

A COMBINED NEAR FIELD OPTICAL AND FORCE MICROSCOPE

M.H.P. Moers, R.G. Tack, N.F. van Hulst* and B. Bölger

MESA Research Institute, University of Twente,
P.O. Box 217, 7500 AE Enschede, The Netherlands

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Abstract

Scanning near field optical microscopy (SNOM) is the optical alternative of the scanning probe microscopical techniques which enables a lateral resolution down to about 10 nm, unlimited by diffraction. Moreover, the potential of non-destructive imaging of chemical and biological samples with nanometer resolution in ambient conditions is a crucial advantage over electron microscopy.

An integrated microscope has been constructed which allows simultaneous detection of optical and force interaction between a microfabricated SiN probe and a sample surface. Images are obtained in a transmission mode by detection of the light emanating from a "super-tip" in contact with the sample surface and illuminated by total internal reflection. Physical and technical aspects of the instrument are discussed and illustrated with typical images.

Key Words: Near field optics, scanning probe microscopy, evanescent wave, atomic force, feedback, integrated microscopes.

Introduction

Although the concept of imaging beyond the diffraction limit has been formulated already by Synge (1928), experimental near field microscopy in the optical domain has been stimulated entirely by the invention of scanning tunneling microscopy and advances over the last years have been mainly involved in development of suitable probes (Pohl, 1990). Currently used probes are to be divided in aperture and dielectric type.

The aperture probe is a sharpened dielectric tip coated with metal, leaving an aperture at the apex, where 20 nm diameter is a practical lower limit. Betzig *et al.* (1991) have demonstrated 12 nm lateral resolution using aperture probes and recently Betzig *et al.* (1992) demonstrated the capacity of these probes for reading and writing of magneto-optical domains down to 60 nm resolution. Although the efficiency of these metal coated aperture probes is relatively high, their application is limited to samples with low topography as the outer diameter of the probe is about 0.25 μm .

The dielectric probe type, which is generally a sharpened fibre, is used to frustrate an evanescent wave and convert it into a propagating wave, where the evanescent wave is generated at the sample surface by total internal reflection. Due to similarities with electron tunneling, this technique is often referred to as photon scanning tunneling microscopy (PSTM) (Reddick *et al.*, 1989; Courjon *et al.*, 1989; van Hulst *et al.*, 1991). Although PSTM is capable of super-resolution, the technique has the disadvantage that, due to scattering at a sample surface, both evanescent and propagating waves are generated, which cannot be discriminated in the detected optical signal (van Hulst *et al.*, 1992). Consequently PSTM images generally display a combination of localized near field and long range far field effects and high resolution images can only be obtained at a probe-sample distance below 20 nm where the near field is dominant over any far field contribution. Hence, PSTM is also limited to transparent objects which exhibit only limited scattering contribution, i.e., structures with size far below the wavelength.

We present an alternative to the PSTM taking advantage of the microfabricated silicon-nitride probes

*Address for correspondence:

N.F. van Hulst
MESA Research Institute,
University of Twente,
P.O. Box 217
7500 AE Enschede,
The Netherlands

Telephone number: + (31) 53 893172/893157
FAX number : + (31) 53 309549

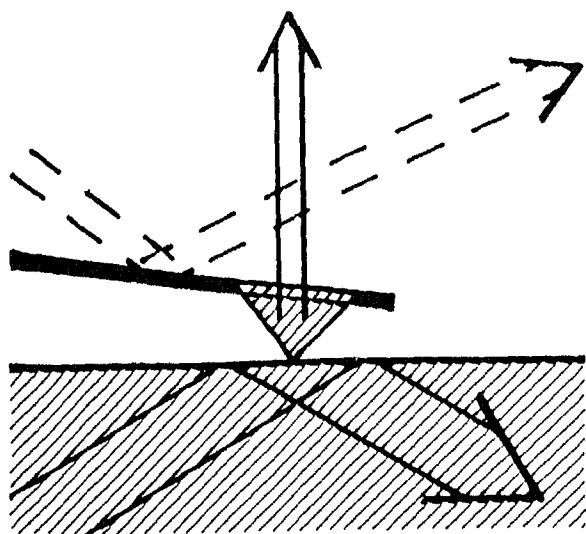


Figure 1. Schematic set-up of the combined microscope using a SiN probe with near field optical detection based on localized frustrated total internal reflection (FTR), and force detection based on optical beam deflection.

with "supertip" as they are commonly used in force microscopy. These probes can be operated in close contact simultaneously as an optical and a force probe (van Hulst *et al.*, 1993).

