Near-field Scanning Optical Microscopy

Project Report

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Abstract:

This paper summarizes the basics of near-field scanning microscopy, one of the most advanced methods for studying surface properties. The principles of operation of the near-field optical microscope, the most widely used in scientific researches. The technological development of varieties of NSOM types is still ongoing, and some of the new technology updates is reviewed in this paper.

Contents

Work plan for Capstone II

1. Further development of the cell liquid chamber to work in liquid (to multi user construction to fit 15 mm x 15 mm square glass coverslip);
2. Further development of NSOM sensor;
3. Nanoscopic study of synthesized nanoparticles and living cells;
4. Solving the “grounding” problem of the NSOM head stage;

Unfortunately, none of this planned works are finished due to the problems we have faced. First of all, at the beginning of the semester we stacked with problem that our software program which allows to work with NSOM stopped running. We predicted that it was due to windows updates. Then we messaged the manufacturer company and they send us new drivers and luckily the program started to run. However, the sensor stopped adjusting properly. To be precise, after our check that the fiber probe is enough close to the sample and located at the right place, we adjust the sensor and command the sensor to search for the contact with the sample. In practice, the plate with the sample starts to come closer to the fiber probe tip until it slightly touches the surface of sample or gets really close up to few nanometers. Nonetheless, the sensor instead of getting closer to the sample were moving away. We monitored such occurrence several times and again wrote to the engineers who have constructed the NSOM in our laboratory describing our problem. According to our description they proposed that the problem is in the Z-translator which is responsible for the movement of sample plate and gave us further instructions of adjusting the Z-translator. Unfortunately, after several attempts we understood that are not able to fix the problem and were afraid of damaging the equipment and did not experimented on it. Since the equipment was still under guarantee, it was decided to contact the manufacturer company again to solve the problem. Before the quarantine started, the manufacturer company engineers did not show up. Consequently, we did not catch up to work on NSOM properly. Thus, for the final report in agreement with my supervisor, it was decided to do some literature review of the new technological updates of NSOM and near field techniques.

Introduction

Obtaining optical images of any matter by conventional methods have considerable limitations associated with light diffraction. The size of an object R due to diffraction limit can be found by using the fundamental formula in optics $R = \lambda/2n$, where n is the refractive index and λ is the wavelength of light being used for the image construction [1]. For the optical wavelength range, the limiting size is of the order of 200-300 nm. An image of an object can be constructed by alternative ways in the near-field optical microscopy, which allow one to overcome the difficulties associated with light diffraction and realize a spatial resolution of 50 nm or better [2].

Near field Optical Microscopy

Near-field optical microscope operates on the idea that the light goes through subwave diaphragms (the diameter of holes is apparently smaller compared with the wavelength of the incident radiation) [4].

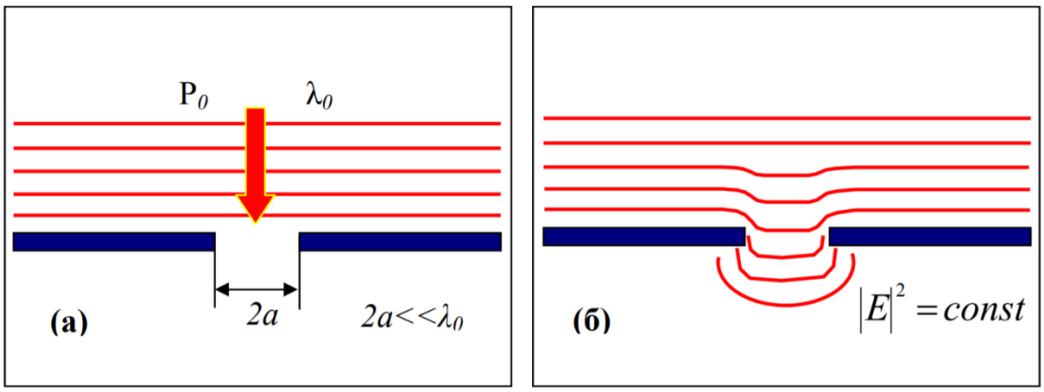

Figure 1. *(a) The light pass through an opening in a screen with a subwave aperture. (b) Optical radiation near subwave hole* [4].

