Polarization contrast in photon scanning tunnelling microscopy combined with atomic force microscopy

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Summary

Photon scanning tunnelling microscopy combined with atomic force microscopy allows simultaneous acquisition and direct comparison of optical and topographical images, both with a lateral resolution of about 30 nm, far beyond the optical diffraction limit. The probe consists of a modified microfabricated silicon nitride tip mounted on a cantilever, commercially available for atomic force microscopy. The lateral resolution is further improved using 'supertips', by depositing a small needle on the silicon nitride tip. The combined microscopic technique is applied to thin films of indium tin oxide because of the small grain size and high surface flatness, providing high-resolution optical contrast and limited far-field scattering contribution. Polarization contrast is shown in experiments both changing the polarization of the incident and detected light. Approach curves, both measuring the optical signal and force interaction, show a difference in the optical coupling between p- and s-polarized incident light. p-Polarized light always provides optical contrast more correlated to topography than s-polarized light, both for incident and detected light, in agreement with theoretical models.

Introduction

Scanning near-field optical microscopy (SNOM) combines the merits of traditional optical microscopy, e.g. absorption contrast, polarization contrast and phase contrast, with a resolution unlimited by diffraction. In SNOM a subwavelength probe is scanned in close proximity over a sample surface to generate or detect the optical near-field. Thus, high-resolution information about the local optical

characteristics of the sample surface is acquired (Pohl, 1990; Courjon & Bainier, 1994). Polarization contrast in SNOM using aperture probes has been shown by Betzig *et al.* (1991, 1992), Vaez-Iravani & Toledo-Crow (1993), Moers *et al.* (1994a) and Jalocha & van Hulst (1995).

In photon scanning tunnelling microscopy (PSTM) the 'illumination' of the sample is realized by placing the transparent sample on a glass–air transition at which total internal reflection (TIR) occurs. Beyond the sample–air transition perpendicular to the boundary an exponentially decaying evanescent field arises. A dielectric, optically transparent probe brought in close proximity ($\ll \lambda$) to the sample surface frustrates the total reflection (FTIR). Consequently, the evanescent wave is converted into a radiating wave, i.e. light is coupled into the probe. As such, photon tunnelling can be regarded as the optical analogue of the quantum mechanical electron tunnelling through a potential barrier. The coupling of light to the probe depends on the distance, topography and dielectric properties of the sample; therefore, also with the PSTM technique it is possible to obtain information about the optical properties of the sample surface. Conventionally, the probe is a sharpened fibre tip (Reddick *et al.*, 1990; van Hulst *et al.*, 1992b). However, unless a reliable feedback mechanism is used to control the tip–sample distance, a fibre tip is damaged quite easily. The exponentially decaying evanescent field theoretically allows an optical feedback mechanism, but is not reliable in practice because of the contribution of scattered waves (van Hulst *et al.*, 1992a). Using fibre tips in PSTM polarization contrast has been shown by van Hulst *et al.* (1991, 1992a) and Courjon *et al.* (1992). Depending on the direction of the incident polarization, different images of the sample and a different behaviour of the optical signal as a function of tip–sample distance (coupling-distance curves) have been observed. In

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general, p-polarized light tends to display a larger contrast than s-polarized light. A disadvantage of this technique is that the topography and the optical image of the sample cannot be viewed simultaneously, which would increase the understanding of the optical contrast mechanism in the PSTM images enormously. As such, a feedback mechanism based on a force interaction between probe and sample can be used. Commercially fabricated atomic force microscope (AFM) cantilevered tips are suitable for this purpose. Neither tip nor sample are damaged upon contact, because the optically transparent tip is mounted on a cantilever with low spring constant. The deflection of the tip is measured by the optical beam deflection method (Putman *et al.*, 1992a). Thus, in this form of PSTM-AFM, the optical signal and topography of the sample are recorded simultaneously, leading to promising results (as demonstrated by van Hulst *et al.*, 1993a, b; Baida *et al.*, 1993; Moers *et al.*, 1994b). Recently, Borgonjen (1994) has shown the potential of the combined PSTM-AFM technique in an application to map the field distribution in waveguide structures. With the PSTM-AFM set-up, polarization contrast has not yet been observed. Theoretical approaches (Girard & Bouju, 1992; van Labeke & Barchiesi, 1992; Barchiesi, 1993) have shown that p-polarized light displays optical contrast more correlated to the topography than s-polarized light. A verification of these predictions is only possible with a combined optical force microscope. In this paper we present the first investigation of polarization contrast using an integrated photon scanning tunnelling and atomic force microscope.

