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Surface chemical analysis — Scanning-probe microscopy — Definition and calibration of the lateral resolution of a near-field optical microscope

Analyse chimique des surfaces — Microscopie à sonde à balayage — Définition et étalonnage de la résolution latérale d'un microscope optique en champ proche

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Foreword

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ISO 27911 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 9, *Scanning probe microscopy*.

Introduction

The near-field scanning optical microscope (NSOM or SNOM) is a form of scanning-probe microscope (SPM) that uses an optical source but achieves, through the use of the near field, a spatial resolution significantly superior to that defined by the Abbe diffraction limit. NSOM instruments are mainly either apertured, when the resolution is governed by the aperture size, or apertureless, when the resolution is more complex. In apertureless NSOMs, a very sharp scannable tip is used to probe the surface, or molecules on the surface, through local scattering of light from the test specimen surface or the tip apex. The spatial resolution for scattering NSOMs is a complex phenomenon and is less easily characterized in terms of an instrumental property, and so this International Standard focuses on, and is limited to, the lateral spatial resolution of apertured NSOM instruments.

Although the term spatial resolution has a clear meaning, it is often characterized in different ways. In this International Standard, one convenient and effective method for measuring the spatial resolution of an apertured NSOM instrument is presented, suitable for use by non-expert operators.

Surface chemical analysis — Scanning-probe microscopy — Definition and calibration of the lateral resolution of a near-field optical microscope

1 Scope

This International Standard describes a method for determining the spatial (lateral) resolution of an apertured near-field scanning optical microscope (NSOM) by imaging an object with a size much smaller than the expected resolution. It is applicable to aperture-type NSOMs operated in the transmission, reflection, collection or illumination/collection mode.

2 Normative references

The following referenced documents are indispensable for the application 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 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.

3.1
far field
electromagnetic field at a distance from a light source significantly greater than the wavelength of the light

3.2
point spread function
response of an imaging system to a point source or point object

4 Symbols and abbreviated terms

APD avalanche photodiode

FWHM full width at half maximum

NA numerical aperture

PMT photomultiplier tube

PSF point spread function

QD quantum dot

δ FWHM of the PSF of the NSOM, i.e. the lateral resolution of the NSOM instrument

5 General information

5.1 Background information

The NSOM is a form of scanning-probe microscope with a probe that has an optical aperture that can illuminate, and/or collect the light from, the surface of a test specimen in the distance within a fraction of the wavelength of the light, this region being called the near field. A two-dimensional NSOM image consists of pixels that contain optical information (normally, the light intensity or photon counts obtained at each pixel position). For an apertured NSOM, an open optical aperture of subwavelength diameter is located at the apex of a sharp probe, and light is emitted and/or collected by it. The NSOM probe is scanned over the specimen surface in the near field. Because the aperture is so close to the surface, the size of the spot illuminated on the surface (or from which light is collected) is determined not by the light wavelength but mostly by the aperture size. Since the aperture size can be made as small as a few tens of nanometres, spatial resolution far better than the theoretical resolution limit of the conventional far-field optical microscope can be achieved by an NSOM. The spatial resolution achievable by reducing the size of the aperture is limited by the skin depth of the metal coating of the NSOM probe, which defines the aperture, and by the fact that optical throughput decreases rapidly with decreasing aperture diameter, going beyond the limits of practical detection.

5.2 Types of NSOM operation

5.2.1 General

Below we describe different modes of NSOM operation. This International Standard is concerned with apertured NSOMs operated in the illumination, collection or illumination/collection mode. Control of the gap between the specimen and the probe is achieved by shear-force detection using optical or electrical transduction for a straight-fibre probe, and by cantilever deflection using optical transduction for a bent or cantilevered probe.

5.2.2 Classification

5.2.2.1 NSOMs can be classified on the basis of how the light is transmitted to/collected from the specimen:

a) Illumination mode: The light emanates from the aperture and is collected with a lens in the far field.

b) Collection mode: The specimen is illuminated by light from a far-field source or excited to emit light by another means and light is detected (collected) using the NSOM aperture.

c) Illumination/collection mode: The NSOM aperture is used for both illumination and collection.

5.2.2.2 NSOMs can also be classified on the basis of the position of the collection optics with respect to the illumination optics:

a) Reflection mode: Both illumination and collection are carried out on the same side of the specimen in any of the three modes defined above.

b) Transmission mode: The collection and the illumination optics are located on opposite sides of the specimen. In most cases, including the reflection mode a) above, a high-NA lens is used for high collection efficiency.

