Optical contrast in near-field techniques

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Abstract

In this paper results of experiments with a scanning near-field optical microscope with shear-force feedback are presented. The setup will be described and the shear-force signal as function of distance is shown. Images of latex spheres and Langmuir-Blodgett layers of pentacosa-acid with about 100 nm lateral resolution are presented which show a true optical contrast due to fluorescence and polarization.

1. Introduction

Scanning near-field optical microscopy (SNOM) offers the possibility to combine conventional optical contrast mechanisms with the high resolution of scanning probe microscopes [1]. The advantages of SNOM are first fully exploited when the optical image shows features which cannot be obtained with another scanning probe method, e.g. atomic force microscopy, as well. New information about the sample must be gained. In this paper a SNOM setup with a feedback on shear force is described. The force part of the system is treated in more detail. Measurements are presented which show contrast due to fluorescence and polarization.

2. The instrument

The instrument used in these experiments is shown in Fig. 1. The setup is basically the same as previously described by Betzig et al. [1,2]. An Ar+ laser (514 nm) is coupled into a multimode fiber via a quarter lambda plate and a polarizer. The other end of the fiber is adiabatically tapered using a fiber puller (Sutter P2000) and coated with about 200 nm Al in such a way that the tip end remains uncovered. The optical power coming out of this aperture typically is 10–100 pW, whereas the power coupled into the fiber is 10 mW.

In the experiments described here the near-field optical light source is used for the local excitation of fluorescent material. The system is mounted on an inverted microscope (Zeiss Axiovert 135M), with a 40×, 0.60 NA objective. The fluorescence is detected using a PMT. In the optical path a dichroic mirror (λ = 560 nm) and high-pass filter (λ > 590 nm) are used.

The tip is placed near the sample while the distance is controlled with a feedback on shear force [2,3]. The shear-force detection scheme used in this setup is different from the setup described by Betzig et al. [2]. We have chosen for an external detection of the oscillation of the tip, whereas Betzig et al. have used a detection through the...

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sample, which might induce a coupling between optical properties and the shear force. A laser diode is focused in front of the fiber, which is oscillating at its resonance frequency (10–20 kHz) driven by a multimorph piezo element. The laser beam crosses the fiber at about 1 mm above the actual tip end and has no interaction with the sample surface. The shift of the diffraction pattern is monitored using the difference signal of a split detector. Gain and phase are detected by a lock-in amplifier, on which the gain, the time constant and the set point can be preselected. The output of the lock-in is amplified and connected to a multimorph piezo element which controls the tip–sample distance.

The sample is scanned using a piezo scanner which is built around the objective of the inverted microscope and which allows support of standard microscope object glasses. A computer is used to generate the scan pattern and to collect the lock-in and optical signals. Scan time is determined by the response time of the shear-force feedback loop (~1 ms). A 300 × 300 pixels image requires typically 15 min.

3. Results

In Fig. 2 the voltage of the lock-in amplifier \( R \cos \theta \) is shown as a function of the distance between the probe and the sample. This is done for several lateral dither amplitudes of the fiber end. On approaching the fiber to the sample surface the signal rapidly decreases for distances smaller than a few nanometers. This relatively short distance behavior differs from observations by other groups, where a change in signal is reported already at about 100 nm separation. An
Fig. 2. The shear-force lock in voltage as function of tip-sample distance for several lateral dither amplitudes.

explanation might be the fact that the signal is detected through the sample, such that optical effects play a role [2,4]. From several contributions in this issue it appears that although the mechanism on which the shear-force regulation depends is far from understood, it is widely in use, and with success.

Fig. 3 shows a 3.5 × 3.5 μm² image of a double layer of latex spheres (Molecular Probes), 282 nm in size. Actually the layer contains a mixture of two types of fluorescent spheres, equal in size, but one type (Nile red) absorbing at ~551 nm and fluorescing at ~636 nm, the other type (Navy blue) absorbing at ~650 nm and fluorescing at ~700 nm. Fig. 3a shows the shear-force image in which the latex spheres in cubic packing look identical. Fig. 3b shows a fluorescence image (λ > 590 nm) with excitation through the tip at 514 nm. Here only the Nile-red stained spheres fluoresce and the difference between the two types of spheres is clearly visible. In combination with the shear-force image each sphere can be identified.

In Fig. 4 a Langmuir–Blodgett film on quartz glass with domains of (10,12)-pentacosadiynoic acid (PCA) is shown [5–10]. Some properties of these particular samples are: (i) the PCA domains are thin, approximately 6 nm, (ii) they show a strong absorption at ~500 nm and emission at ~600 nm, (iii) the absorption and emission moments are oriented uniform in each distinct domain, caused by the anisotopic growth of the polymer under UV exposure. Fig. 4a is the shear-force height image, showing several domains of PCA with 6 nm average height on the quartz substrate. Figs. 4b, 4c and 4d are fluorescence images in which the polarization is tuned by changing the polarization of the light coupled into the fiber. In Fig. 4b the incident polarization is circular and all domains are expected to fluo-
resce. However, upon comparison with the force image also some non-fluorescing domains can be identified. Between Figs. 4c and 4d the incident linear polarization is rotated over 90°. In Figs. 4c and 4d it is clearly visible that the relative fluorescence intensities of the different domains depend on the direction of linear polarization. The relative intensities change because the absorption depends on the angle between the polarization of the excitation and the absorption moment in each domain. One should note, that because the fiber is not polarization preserving, the incident linear polarization is not completely maintained at the probe end. The degree of polarization is about 0.8.

4. Conclusions

The setup described in this paper clearly allows to obtain pure optical contrast in addition to the topographical force image. The shear-force signal as a function of distance displays a short-distance behavior which is not fully understood. Although this is a common problem with shear-force regulation, the method shows to be a good feedback mechanism. Fluorescence images of the latex spheres and PCA films have been obtained with a rather high lateral resolution of about 100 nm. Fluorescent and non-fluorescent material can be distinguished by comparing with the force image. In addition the PCA films show polariza-

![Image](https://via.placeholder.com/150)

Fig. 4. 4.4 × 4.4 μm² image of a polymerized PCA film: (a) the shear-force height image, (b) fluorescence image with circularly polarized excitation, (c) with linear polarization and (d) with linear polarization rotated over 90° with respect to (c).
nation contrast, which allows determination of the orientation of the polymer backbone. Although the resolution can be improved, the set-up is already promising for application to chemical and biological samples.

References