Theory

At the transition from a medium 1, with refractive index n_1 , to an optically less dense medium 2, with refractive index n_2 , TIR can occur when the angle of incidence θ is larger than the critical angle $\theta_c = \arcsin(n_2/n_1)$ (Reitz *et al.*, 1979). The incident light is totally reflected, which means that no energy is transferred to the second medium. However, in the second medium an evanescent, oscillating electric field arises. Parallel to the boundary and parallel to the plane of incidence, this electric field varies periodically, with periodicity $p = \lambda/n_1 \sin \theta$. Perpendicular to the boundary the field decays exponentially, with a decay $(1/e)$ -length or penetration depth d_p given by

$$d_p = \frac{\lambda}{2\pi n_2 \sqrt{(\sin \theta / \sin \theta_c)^2 - 1}}. \quad (1)$$

To describe reflection and transmission at the interface the electric field is conventionally divided into two perpendicular components, a p-component parallel and an s-component perpendicular to the plane of incidence. The ratio of the s- and p-components of the electric

Fig. 1. Schematic set-up of the combined photon scanning tunnelling and atomic force microscope using a silicon-nitride probe: tip + cant, cantilevered SiN probe; GB, glass-block; scanner, xyz-piezoelectric positioner; HeNe, laser; L1, L2, L3, lenses; m1, m2, m3, m4, 45°m, mirrors; pol.1, pol.2, polarizers; LD, laserdiode; QD, quadrant detector; BS, beamsplitter; CCD, camera; PMT, photomultiplier tube.

field in the second medium can be described as follows:

$$\frac{E_{2s}}{E_{2p}} = n_1 \sqrt{(\sin^2 \theta)/n_2^2 - (\cos^2 \theta)/n_1^2} \frac{E_{1s}}{E_{1p}}. \quad (2)$$

Note that in the case of TIR, in the second medium the p-polarized field is perpendicular to the boundary and the s-polarized field parallel to the boundary. FTR can quite easily be described (Zhu *et al.*, 1986) when a semi-infinite dielectric medium is brought in close proximity to the boundary where TIR takes place. The penetration depth is equal for both incident polarization directions, but the phase shift that occurs at the boundary is different. Therefore, the coupling-distance curves are different for both polarization directions. At larger distances the shape of both curves approaches exponential behaviour. In close proximity to the surface a deviation from the exponential shape can be seen, i.e. the slope of the coupling-distance curve decreases. Close to the critical angle the distance at which this deviation starts is larger for p-polarization and the optical coupling is stronger for p-polarization than for s-polarization. When the frustration is caused by a subwavelength

probe, the description of FTR is less straightforward. Theoretical calculations for specific situations have been performed (Labani *et al.*, 1990; Salomon *et al.*, 1991; Dereux & Pohl, 1993), but comparison with the actual experimental data remains problematic.

Experimental set-up

The experimental set-up is shown in Fig. 1. The heart of the set-up is the silicon-nitride (SiN) probe with cantilever (tip + cant, Park Scientific Instruments). The sample is mounted on a glass block (GB, BK7, $n = 1.515$), using immersion oil and scanned by means of a commercial *xyz*-positioner (scanner, Photon Control microblock) with piezoelectric elements ($16\ \mu\text{m}$ extension at 150 V) in the three perpendicular directions.

The total set-up consists of three microscopes.

The PSTM

In the glass block, total internal reflection occurs by choosing the angle of incidence (48°) larger than the critical angle of 41.3° . The HeNe laser beam is focused to the position where the TIR takes place via a lens (L1, $f = 100\ \text{mm}$) and adjustable mirrors (m1, m2). The polarization direction of the incident light is controlled with a polarizer (pol.1). In the optical path of the PSTM signal a 'long-working-distance' microscope objective (L3, Nikon, $40\times$, NA 0.5, 10.1 mm working distance) is used to image the backside of the probe on a pinhole (ph, $\phi\ 100\ \mu\text{m}$). Magnification and size of the pinhole are chosen such that only light originating from the backside of the probe reaches the photomultiplier tube (PMT). Additionally, an interference filter (int), bandwidth 10 nm, is used to block all optical signals with wavelength different from the excitation at $\lambda = 633\ \text{nm}$. In front of the PMT a polarizer (pol.2) can be mounted to control the polarization direction of the detected light.

