Quantitative Cantilever Near-field Scanning Optical Microscopy

F. Marinello[1], P. Schiavuta[2], M. Balcon[1], S. Carmignato[3], E. Savio[1]
[1]*DIMEG, Dip. di Innovazione Meccanica e Gestionale, University of Padua, Italy*
[2]*CIVEN, Coordinamento Interuniversitario Veneto per le Nanotecnologie, Italy*
[3]*DTG, Dip. di Tecnica e Gestione dei Sistemi Industriali, University of Padua, Italy*
francesco.marinello@unipd.it

Abstract

Aperture Near-field Scanning Optical Microscopy (NSOM) is an important technique for high resolution optical imaging, beyond the diffraction limit; nevertheless, its quantitative application is still a challenge, due to a number of factors which influence the quality of the measurement output and consequently extrapolation of quantitative parameters. For the present work a systematic study was done, analysing in particular the effect of scan configuration in cantilever NSOM measurements.

1 Introduction

Conventional microscopy is among the most popular techniques for laboratory and industrial investigations, basically for the possibility of carrying out simple, fast and non-invasive analysis of different samples. Limitations for optical microscopy are mainly connected with the possibility of achieving three dimensional reconstructions of surface topography and with the limitations connected with the lateral resolution, due to the diffraction limit. Considering the visible light (say λ of 550 nm) propagating in air, with the maximum numerical aperture for a magnification lens NA=0.95, and considering negligible other optical aberrations and distortions, the best achievable resolution is d~200 nm, calculated through $d = \lambda/(2\pi NA)$.

Near-field scanning optical microscopy (NSOM, see Fig. 1) firstly proposed in 1984 by Pohl et al. [1], was introduced to partially overcome such limitations. Producing high-resolution optical images and providing information on surface topography with nanometer resolution, NSOM has opened up for important advances in different research domains such as lifescience (with markers), material science (absorbance, emission), sensors (plasmonic structures, resonant optical antennas) [2,3].

However quantitative application of NSOM is still difficult, due to a number of factors which commonly influence the quality of the measurement output and consequently extrapolation of quantitative parameters. For the present work a systematic study has been done, analysing effect of the most important influencing factors, mainly arising from tip geometry and aperture, scanning configuration and scan mode. Main results on scan configuration are reported in the following.

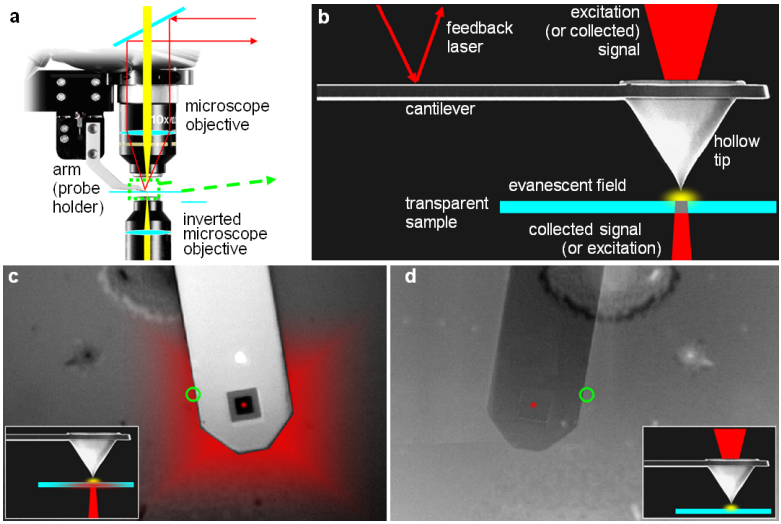

Figure 1: Schematic of the NSOM (a) and probe (b). (c) Collection mode, with the probe seen from the top, and the laser focused from the bottom through an inverted microscope; (d) illumination mode, with the probe imaged from the bottom by the inverted microscope, and the laser confined within the tip aperture.

2 Scan configurations

Two main configurations are normally used for NSOM analysis: “illumination” and “collection”. In the so called illumination configuration (Fig. 1b,c) an aperture probe is used to generate a near-field emission (“illuminates”) on a small area of a sample surface. The interaction of the emission (evanescent field) with the sample results in scattered light (propagating wave): the signal is transmitted through the sample and is eventually collected in the far-field by an inverted microscope.

