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N.F. van Hulst, M.H.P. Moers, O.F.J. Noordman, T. Faulkner, F.B. Segerink,
K.O. van der Werf, B.G. de Grooth & B. Bölger

Department of Applied Physics, University of Twente,
P.O.Box 217 7500AE Enschede, the Netherlands

ABSTRACT

Images obtained with a Scanning Near field Optical Microscope (SNOM) operating in reflection are presented. We have obtained the first results with a SiN tip as optical probe. The instrument is simultaneously operated as a Scanning Force Microscope (SFM). Moreover the instrument incorporates an inverted light microscope (LM) for preselection of a scan area. The SiN probe is operated in the contact regime causing a highly improved lateral resolution in the optical image compared to an alternative set-up using a fibre probe, which is also presented. The combined microscope is operated either in open loop or as a force regulated SNOM. Near field optical images can be directly compared with the topography displayed in the simultaneously recorded SFM image.

1. INTRODUCTION

A major difference of Scanning Near field Optical Microscopy (SNOM), compared to STM and SFM, is the fact that the interaction is optical and consequently all techniques of conventional optical microscopy, e.g. detection of luminescence and polarization effects, can be adapted to SNOM provided a sufficient signal. However, the potential of SNOM depends strongly on the fabrication of efficient and reliable probes. This problem has stimulated the development of several types of near field probe tips over the last years.

The basic probe, developed by Pohl *et al.*^{1,2}, is a sharpened dielectric tip coated with metal, leaving an aperture at the apex, 20 nm diameter being a practical lower limit. Betzig *et al.*³ have improved these probes by pulling adiabatically tapered single mode fibres which enable to illuminate the aperture with a higher efficiency. With these probes 12 nm lateral resolution and polarization contrast has been demonstrated. Recently Tortonese *et al.*⁴ have succeeded in fabricating single crystal silicon apertures down to 250 nm. The application of aperture probes is limited to samples with gentle surface roughness as the outer diameter at the apex is generally larger than 250 nm.

An interesting alternative probe type is formed by micro-pipettes, pulled to an inner diameter of about 50 nm and subsequently coated with metal. Betzig *et al.*⁵ have obtained a lateral resolution of about 50 nm using these tips as detection probe. Lewis *et al.*⁶ have filled these metal coated pipettes with fluorescent dye, thus generating a miniature fluorescence light source with relatively high brightness. Again these probes are restricted to flat samples.

An alternative approach based on localized frustrated total internal reflection has been described by Reddick⁷, Courjon⁸ and our group^{9,10}. In this case an evanescent field is generated at the sample surface by total internal reflection. The evanescent field is frustrated by a sharp dielectric probe and converted into a propagating wave. A lateral resolution down to 30 nm has been observed⁹. This alternative probe circumvents the problem of aperture fabrication, however it is restricted to transparent samples and rather sensitive for radiatively scattered waves¹⁰.

Recently Fischer¹¹ has presented the concept of an optical coaxial line, i.e. an optical fibre with a metal core. This type of probe seems promising in view of its expected efficiency.

In the first part of this paper images obtained with a SNOM operated in reflection are presented, where the optical probe is a sharpened fibre. The microscope is applied to compact disc test structures, scanned at constant height. Distinct differences between the optical image and the surface topography have been observed, e.g. the reflected signal is enhanced when scanning over pits. The lateral resolution is about 100 nm, mainly limited by tip sharpness and minimum distance between tip and surface.

All optical probes mentioned above have their specific limitations: generally a single tip touch is fatal, the efficiency is low, tips are hard to reproduce and samples should be flat. Yet for high resolution near field optical imaging close contact scanning at a distance below 20 nm is essential. On arbitrary surfaces this can only be accomplished by keeping the force between probe and sample low enough ($< 1 \mu\text{N}$) in order not to destroy the optical tip. We have realized an alternative SNOM where we take advantage of the unique features of the micro fabricated SiN probes commonly used in SFM. Hereto it should be noted that SiN, besides its ideal properties for micro fabrication and its mechanical strength, is also an ideal optical material ($n = 2.0$) with transparency down to 290 nm. Thus the SiN probe is a high index optical structure with 20 -50 nm apex, very similar to the probe types mentioned previously, with the advantage that it is integrated with a cantilever and can be scanned in close contact over a surface without damage.