5.2.3 Control of gap between probe and specimen surface

The gap between the NSOM probe and the surface is typically controlled in one of two ways, depending on the type of probe:

a) Shear-force detection type: The NSOM probe is attached to a piezo tube or tuning fork and vibrated laterally to the surface with an amplitude of a few nanometres. Feedback is provided to keep the amplitude, phase or frequency of the vibration constant. For homogeneous surfaces, this would provide a constant gap; for most materials with a structured surface, the situation is more complicated, but often the constant-gap approximation holds.

b) Cantilever type: The NSOM probe is cantilevered so that various ways of controlling atomic-force microscope tips can be used. In particular, the deflection of a laser beam off the end of the cantilever can be used to sense the surface topography and maintain a constant gap distance.

NOTE Care is required to ensure the correct way of doing this.[1]

5.3 Methods of measuring the lateral resolution of an NSOM

The spatial resolution of an NSOM is mainly determined by the size of the aperture probe, its distance from the surface, and the contrast mechanism. In addition, the nature of the specimen, pixilation and signal-to-noise issues can affect resolution. Therefore the spatial resolution of the NSOM can be defined only for a particular instrument and a particular specimen and, accordingly, any claim of spatial resolution should specify the details of the experimental conditions[2], such as the properties of the specimen, the type of imaging mode, the height regulation mechanism, the type of NSOM probe and other factors that could affect the measurement of the spatial resolution.

Measurement of the spatial resolution of an NSOM instrument has been estimated by several methods, including measurement of the size of the smallest feature appearing in the NSOM image[3], imaging small objects in a fluorescent mode[4] to [7] and imaging a specimen having an abrupt optical-contrast edge[8].

The method chosen here is the imaging of a small object. It is based on the concept of the PSF[9], which is a critical concept that determines the spatial resolution of an optical microscope. In using this method, the following limitations of the method should be noted:

a) It is recognized that, with NSOMs, the resolution is a result of near-field interactions between a specimen and a probe. The intensity profile in the near field of an aperture, even for the simplest possible case of an aperture in an infinite plane, and in the absence of interactions with a specimen, is not a simple Gaussian one[10]. In general, the field shape varies with the aperture shape, the condition of the outer metal coating and the polarization of the input light, etc.

b) Topography-induced artefacts that appear in the optical images produced by NSOMs are sometimes mistaken for optical contrast[11]. If the optical contrast of the specimen is low compared to the background signal, which is not specific to the optical characteristics of the specimen, the contrast appearing in the NSOM optical image could originate totally or in part from topographic change in the specimen surface. To minimize the influence of topographic change on the NSOM optical image, this International Standard describes fluorescence mode NSOM, and the topographic heights of the objects to be imaged are limited to one-tenth of the expected value of the lateral resolution.

5.4 Parameters that affect the lateral resolution

5.4.1 General

The measurement of lateral resolution can depend upon a number of experimental factors, including the physical properties of the NSOM aperture, the specimen, the contrast mode, the feedback conditions, the relative positions of the source and detector, and the instrumental noise. Improperly formed images suffering from the effects of pixilation can also affect resolution, but do not present any fundamental limitations and are easily eliminated.

5.4.2 Aperture size of NSOM probe

The aperture size of the NSOM probe is of primary importance. A smaller aperture size results in a better resolution. There is a trade-off between aperture size and the signal-to-noise ratio: smaller apertures have a lower throughput, which results in a poorer signal-to-noise ratio. Apertures are produced in different ways. Coating the outside of the probe with a metal is the most popular method.

5.4.3 Condition of outer metal coating

For metal-coated probes, it is crucial that they do not have pinholes in the coating, as pinholes greatly compromise both resolution and contrast. The method of formation of the aperture in the metal coating is also important. For example, if it was by focused ion beam milling[12] or by the pounding method[13], it will produce a rather blunt-ended probe shape but a well-defined aperture, whilst the shadowing method[14] will usually result in a sharper probe end but the aperture boundary might not be so clearly defined. In general, coatings that are smooth and homogeneous also offer electromagnetic fields that are more predictable and better confined and therefore provide better imaging qualities.

5.4.4 Vertical size of the specimen

This is particularly important because topographic change in the specimen surface has been known to contribute to the optical contrast of the NSOM image. This phenomenon, caused by an effective crosstalk between the topographic-signal channel and the optical-signal channel, results in topographic artefacts in the optical image[11]. Any change in the topography signal might induce modulation in the optical signal, so that one ends up with similar features in both the optical image and the topography image, even though no corresponding optical contrast exists in the specimen. This effect is more often observed if there is a strong background signal in the optical-signal channel that can be perturbed by the modulation of the topography signal. Therefore, transmission or reflection images that have an intrinsic background optical signal throughout the whole area of the image are especially subject to this topographic artefact. The effect is also more likely when a blunt probe is used to image a surface with a rapidly varying topography. In this case, the variation (with changing tip-specimen separation) of the optical coupling between the NSOM probe and the specimen surface results in topographic artefacts.