The AFM

The AFM is constructed as an integrated box, in which a cantilever (cant) with tip and a compact optical beam deflection system are built around the microscope objective (L3). In the optical beam deflection system laser diode (LD) radiation is focused via a compact disc-lens (L2) on the backside of the cantilever, from which the laser beam is reflected to a quadrant detector (QD). A filter (f) in front of the quadrant detector suppresses the HeNe light ($\lambda = 633\ \text{nm}$) and allows the laser diode radiation ($\lambda = 780\ \text{nm}$) to pass. Adjustable mirrors (m3, m4) are present for alignment. Using this beam deflection method both deflection and torsion of the cantilever are measured (Meyer & Amer, 1990). The deflection signal is fed back to

the piezoelement controlling the tip-sample distance (*z*-component of *xyz*-positioner), in order to keep the deflection (force) constant. This creates the possibility of measuring in the 'error-mode' (Putman *et al.*, 1992b), in which the feedback loop mainly acts as a high-pass filter which filters out all low spatial frequencies of the surface. Three signals are thus simultaneously acquired: (1) 'error-mode' deflection (high spatial frequencies), (2) *z*-piezo signal (low spatial frequencies) and (3) torsion (frictional forces).

A conventional optical microscope

In the optical path (PSTM) a beam splitter (BS, 50/50) is present to direct half of the light towards a CCD camera, which enables the tip-cantilever position on the sample to be viewed. This is very practical when specific areas of the sample are of particular interest (biological applications). It also facilitates the alignment of the optical path. In Fig. 1 a 45° mirror (45°m) is drawn, which means that the optical path is bent over 90° . Therefore, the optical detection path is horizontal, enabling the installation of the components (PMT, CCD camera, interference filter, pinhole, polarizer, beamsplitter) on a second-level optical table. Thus a practical and stable set-up is created.

All measurements are performed using a standard personal computer (PC-80486) with a data-acquisition card (Advantech PCL818), which supplies the scan voltage and controls the data handling: the three AFM and the PSTM signals are recorded simultaneously.

The definition of the polarization directions of the incident and detected light is again related to the plane of incidence. In Fig. 1 the *p*- and *s*-polarization of both incident and detected light are shown. Note that the *p*-direction of the detected field is not identical to the *p*-direction of the evanescent field.

A thin layer of indium tin oxide (ITO) is used as sample surface to investigate polarization contrast, because it proved to provide sufficient optical contrast (Moers *et al.*, 1994b). ITO (Balzers, Baltracon) is an optically transparent, electrically conductive material, deposited in particles with dimensions of approximately 30–200 nm, on a glass substrate. The average thickness of the layer is approximately 100 nm. The surface flatness (relative to λ) and small grain size cause only very limited optical scattering contribution, making ITO ideal for acquiring high-resolution optical contrast with PSTM.

Experimental results and discussion

Three different kinds of probes based on the standard AFM SiN probe have been used for imaging. The backside of the cantilever of a commercially available AFM probe is coated with a gold layer to improve its reflectivity. The presence of the gold layer influences the optical signal rather unpredictably, making this standard AFM tip not very

suitable for optical measurements. The second kind of probe has the gold layer removed by etching and provides the largest optical contrast; the presented measurements are thus mainly from this kind of probe. The third kind of probe is similar to the second, but with an additional 'supertip' grown on top of the pyramidal SiN tip, using e-beam deposition in a scanning electron microscope (SEM). The tip radius of the supertip is ~ 10 nm. With the supertip the detected optical signal with crossed polarizers approaches the lower limit of detection (~ 1 pW). In this case the optical transmission is partly reduced because, during the production of the supertip in the SEM, rest material is deposited on the backside of the cantilever.

Figures 2 and 3 are 'error-mode' deflection (AFM) images of ITO, which illustrate how the choice of a different tip influences the apparent topographic image of the sample. In Fig. 2 a conventional AFM tip is used, which causes quite large tip artefacts because of the convolution with the pyramidal shape in the tip. In Fig. 3 a supertip is used and the difference is obvious. The ITO particles are displayed with more variety in size and shape. These tip artefacts must be taken into account because similar effects can be expected in the optical images.

Figure 4 shows the polarization contrast as induced by changing the polarization of the incident beam. Both AFM images (Fig. 4a, b) show only little position shift

1 μm

Fig. 2. A $1.5 \times 1.5\text{-}\mu\text{m}^2$ AFM image of an ITO surface, scanned in 'error-mode' with a conventional SiN tip (Park Scientific Instruments).