When light passes through a sub-wave hole, various things can be examined. The construction of the electromagnetic field in the diaphragm is very complicated. Directly behind the hole at distances Z<100A, there is the so-called near zone, in which the electromagnetic field occurs as an evanescent (non-propagating) modes, which are restrained near the diaphragm's surface [5]. In the range of distances Z>100A, a distant zone is located in which only radiative modes are observed. The radiation power behind the subwave diaphragm in the far zone can be estimated by the following formula:

$$P_{tr} = \frac{128}{27\pi} k^4 a^6 W_0$$

where k is the wave vector, W_0 is the power density of the incident radiation [4,6]. In one hand, at the beginning one may argue that it is impractical to use small holes for constructing optical images of the studied samples. Nonetheless, when placing the studied sample straight behind the hole in the near zone, because of the cooperation of the evanescent modes with the sample, piece of the electromagnetic field energy goes into radiative modes [7]. An optical photodetector allows to distinguish the intensity of the energy. Thus, a near-field image is obtained when the test sample is scanned with a diaphragm with a sub-wave hole and is recorded as a distribution of the intensity of optical radiation depending on the position of the diaphragm I (x, y) [8]. The NSOM image contrast is dependent on the reflection, refraction or scattering of light whereas this physical phenomenas are dependent on optical characteristics of the sample [9].

NSOM Instrumentation and operation

To date, several constructive schemes of a near-field optical microscope are used. The NSOM configuration capable to work in liquid is shown schematically in fig. 2 [10]. The most frequently implemented scheme is in which the optical radiation of a laser is localized in space using a fiber probe [11]. Such a scheme makes it possible to obtain the maximum radiation power in the region of the subwave hole. To increase the sensitivity, a photodetector collects the radiation produced from the studied sample by focusing lens. In addition, this NSOM configuration is also commonly adopted in near-field optical lithography examinations [12].

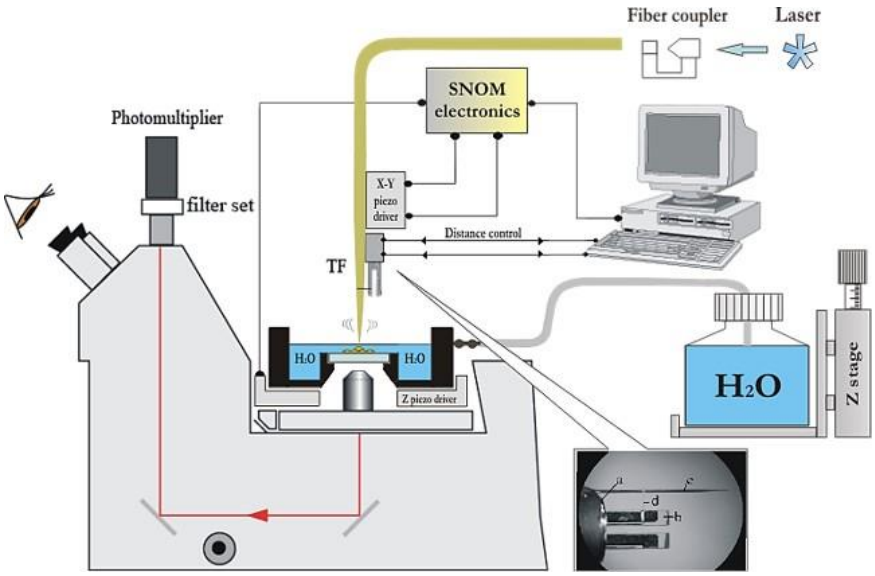

Figure 2. *NSOM configuration schematics* [10].