Experimental Set-up

The microscope, as sketched in Figure 1, is basically a normal PSTM configuration in which an evanescent wave is generated on a glass substrate by total internal reflection of a HeNe laser beam. At variance in this set-up, the optical probe is formed by a micro fabricated SiN probe (Park Scientific Instruments) which is commonly used in force microscopy. This probe consists of a pyramidal SiN tip with 20-50 nm apex and is integrated on a cantilever with low spring constant (0.06 N/m) such that it can be scanned with a low interaction force in close contact over a surface. SiN is an optical material ($n = 2.0$) with transparency down to 290 nm, making the SiN probe a high index optical structure with 20-50 nm apex, which is ideal as a near field optical probe. A long working distance objective (40X, 0.45 NA, 10 mm) collects the light generated by frustrated total internal reflection (FTR) at the SiN apex. A 100 μm pinhole in the imaging plane is adjusted such that only light from the SiN apex passes onto the detector. The light path after the objective is partly split towards a camera which facilitates alignment of the system and location of a sample area. This feature is very practical in locating isolated objects. The interaction force between tip and sample is determined by the bending of the cantilever which is detected in an optical beam deflection system, formed by a laser diode focused onto the cantilever where the reflected beam is detected on a quadrant de-

tor. This optical beam deflection system enables detection of both cantilever deflection and torsion caused by the interaction force between tip and sample. A near field optical image and a force image are obtained simultaneously by scanning the sample in contact with the probe, either in open loop or at constant force with a feedback on the beam deflection signal. In order to improve the localization of the optical coupling and to avoid tip convolution effects, a so called "supertip" (Keller and Chih-Chung, 1992) has been grown on the standard SiN tip by electron beam deposition. Figure 2 shows a scanning electron micrograph of a typically 300 nm long "supertip".

Results

On approaching the probe towards the sample surface, an exponential increase of the optical signal is observed corresponding to the coupling to the evanescent wave which has exponential distance dependence. At some distance, the tip jumps into contact caused by the adhesion force (~ 10 nN) of the water film on the surface. While scanning, the variation of the optical signal in contact is monitored.

As a typical example, Figure 3 displays corresponding force (a) and near field optical (b) images, respectively, of a commercially available indium-tin-oxide film (Baltracon). Crystallites varying in size between 30 and 100 nm are clearly resolved in the force image. In the optical image, the same granular structures are resolved, however, the grains appear dark against the background due to their higher refractive index and limited transmissivity at $\lambda = 632.8$ nm. Between the grains, rather sharp light rims are observed with a width of about 20 nm, indicating that the supertip reaches towards the glass surface in the intermediate regions.

Conclusions

The use of SiN probes eliminates several practical problems encountered in fibre based PSTM designs and allows routine non-destructive detection of localized optical interaction in close contact scanning on arbitrary surfaces. Combination with force detection provides an independent feedback mechanism and enables direct comparison of topographic and dielectric properties. Near field optical images with a lateral resolution down to 20 nm have been obtained, where the optical contrast is determined by surface topography, refractive index and transmissivity variations. Clearly, understanding of the optical contrast mechanism requires further study.

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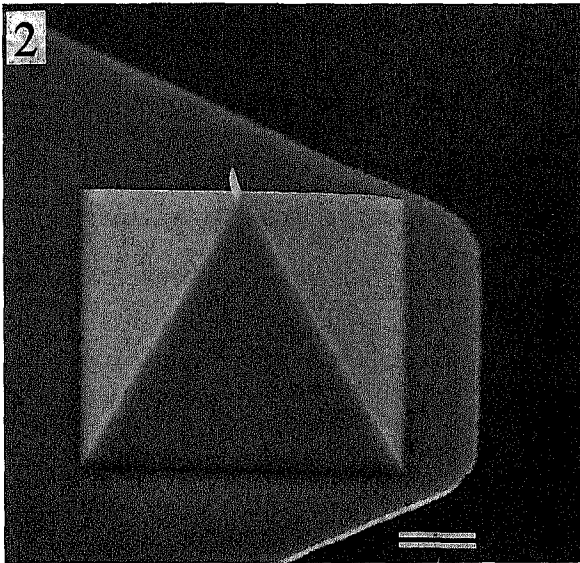


Figure 2. Scanning electron micrograph of a "supertip" fabricated by electron-beam deposition. Bar = 1.25 μm .