In order to avoid the above artefacts, one should eliminate both topographic changes in the specimen and the optical background signal as far as possible. A test specimen that is topography-free but still provides optical contrast is a good way of eliminating topography changes in the specimen surface, even though the preparation of such a specimen could be technically challenging.[3] The optical background could be effectively eliminated by using fluorescence imaging where Stokes-shifted luminescence is detected while incident laser light is blocked. This way, one can isolate the true optical signal from the background signal that can be affected by the crosstalk, because the background signal and the fluorescence signal are spectrally separated.[4] However, even using fluorescence imaging, if the whole or part of the surface to be imaged has a background fluorescence signal, any optical contrast riding on this background signal could be due to the topographic artefacts. Therefore, isolated fluorescent objects of nano-size on a non-luminescent substrate would be desirable as the test specimen.[4]

5.4.5 Lateral size of the specimen

This is also important in measuring the lateral resolution of an NSOM. When imaging a small object, the recorded NSOM image is the convolution of the object and the PSF of the NSOM. Therefore, the apparent size of the object is larger than the PSF of the NSOM. The PSF of the NSOM cannot be regarded as a simple Gaussian or Lorentzian distribution, but rather as having a complicated structure that depends on the aperture shape, the condition of the outer metal coating, the input polarization, the light-coupling conditions, etc., and it is virtually impossible to de-convolve the true PSF of the NSOM from the observed profile of the small object in the NSOM image. Therefore, it is recommended that the effect of the object size be minimized by using objects as small as possible, so that one can regard the FWHM of the observed profile of the small object, to a reasonable approximation, as the FWHM of the NSOM PSF (see 6.2).

5.4.6 Polarization of illumination light

Different polarizations can produce different NSOM images of the same specimen in otherwise the same experimental conditions; therefore, one should take care in selecting the input polarization of the incident light. The polarization strongly affects the field distribution around the aperture of the NSOM probe both when the aperture of the NSOM probe is isotropic and when it is not. This will also affect the PSF of the NSOM instrument and thus the estimation of the lateral resolution. In cases where the NSOM image shows an apparent influence on the measurement of the lateral resolution due to a certain polarization status, it is suggested that the polarization of input light be scrambled.

There also exist certain polarization-sensitive specimens with anisotropic absorbance or fluorescence[16] which will give a different optical contrast in the resultant NSOM images, depending on the polarization of the input light. Therefore, these kinds of specimen should be avoided as test specimens for NSOMs.

5.4.7 Gap between the probe and the specimen surface

The gap between the NSOM probe and the specimen surface has a direct influence on the resultant NSOM image. The near-field distribution on the specimen surface is dependent upon the distance from the specimen surface and, as the distance between the probe and the surface increases, the spatial resolution and the optical intensity rapidly decrease. Therefore, the gap distance shall be regulated so that it has no influence on the NSOM image. In theory, the smallest gap would produce the best resolution; however, an optimum gap distance should be chosen for the stable operation of the NSOM.

5.4.8 Collection optics

For good contrast, it is generally best to have high-NA collection optics. Better contrast often translates into better resolution through better signal-to-noise (S/N) ratios, enabling the use of smaller apertures. A confocal pinhole in front of the photodetector could enhance the contrast by excluding background light.

5.4.9 Photodetector

The light from, or collected by, the NSOM probe is relatively weak, sometimes only a few picowatts. The use of highly sensitive photodetectors, such as PMTs or APDs, is recommended to improve the S/N ratio and help the use of smaller-apertured NSOM probes.

5.4.10 Contrast mode

Depending on what contrast mode is used, one might obtain a different value of the lateral resolution even using the same specimen. In this International Standard, only the fluorescence mode is described to determine the lateral resolution of an NSOM.

6 Measurement of lateral resolution by imaging a very small object

6.1 Background information

Imaging of a very small object to estimate the lateral resolution has the advantage that one can obtain a two-dimensional profile containing information on the PSF of the NSOM from a single NSOM image (see Figure 1). The lateral resolution can be defined as the FWHM of the PSF. Because the finite size of the small object contributes to the observed size of the object in the NSOM image, one should understand that the observed image of the object is the convolution of the PSF and the spatial distribution of the small object. There is a wide range of specimens that can be used in this method, and nanoparticles such as nano-scale polymer beads[4], QDs[5] and single molecules[6],[7] have been used. To minimize the possible effect of topography-induced contrast, Stokes-shifted photoluminescence imaging should be used to reduce the background optical signal, and the particle size shall be small compared to the expected resolution of the NSOM (see 6.2).