In the so called “collection configuration” (Fig. 1b,d), the sample is irradiated by far-field as in classical microscopy: the irradiation is brought to the sample surface

through an inverted microscope. The evanescent wave generated on the sample surface is picked up by the probe located within the near-field region.

3 Configuration mode effect and discussion

Let's consider the collection mode first with the excitation light illuminating the sample. As depicted in Fig. 1, while the light spot (using high magnification power objectives) has a size of a few squared microns, due to light scattering a larger area is excited, often in the order of hundreds of squared microns (then an area larger than the cantilever). In the collection phase, the cantilever acts as a mask for blocking spurious signals and light scatter arising from the sample: nevertheless if light signal is coming from outside the probe, this is collected by the detector concurrently with the signal from the tip. The result is a worsening of the signal to noise ratio.

This phenomenon was studied using a transparent glass slide (with a 300 μm thickness). A He-Ne laser (wavelength $\lambda = 514$ nm) was focused at different heights, so that the laser spot was ranged from a minimum diameter of 30 μm (in focus position) up to 300 μm (defocused position), through a 60x magnification objection. The test was repeated implementing a 20× magnification objection to focus the laser light. For each position, the signal to noise ratio was checked through the photodetector. Independently from the used objective, the signal to noise ratio worsens as the spot size gets larger. A sudden worsening in particular occurs if, due to inappropriate defocusing, the laser spot is larger than the cantilever width (140 μm).

A different situation occurs when measurements are performed in illumination mode. Indeed in such case the presence of a large hollow tip allows for confinement of the light excitation. As a consequence the presence of disturbance arising from light scattering or spurious reflections is drastically minimized, with the final results of highly better signal to noise ratios: in the same analysis conditions, the signal to noise ratio exhibited values higher than 150.

The implemented configuration plays a role not only for the quality of the revealed signal, but also for the quantity of energy brought to the sample.

Indeed, while in the collection mode the excitation intensity is spread over the focused spot area of the sample, in the illumination mode the whole laser intensity is confined within the probe tip. The intensity of the laser sources most often

implemented as excitation light are normally in the range between 10^{-3} and 10^{-6} W. Considering that the tip has a volume of a few hundreds of cubic microns (corresponding to a few nanograms), the relative heating of the tip can be very significant. This is particularly evident when the signal is collected in spectroscopy mode, and an acceptable signal to noise ratio is achieved only using very low scan rates (normally a few tenths of seconds per point). In such condition the overheating of the tip causes an overheating of the measured area, and as a consequence evident defects on the sample surface (plastic deformation or burning). Additionally, the overheating of the tip can also damage the tip coating (with a consequent loss in signal quality) or induce distortions of the cantilever, with frequent loss of contact or relevant drift phenomena.

To verify this phenomenon, an experimental test was carried out on a polymer blend deposited on a glass slide through spin coating, in order to obtain a thin film deposition (thickness less than 10 µm). The polymer blend had a glass transition temperature T_g higher than 200°C. Repeated NSOM measurements were done on the same sample position, over a range of 10x10 µm, with a sampling of 256 lines and 256 points per line. Acquisition was carried on at different scan rates, with speeds during the forward scanning respectively at 10 µm/s, 2 µm/s, 1 µm/s and 0.5 µm/s. Signal was revealed in illumination mode, through a photodetector. Analyses clearly showed how at low scan rates, heat accumulated on the tip causes a blurring of the image, due most probably to a distortion of the sample (the two polymer phases are no more clearly separated). Also an overall reduction of the signal can be seen at slow scan rates, most probably due to polymer contamination attaching to the tip apex, in proximity or in correspondence of the tip aperture.

References:

[1] Pohl D.W. et al., Optical stethoscopy: image recording with resolution λ /20, Applied Physics Letters, 44, 651–653,1984.

[2] Enrichi F. et al., Investigation of luminescent dye-doped or rare-earth-doped monodisperse silica nanospheres for DNA microarray labeling, Optical Materials, 32/12, 1652-1658, 2010.

[3] Brovelli S. et al., White electroluminescence by supramolecular control of energy transfer in blends of organic-soluble encapsulated polyfluorenes, Advanced Functional Materials, 20, 272-280, 2010.