The main two components of an optical fiber are a core and a cladding. Outside, the fiber is coated with a protective layer. The core and shell are usually composed from quartz glass. It should be pointed out that the glass consumed in the core has higher refractive index compared to that of the shell [10,12]. (In practice, the refractive index of glass is controlled using alloying additives, so that the refractive indices of the core and shell differ by about 1%). Such a system, due to the phenomenon of total internal reflection, makes it possible to restrain optical radiation in the core area and to carry it over long distances with almost no loss [13].

For NSOM to work, it is crucial to bring the probe at a distance of 10 nm or less. There are various solutions to this problem, however, NSOM with the so-called "shear force" method of controlling the distance between the probe and the sample is most widely used. Most often, shear-force control circuits are used using a piezosensor based on a tuning fork type quartz resonator [14]. The NSOM probe is attached to the quartz resonator with glue. When the resonance frequency of the forced vibrations of the sample surface is almost equal to that of the probe - quartz resonator system, it is excited using an additional piezo-vibrator. In this case, the surface of the sample oscillates perpendicularly to the tip of the probe. In order to measure the force of interaction of the probe with the surface transformations in the phase and amplitude of the bending vibrations of the quartz resonator at the excitation frequency are recorded [15]. Since the concept of shear force control is quite compound, we restrict ourselves to only approximate considerations [16].

In order to test the operation of NSOM in air TGX01 calibration grating was used. It is made as chessboard with step height of 1um. From the figure 3, and 4 you can observe the three-dimensional topographical image and the cross-section profile of the calibration grating. The given step height of approximately 1um was proven. Also, using the specified tool Femto Scan Online, 3D topographical image of same calibration grating was constructed. However, testing NSOM operation in liquid remained as an uncompleted challenge for us. Many attempts were done in order to get thin water layer covering the TGX01 calibration grating, however due to its hydrophobic surface it was impossible to get the sensor adjusted as for the stable work water level was too deep. In order to get high resolution image NSOM needs to scan the sample for a quite long. For instance, to scan the sample with a surface area 12x12um and resolution of 300p it can take up to 2-3 hours. So, the biggest challenge was to keep the water for the entire scan duration. Moreover, scan time proportionally depends on the area, whereas the maximum scan area can get up to 40x40um.

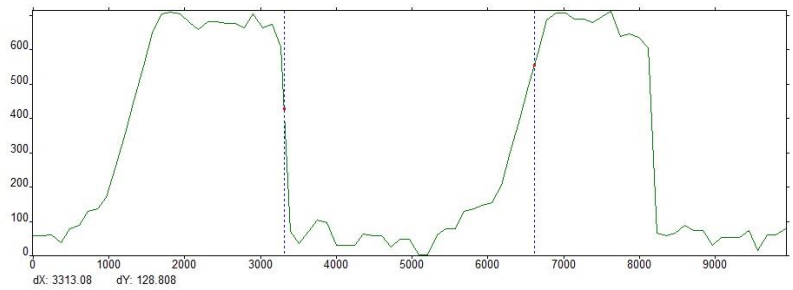

Figure 3. *Object cross-section profile with an average height of 800nm.*

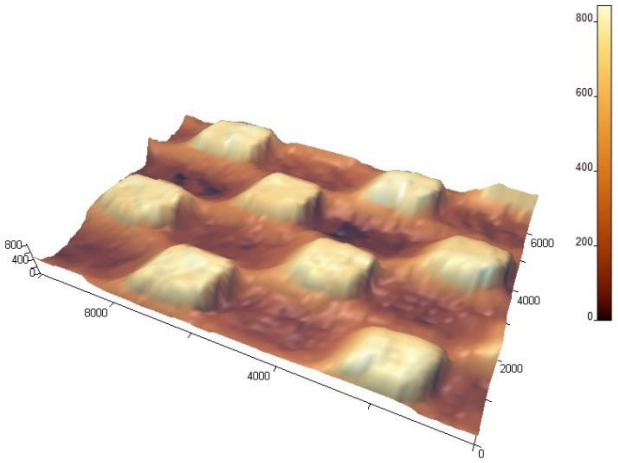

Figure 4. *Three-dimensional topographical image of same calibration grating.*

Scanning probe spectroscopy of nanometer spatial resolution

Combined scanning probe microscopes and spectrometers, integrating the methods of high-resolution measurements of topography and various physical properties of surface structures, have developed rapidly [17]. Devices make it possible to obtain information both on the substantial attributes of surface structures and on the qualitative configuration of the surface from the data of luminescent spectroscopy, Raman spectroscopy and high-resolution infrared spectroscopy [18].