In the second part of this paper we present preliminary results of near field optical imaging in a modified SFM using a micro-fabricated SiN probe with the SiN tip itself as an optical probe. The inner side of the SiN tip is illuminated and the reflected light is detected, thus constituting a reflection SNOM. Simultaneously the cantilever deflection is detected for force imaging. The combined SNOM-SFM microscope has been applied to the same compact disc test structures.

2. THE EXPERIMENTAL SET-UP

2.1. Reflection SNOM with fibre probe

In this set-up the probe is formed by a fibre, sharpened by chemical etching in HF⁹. A light beam is coupled into the cleaved end of the fibre and reflected at the sharpened end where the dimensions are below the cut-off size. An evanescent field is generated at the end of the fibre itself. Simultaneously a small fraction of the light is emitted as a radiative wave. A dielectric surface brought into close proximity frustrates the evanescent wave and part of the radiative wave is reflected, both affecting the effective intensity of the reflected light. The microscope is sketched in Fig. 1. The light source is a laser diode (5 mW, 780 nm) with short coherence length in order to avoid the contribution of interference between the fibre ends and collimating optics. The light reflected from the tip ($\sim 1 \mu\text{W}$) is directed towards a photo diode via a beam splitter.

Figure 1: Experimental set-up of the reflection microscope with fibre probe.

The sample is mounted on a commercial manipulator stage (Photon Control) with both mechanical and piezo-electric adjustments in x,y and z directions. The presence of a sample changes the reflected signal less than 1 % for transparent surfaces to up to 10 % for highly reflecting surfaces. The reflection signal is passed through a high-pass filter before further amplification in order to remove the DC offset. The cut-off frequency (0.1 Hz) is low enough compared to the line frequency (2 - 10 Hz) in order not to differentiate the signal. An image is obtained by scanning the sample at constant distance to the tip, without feedback. A similar "external reflection" microscope using a HeNe laser and 3 dB fibre couplers has been described by Courjon *et al.*^{8,12}.

2.2. Combined SNOM-SFM-LM with integrated SiN probe

The set-up is sketched in Fig. 2. The microscope is based on the design of a scanning force microscope with integrated optical microscope as developed by Putman *et al.*¹³. The heart of the instrument is a commercial micro fabricated SiN cantilever with integrated pyramidal tip¹⁴, where the cantilever is placed at 15° angle to the sample surface. The position of the cantilever is fixed during operation, while the sample is scanned using a piezo-electric tube.

Figure 2: Experimental set-up of the combined SNOM (top) - SFM (middle) based on a SiN cantilever, combined with an inverted epi-illumination light microscope (LM, bottom). For clarity the size of the SiN cantilever has been exaggerated.

In Fig. 2 three different optical paths should be distinguished :

(i) SNOM-system : Above the cantilever is a lens which focuses a HeNe laser beam at normal incidence into the SiN tip. The laser spot at the cantilever has 2-3 μm diameter, which fits within the 3 x 3 μm bottom facet of the SiN pyramidal tip and is thus illuminating the inner side of the SiN apex. A magnified image of the tip in reflection is formed outside the microscope head. A diaphragm in the imaging plane is adjusted such that only the beam reflected at the centre of the tip is passed onto a photo diode. Part of the detected light intensity ($\sim 1 \mu\text{W}$) comes from direct reflection at the Au coating on the back of the cantilever. At the apex of the illuminated tip we expect an evanescent field, caused by total internal reflection, while part of the light is transmitted as a radiative wave.