1 μm

Fig. 3. A $2.5 \times 2.5\text{-}\mu\text{m}^2$ AFM image of an ITO surface, scanned in 'error-mode' with an e-beam deposited 'supertip'.

between the two consecutive measurements. The optical images are recorded simultaneously with the AFM images. The corresponding PSTM images show that with p-polarized light (Fig. 4c) the optical image contains high-resolution information strongly resembling the topography of the sample, superimposed on an interference-like pattern. The ITO grains always appear dark on the glass substrate in the PSTM images, because the optical coupling is reduced by a combination of refractive index, absorption and topographic effects. With s-polarized light (Fig. 4d) the image hardly shows any high-resolution information but merely an interference-like pattern with a periodicity of typically $0.7\text{ }\mu\text{m}$. The standing wave interference pattern reflects the beat of homogeneous waves propagating along the surface caused by the residual scattering at the ITO particles, where the periodicity can vary from $\lambda/2$ up to micrometre-scale depending on the relative propagation directions (van Hulst *et al.*, 1992a; Meixner *et al.*, 1994). The measured average optical power is 260 pW ($\pm 20\%$) and 160 pW ($\pm 30\%$) with p-polarization and s-polarization, respectively. The given percentages denote the relative variation of the signal over the entire image, $(\frac{1}{2}[\text{max} - \text{min}]/\text{average})$. The observed average ratio of the optical power for p- and s-polarized light is approximately 1.9. The calculated p- and s-electric fields on the sample surface are approximately of equal size, according to Eq. (2), taking into account the polarization-dependent reflectivity

Fig. 4. A $2.5 \times 2.5\text{-}\mu\text{m}^2$ image of an ITO surface, scanned with an etched SiN probe: (a, b) AFM 'error-mode' images; (c) PSTM image corresponding to (a) with p-polarized incident light; (d) PSTM image corresponding to (b) with s-polarized incident light.

and transmission, before the HeNe beam has reached the TIR plane. An explanation for the difference can be found in the polarization-dependent coupling to the tip and/or the polarization-dependent transmission of the tip.

Polarization contrast by changing the polarization direc-

tions of both the incident and the detected light is shown in Fig. 5. The crossed polarizer cases, p-polarized incident and s-polarized detected light (PS, Fig. 5a) and the reverse situation (SP, Fig. 5d), display images with some high-resolution information resembling the topography of the

Fig. 5. A $2.5 \times 2.5\text{-}\mu\text{m}^2$ PSTM image of an ITO surface, scanned with an etched SiN probe: (a) PS, p-polarized incident and s-polarized detected light; (b) SS, s-polarized incident and s-polarized detected light; (c) PP, p-polarized incident and p-polarized detected light; (d) SP, s-polarized incident and p-polarized detected light.

sample surface, but with mainly an interference-like pattern. The PP-situation (Fig. 5c) clearly shows highest contrast. For SS-polarization (Fig. 5b) the image only shows an interference pattern, in which sometimes an

ITO particle is weakly visible. Consequently, when the same polarization of the detected light is viewed, p-polarized incident light provides the best and highest resolution optical contrast. When the same polarized incident light

is used again, p-polarized detection provides the best contrast. The measured optical power reveals more details. The measured optical powers are: PS, 24 pW ($\pm 45\%$); SS, 130 pW ($\pm 40\%$); PP, 150 pW ($\pm 25\%$); SP, 54 pW ($\pm 35\%$). With p-polarized incident light the detected light is thus predominantly in the p-direction, but 15% depolarized. For s-polarized incident light the average depolarization is about 40%. With a supertip similar measurements were performed, resulting in the following series: PS, 1.5 pW ($\pm 50\%$); SS, 12 pW ($\pm 35\%$); PP, 21 pW ($\pm 25\%$); SP, 2.4 pW ($\pm 40\%$). In this case the polarization conservation is larger than for a conventional tip, indicating that the conservation of polarization is also determined by the tip shape. The PS and SP situation did not provide suitable contrast for imaging owing to the small optical power. Figure 6 shows a PSTM image with PP polarization scanned with a supertip, where the smallest resolved ITO particles are about 50 nm in size. Comparison with Fig. 5(c), scanned with a conventional tip, clearly shows that the optical image can also be influenced by tip artefacts.

In order to obtain more insight into the mechanism of optical coupling, approach curves for different polarization situations have been measured where simultaneously AFM deflection and PSTM signal are recorded. Figure 7 displays a complete approach curve for PP-polarization. The PSTM curve is the result of averaging 300 approach curves, at

1 μm

Fig. 6. A $2.5 \times 2.5\text{-}\mu\text{m}^2$ PSTM image of an ITO surface, scanned with a 'supertip', p-polarized incident and p-polarized detected light (PP).