Currently, the first versions of devices for infrared near-field microscopy combined with atomic force microscopy with lateral resolution up to 10 nm have been created. As a source of infrared radiation, a CO2 laser and a Michelson interferometer which is able to tune along the wavelength between 10.3–10.8 μm is currently used [17]. Conductive coated probes are used to initiate scattering. When the probe gets close to the surface of the sample, system catches the inelastic scattering which is moderated by the oscillative radiation.

The use of such systems makes it possible to detect changes in the dielectric constant of samples, as well as inelastic interaction signals due to the excitation of vibrational modes of molecules on the surface of a sample [19]. Additional improvement of instruments, including the potential of atomic force microscopy and spectroscopy, engages the combination of AFM, luminescent and Raman spectroscopy and NSOM methods with the expansion of the spectral range of the latter using cascade lasers [17,20]. This will make possible to obtain complicated data about the topography and various physical properties of surface structures, as well as about the chemical composition of surface layers [17,20].

Near field scanning optical microscopy imaging in liquid

The authors [22] used a near-field scanning microscope with two scanners, providing movement in the x, y plane with a resolution of 2.5 microns to study biological objects. An anodized aluminum plate was used to prevent a short circuit and reduce the effects of external fields on the probe. As a model water was used to explain the properties of biological objects, citing the fact that the dielectric properties of most biological objects are determined by the water contained in them. To study these properties, the shift of the resonance frequency f_r and the change in Q using a near-field scanning microscope were measured. As the end of the probe approaches water, fr and Q decrease. This decrease comes from the values of f_{r0} and Q_0, equal to 1413 MHz and 930, respectively, and characteristic of air in the absence of liquid. Using the microscope described above, an image of superficial vessels in bone tissue was obtained (see fig. 5).

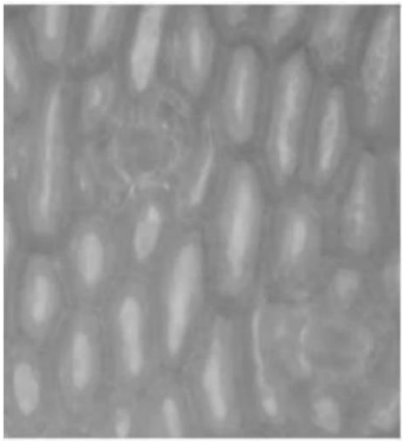

Figure 5. *Superficial vessels in bone tissue* [22].

This became possible due to the fact, that the bulk of the fluid in the vessels has a significantly higher dielectric constant than bone tissue. The values of f_{r0} and Q_0 were 1409 MHz and 800, respectively. The authors [22] also cited the results of measuring the properties of a NaCl solution. At the same time, they used a probe with a tip diameter of 240 μm to avoid the effect of surface tension on the measurement result. Variation of f_r and Q depending on the concentration of NaCl in deionized water from small values of concentration to the state of saturation of f_{r0} and Q_0 in the absence of NaCl were 1389 and 800, respectively. With an increase in the concentration of NaCl, the total conductivity of water increases; as a result, the real and imaginary parts of the complex dielectric constant increase and f_r and Q decrease at the same time. Such a dependence on Q is explained by a decrease in losses in the material with an increase in its conductivity. As a result of the studies, it is concluded that the near-field microscope can be an important appliance for studying the dielectric properties of biological objects.