(ii) SFM-system : Close to the cantilever, within the microscope head, is an optical beam deflection system (Meyer *et al.*¹⁵) for detection of the interaction force between tip and sample. The laser diode (3 mW, 780 nm) is focused onto the cantilever at 30 ° incidence angle and the reflected beam is detected on a split detector. In fact the detector is a 4-segment photo diode which enables separate detection of cantilever deflection and cantilever torsion.

(iii) LM-system: Under the sample is an inverted optical microscope with epi-illumination. The objective, a commercial CD-player lens with 0.45 NA, is mounted within the tube and can be manipulated independently in x,y and z directions. The illumination source is a 100 W Hg arc lamp. The LM facilitates simultaneous viewing of the sample, the position of the tip over surface, the position of the HeNe beam in the SiN pyramid and the position of the laser diode spot on the cantilever. Thus the LM enables preselection of a required scan area and alignment of the HeNe beam focus within the SiN tip.

The instrument is operated in air, in the contact mode, either in open loop or at constant force with a feedback on the optical beam deflection signal. Operation with released tip is also possible, however, the minimum distance out of contact is $\geq 100 \text{ nm}$ due to attractive surface tension forces caused by the water film on the surface (Weisenhorn *et al.*¹⁶).

3. SAMPLE

The performance of the microscopes has been verified on compact disc test structures, obtained from Philips Research Labs. the Netherlands. Figures 3 a & b show images of the CD-structure as observed with the LM. The polycarbonate structure, fabricated by replication from a stamper, consists of indented tracks varying from dotted patterns to long trenches. The track spacing is 1.25 μm . The trench width is $175 \pm 25 \text{ nm}$.

Figure 3: 18 x 13 μm area of the compact disc test structure observed with the inverted optical microscope, displaying trenches, pits of 2.0 μm length (a) and circular pits (b).

4. RESULTS

4.1. Images with the fibre probe system

When decreasing the distance between the fibre tip and the sample surface a characteristic Fabry Perot fringe pattern is observed, caused by interference between tip and sample. The fringe amplitude is increasing rapidly towards the surface, down to a distance below $\lambda/2$, where an abrupt deviation from the fringe pattern is observed, corresponding to the near field regime. The deviation is rather variable depending on the sample surface and the fibre tip used.

Figure 4a: 7 x 7 μm image of the reflected signal over a structure with Ag coated pits of 2.0 μm length and 1.25 μm period.

Figure 4b: 9 x 9 μm scan over an area with Ag coated circular pits (200 nm diameter) and trenches, both with 1.25 μm period.

Fig. 4a shows a 7 x 7 μm 3D image of the reflection signal over a part of the area displayed in Fig. 3a, scanned at a constant height of about 100 nm. The CD pits have been coated with Ag for higher reflectivity. The actual pits are 2.0 μm long and 175 nm wide. The pits stand out on their surrounding area as they appear to enhance the reflected signal. This has been observed also at larger distances up to 3 μm , be it with less intensity. It appears that the pits enhance the total level of reflected signal, independent of the distance dependent fringes on top of the it. Probably the pits act as focusing objects. The signal level directly beside the pits is slightly below the average signal level of the surrounding area. This feature disappears when increasing the distance. The observed width of the pits is about 500 nm and increases with the working distance. The lateral resolution is about 100 nm, mainly determined by the sharpness of the tip and the non-zero distance and the effective acceptance angle of the tip for reflected light. In the upper right corner of Fig. 4a are interference fringes with $\lambda/2$ period.

Fig. 4b shows a 9 x 9 μm reflection scan over an area as in Fig. 3b, displaying both circular pits and long trenches. Again the indented pits enhance the reflected signal. They stand out as bumps with $\sim$ 500 nm diameter, while the actual size is $\sim$ 200 nm. The trenches appear as a slight decrease in the average reflectivity, whereas the intermediate region appears more pronounced with stronger signal decrease. The appearance of the trenches is probably dominated by mutual interference of the reflected light from adjacent trenches.

We have found that the images depend on the shape of the etched fibre tip. Although the general features reproduce, distinct asymmetric structures can be observed from tip to tip.