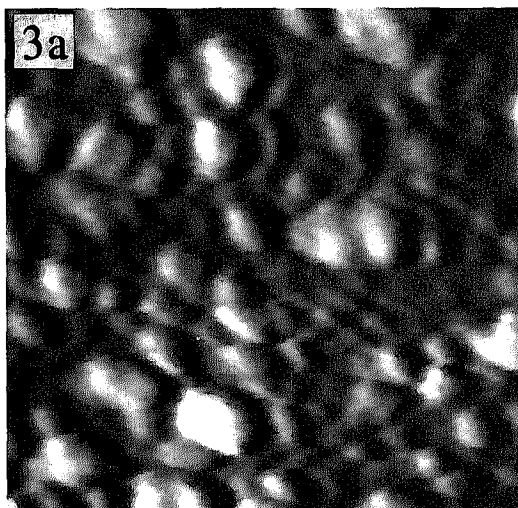
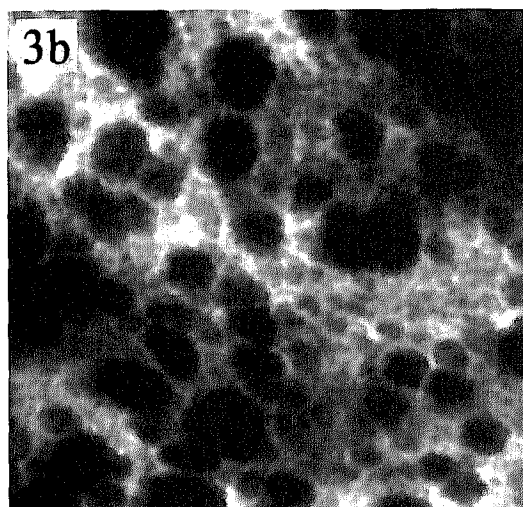


Figure 3. Force (a) and corresponding near field optical (b) images (at $\lambda = 632.8 \text{ nm}$) of indium-tin-oxide grains, scanned with a "supertip". Scan area 700 x 700 nm^2 .



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Discussion with Reviewers

T.L. Ferrell: In frustrated total internal reflection using a fiber tip at very small gaps, there is a positive-argument exponential as well as a negative-argument exponential. Have you measured this as the tip is withdrawn from the surface?

Authors: In withdrawing the SiN probe from the surface it snaps out of contact if the applied force is sufficient to break the adhesion. During this almost instantaneous process, a negative argument exponential signal behaviour has not been observed.

T.L. Ferrell: Small-amplitude oscillations of the tip and use of a lock-in amplifier have proved useful in separating PSTM signals of more rapid spatial variations from those of weaker dependence upon distance from the surface. What considerations have you given to tip dithering and also to wavelength dithering in your experience with PSTM imaging?

Authors: The current experiments are conducted in contact mode, obviously without dithering. A tapping mode operation might prove beneficial in the separation of high and low spatial frequency variations.

T.L. Ferrell: The acceptance angle of a sharpened fiber optic tip limits the scattered light detected in PSTM imaging, although the angle is dependent upon tip shape. In your earlier PSTM work, have you observed any reduction in scattered light as a function of tip shape?

Authors: Far field contributions are often less prominent for sharper tips (van Hulst *et al.*, 1992).

T.L. Ferrell: The term "transparent objects" which you use often refers to objects transparent in white light, but most samples have optical windows in certain frequency bands or transmit above a threshold. This is just a comment that may prove useful for further investigations?

Authors: Indeed, we use "transparent" for a given frequency.

T.L. Ferrell: The term "subwavelength" imaging is more general than "near field" imaging as there are currently many techniques that do not use apertures as in the historically developed near-zone instruments. Would you classify your instrument more as a scanning scatter source subwavelength probe?

Authors: The term "scanning scatter source subwavelength probe" seems appropriate for our instrument.

Authors' note added in proofs: A detailed description of current status of near field optical microscopy can be found in: Proceeding of the International Workshop on Near Field Optical and Related Techniques (Arc and Senans, Oct. 1992). Pohl DW (ed.). Kluwer Academic Publishers, the Netherlands (1993).