NSOM ability to sense the NaCl concentration submerged to the aqueous solution

The authors [23] showed the possibility of using a near-field microscope to record small changes in the concentration of NaCl in an aqueous solution by changing the dielectric constant. For this purpose, invasive devices were commonly used. The microscope proposed by the authors [23] contains a dielectric resonator. The operating frequency of the microscope is ~ 4 GHz. The distance of the tip of the probe submerged to the solution was about 1 μm. Samples with the solution were installed on a platform that provided movement of the sample with the solution in three coordinates. A TE011 type of oscillations was excited in the resonator; the Q factor of the resonator was about 2400. The maximum NaCl concentration was 10 mg/ml. The minimum detectable concentration value was 0.005 mg/ml. The volume of the measured solution was 50 μl. As a result, a change in the nature of the resonance curve was observed with increasing NaCl concentration.

Combination of NSOM with Atomic Force Microscopy

The authors [24] described in detail the design of a near-field scanning optical microscope united with an atomic force microscope and used the device they created to simultaneously obtain images of the same nano-objects in various ways. This gave them the opportunity to compare the information received and identify additional opportunities for describing the characteristics of the objects of study. The authors [24] note that measurements using an atomic force microscope in this case serve as a kind of justification for the reliability of measurements using a near-field microscope. As an example, the image of breast cancer cells on a glass substrate was obtained using their device (see fig. 6). The image obtained using an atomic force microscope shows the topography of a cell with a pronounced structure near its center, showing the environment of the nucleus. Images using a near-field microscope obtained by detecting the amplitude or phase opens up the possibility of analyzing various aspects of the properties of the cell, in particular, the phase image highlights the cell membrane, the amplitude image contrasts its core.

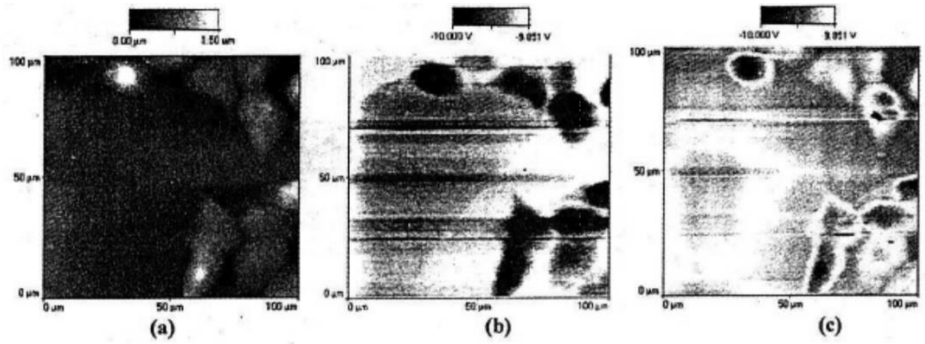

Figure 6. *a) Phase image. b) Amplitude image. c) Topography of a cell* [24].

Conclusion

The creation of near-field scanning microscopes has opened new possibilities for studying the properties of materials and structures, in particular those used in bioengineering. Compared to traditional optical microscopes, with the help of near-field scanning microscopes it is real to envision the form of an object under the surface. The resolution of near-field microscopes in the coordinate space is units of nanometers, which is comparable with the capabilities of atomic force microscopes. Each of the considered types of near-field microscopes can have its own area where its use is most rational. The widespread use of near-field microscopes can be facilitated by the identification of those objects that are significant for science and practice, where their advantages are unique and allow to determine parameters that cannot be measured using other methods. Direction of further researches should be the search for such applications.