Line scans over a grating by Courjon *et al.*^{8,12} display similar fringe-like patterns superimposed over the actual structure and enhanced reflection signal in the grating grooves. Girard *et al.*¹⁷ have evaluated a theoretical model indicating that spurious waves emitted from the tip could also account for the oscillatory patterns observed in reflection imaging.

Force

Optical

(a)

(b)

(c)

(d)

(e)

Figure 5: 6.5 x 6.5 μm scans over a CD structure with tracks and pits:

- (a) SFM image in contact mode. The microscope is scanned in open loop and the force signal is displayed.*
- (b) optical signal recorded simultaneously with (a).*
- (c) SFM image in contact mode. The microscope is scanned in closed loop and the feedback signal is displayed.*
- (d) optical signal recorded simultaneously with (c).*
- (e) optical signal recorded with the tip released from the sample surface. The corresponding SFM image is not displayed as no force effect can be observed.*

4.2. Images with the SiN probe system

Fig. 5 shows images as obtained with the combined SNOM-SFM system. An area on the polycarbonate structure was scanned similar to the area displayed in Fig. 3a., with trenches and pits of 1.0 μm length.

Figures 5a and 5b display the simultaneously recorded SFM and optical image, respectively, as obtained in close contact without feedback on the force signal. Thus the cantilever follows freely the underlying structure. The force image corresponds to the topographic structure, where the finest detail of ~ 30 nm is determined by the tip sharpness. The white lines indicate a short release of the tip during a line scan. The optical image (Fig. 5b) corresponds to changes in the effective reflectivity. The signal is enhanced at the rim of the trenches and pits. The intermediate regions show smooth signal variation, correlated to the tracks. The fine corrugation, present in the force image, has disappeared in the optical image. The bending of the cantilever could influence the optical reflection signal, resulting in an SFM-like contribution. If present, this contribution can be largely reduced by operating in feedback. To verify this we have performed a similar scan, in close contact, this time with a feedback on the force signal. Thus the sample height is corrected according to the detected force, while the cantilever is not bending within the bandwidth of the feedback system. The simultaneously recorded SFM and optical image are displayed in Figures 5c and 5d, respectively. The SFM image in Fig. 5c, displaying the feedback signal, corresponds to the topographic structure as also observed in Fig. 5a. The optical image in Fig. 5d is very similar to Fig. 5b, except for some higher spatial frequency details, indicating that the effect of bending of the cantilever on the optical image is limited. Finally, in order to verify the effect of any other residual force, we have performed a scan with the tip released. Fig. 5e displays the image, which now consists of purely optical effects. The distance between tip and sample is at least 100 nm, in order not to be attracted by the surface tension force. The cantilever deflection was below the detection limit and consequently a corresponding SFM image is not displayed. Fig. 5e still shows the underlying track structure, however any fine structure is lost. Moreover contrast appears to reverse from left to right.

It should be noted that the optical images in figures 5b, d and e originate from the SiN tip as they disappear when the HeNe beam is focused beside the pyramid. The optical signal is of comparable amplitude in open loop, closed loop and with released tip. Positioning of the focus in the pyramid is critical and affects the observed images.

Interpretation of figures 5b, d and e is not straightforward. Based on the experiments we suppose that the detected optical signal consists of the reflected light at the apex, an interference contribution and some residual effects of cantilever bending. Obviously further research should be performed to clarify aspects.

5. CONCLUSIONS

Both types of microscopes presented are capable of optical imaging with structural details surpassing the optical diffraction limit.

The fibre probe system is easy to operate, however the fibre tip is a weak point. Images are mainly determined by geometrical optical effects and interference of radiative waves.

We have obtained the first optical images using the integrated SiN probe. The probe is highly promising for high resolution optical imaging as it can be scanned in close contact without damage. Moreover it can simultaneously be operated as an SFM which enables force regulation and direct comparison with the force images. The optical images display structural features down to 50 -100 nm. Further experiments are necessary to interpret the presented images.

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