Fig. 7. Approach curves: AFM deflection signal and PSTM signal (pW) as a function of the distance between tip and ITO sample, for p-polarized incident and p-polarized detected light (PP).

the same position on the sample. In the curves the tip approaches the sample surface from right to left. The shape of the AFM deflection curve is easily explained. At large tip-surface distances there is no measurable force interaction. Approaching the surface the tip snaps into contact due to the attractive forces of a thin water film on the sample surface, as can be seen in the small dip in the curve. In contact the deflection, and thus repulsive force, increases linearly when the z-position of the sample is increased further. The PSTM signal shows a different behaviour. The optical signal increases exponentially with decreasing distance due to the exponentially decaying electric fields at the sample surface. In contact the optical signal is approximately constant, but when the z-position of the sample is further increased the optical signal almost always still increases slightly, probably due to changes in the alignment of the optical path and/or the directional dependence of the optical signal originating from the backside of the tip. The intermediate regime is more complex. In close proximity the behaviour of the optical signal depends on the polarization and the tip shape.

In Fig. 8 the PSTM approach curves for the four different polarization situations (PS, SS, PP and SP) are shown, emphasizing the difference in shape for the different polarization situations. For every polarization situation the detected optical signal in contact is mainly determined by the coupling to the evanescent wave, as can be observed from the decay of the curves with the expected penetration depth and the resemblance to the coupling-distance curves as predicted by theory. The scattering contribution is always much weaker than the near-field signal. The curves with p-polarized incident light (PP, Fig. 8c and PS, Fig. 8a) display a somewhat different shape than the curves with s-polarized incident light (SS, Fig. 8b and SP, Fig. 8d). The p-incident curves behave exponentially at large distance,

Fig. 8. AFM deflection signal and PSTM signal (pW) as a function of the distance between tip and sample for different polarization situations: (a) PS, p-polarized incident and s-polarized detected light; (b) SS, s-polarized incident and s-polarized detected light; (c) PP, p-polarized incident and p-polarized detected light; (d) SP, s-polarized incident and p-polarized detected light.

but at smaller distance the steepness of the curve decreases. The s-incident curves just show exponential behaviour at small distance. According to theory, this is valid for semi-infinite plates, as suggested by Zhu *et al.* (1986) and van Hulst *et al.* (1991). In addition, it can be observed that the scattering contribution is not the same for every polarization situation. The SS-situation, in particular, shows considerable far-field scattering contribution at larger tip distance. This was also observed in the far-field by placing the second polarizer in front of the CCD camera. The polarization-dependent scattering contribution could partly explain the different depolarization for p- and s-polarized incident light. Considering the far-field scattering with the sample particles as radiating dipole sources s-polarization does indeed give the largest contribution (Propstra, 1994).

Conclusions

The use of optically transparent SiN probes allows simultaneous detection of force and photon tunnelling signal, with a feedback on the force signal for regulation of the tip-sample distance. The method is versatile and nondestructive, especially in comparison with the fibre approach, and suitable for systematic investigation of polarization effects. Force and optical images show good correlation, both with a lateral resolution limited by tip size to about 50 nm. The resolution is further improved to about 30 nm employing 'supertips', but with an order of magnitude less optical signal.

p-Polarized light always displays largest optical contrast with reduced optical light coupling (up to 40%) at the ITO grains. Although inverted, the contrast closely resembles the topography, both with incident and detected p-polarization (PP). Yet the strong PP-contrast is not purely

a topographical effect because it changes with polarization conditions. In addition, the contrast is too large to be determined by absorption in the ~ 100 -nm ITO grains. Thus, we attribute the contrast to a combination of refractive index, absorption and topographic effects. The observed contrast is in agreement with calculations by Girard & Bouju (1992) and van Labeke & Barchiesi (1992), which also predict contrast correlated largely with topography for p-polarized incident light. Direct comparison is still difficult as the calculations assume isotropic index and no absorption and display the signal level at a constant distance from the surface, in contrast to the actual situation of contact scanning.

PSTM does not necessarily reveal subwavelength information as might be expected. Specifically in the case of SS-polarization, the detected signal is definitely determined by coupling to the evanescent wave, as can be seen in the approach curves, yet the PSTM image does not display high-resolution information.

Scattering contributions are significantly present in PSTM, which can obscure the high-resolution near-field contrast. For the small ITO grains scattering is limited, yet residual interference patterns are observed with a period of the order of the wavelength.

The optical coupling is larger for p- than for s-polarized incident light, according to the Fresnell theory for semi-infinite plates, but quantitative agreement requires that the tip and sample configuration on a microscopic scale is taken into account.

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