References

1. D.W.Pohl, W.Denk, M.Lanz – Optical spectroscopy: image recording with resolution λ/20. // Appl. Phys. Lett., v. 44, p. 651 – 653 (1984).
2. U.Durig, D.W.Pohl, F.Rohrer – Near-field optical-scanning microscopy. J.Appl. Phys. 59 (10) 3318-3327, 1986.
3. P.K.Wei, W.S.Fann – The probe dynamics under shear force in near-field scanning optical microscopy. // J. Appl. Phys., v. 83, № 7, p. 3461 – 3468 (1998).
4. D.G.Volgunov, A.V.Buryukov, S.V.Gaponov, V.L.Mironov - Probe - surface interaction in the piezo-resonator "shear force" microscope. // Physics of Low – Dimensional Structures, № 3/4, p. 17-23 (2001).
5. D.Courjon, C.Bainier – Near field microscopy and near field optics. Rep. Prog. Phys. 57, p. 989 – 1028, (1994).
6. Bohm, C., Bangert, J., Mertin, W. and Kubalek, E. Time resolved near-field scanning optical microscopy. *Journal of Physics D: Applied Physics* 27: 2237-2240 (2018).
7. Betzig, E., Fin, P. L. and Weiner, J. S. Combined shear force and near-field scanning optical microscopy. *Applied Physics Letters* 60: 2484-2486 (2019).
8. Dearo, J. A., Weston, K. D. and Buratto, S. K. Near-field scanning optical microscopy of nanostructures. Phase Transitions 68: 27-57 (2020).
9. Gan, Q., Song, G., Yang, G., Xu, Y., Gao, J., Li, Y., Cao, Q. and Chen, L. Near-field scanning optical microscopy with an active probe. Applied Physics Letters 88. 2020.
10. Dukenbayev K., Sekatskii S. K. Serebryakov D. V. Zayats A. V. and Dietler G., *Proceedings of the SPIE*, 6728. 2007.
11. Cherkun A. P., Serebryakov D. V., Sekatskii S. K., Morozov I. V., Letokhov V. S., *Rev. Sci. Instr*., 77, p. 033703 (2006).
12. Lei F. H., Angiboust J.-F., Qiao W., Sockalingum G. D., Dukic S., Chrit L., Troyon M. and Manfait M., *J. Microsc*., 216, p. 229 (2004).
13. Taylor R. S., Vobornik D., Lu Z., Chisholm R. A. and Johnston L. J., *Jour. Of Appl. Phys.*, 107. 2010.
14. Hecht, B., Sick, B., Wild, U. P., Deckert, V., Zenobi, R., Martin, O. J. F. and Pohl, D. W. Scanning near-field optical microscopy with aperture probes: Fundamentals and applications.Journal of Chemical Physics 112: 7761-7774. 2018.
15. Hausmann, M., Perner, B., Rapp, A., Wollweber, L., Scherthan, H. and Greulich, K. O. Near-field scanning optical microscopy in cell biology and crytogenetics. Methods in Molecular Biology. 2015.

16. asdA. G. T. Ruiter, J. A. Veerman, K. O. van der Werf, and N. F. van Hulst, Appl. Phys. Lett. 71, 28. 1997.
17. Alexander J., Magonov S. (2015). High-Resolution Imaging with Different Combination of Tunneling Microscopes. NT-MDT Application note, Vol. 88.
18. Taha H., Strinkovski A., Manevitch A., Khatchatouriants A., Dekhter R., Ammann E. and Lewis A., *Nature Biotechnology*, 21, p. 1378 (2013).
19. Ambrose, W. P., Goodwin, P. M., Martin, J. C. and Keller, R. A. Alterations of single molecule fluorescence lifetimes in near-field optical microscopy. *Science* 265. 2018.
20. Westphal V. and Hell S. W., *Phys. Rev. Lett.*, 94, p.143903 (2005).
21. Kaupp, G. Scanning near-field optical microscopy on rough surfaces: Applications in chemistry, biology, and medicine. International Journal of Photoenergy. 2006.
22. Lofl and S.E., Mazzatenta J.D., Croman J., Tyagi S.D. Multimode near-fi eld microwave monitoring of free water content of skin and imaging of tissue // Phys. in Med. and Biology. 2012. Vol. 52.
23. Babajanyan A., Kim J., Kim S., Lee K., Friedman B. Sodium chloride sensing by using a near-fi eld microwave microprobe // Appl.Phys. Lett. 2016. Vol. 89.
24. Tabib-Azar M., Wang Y. Design and Fabrication of Scanning Near-Field Microwave Probes Compatible With Atomic Force Microscopy to Image Embedded Nanostructures // IEEE Trans. MTT. 2014. Vol. 52.