Advanced Characterization Techniques in Optics for Nanostructures

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The developments in nanotechnology present an outstanding challenge to characterization (measurement) technology by requiring nm-scale 3-D measurement capabilities. While the technology for synthesis has rapidly advanced, optical characterization of nanostructures is still in its infancy. Our NIRT Program [1] is building on the existing expertise and infrastructure at Boston University and University of Rochester and developing a toolbox of novel nano-optical characterization techniques to discover and understand the novel properties of nanostructures. **Solid immersion lens** (SIL) microscopy techniques combined with **enhancement with nanoscale metal-tips** will provide unprecedented resolution for spectroscopy of quantum dots and other semiconductor systems. The ultimate goal of the proposed program is to develop robust and efficient optical techniques at a spatial resolution on the order of 10 nm.

Optical spectroscopy provides a wealth of information on structural and dynamical properties of materials, especially when combined with high-resolution microscopy because the spectral features can be spatially resolved. However, there are fundamental limitations of conventional microscopy. In case of imaging objects with optical fields propagating to the far-field, the basic constraint is the diffraction of light, which limits standard optical microscopy to a spatial resolution comparable to the wavelength of light. For imaging objects through a substrate, which is opaque for short wavelengths, this limitation becomes more stringent. Reducing the wavelength or increasing the collected solid angle can improve the spatial resolution of surface microscopy. We have recently developed novel techniques based on a Numerical Aperture Increasing Lens (NAIL) to study semiconductors at very high spatial resolution. [2] The NAIL is placed on the surface of a sample and its convex surface effectively transforms the NAIL and the planar sample into an integrated solid immersion lens increasing the NA by a factor of square of the index n, to a maximum of NA = n corresponding to NA=3.6 in Si. Figure 1 shows inspection images of Si circuits fabricated by 180nm and 130nm technologies, displaying the striking improvement provided by the NAIL technique. Using an optimized confocal system we demonstrated lateral spatial resolution of approximately 200 nm. The spatial resolution improvement laterally is about a factor of 4 while longitudinally it is at least a factor of 12.5 corresponding to an overall reduction of the volume of interrogation by a factor of 50. One of the important features of NAIL microscopy is improved light collection efficiency (scales with the square of NA), particularly important in the study of quantum dots as well as a variety of semiconductor failure analysis modalities including thermal imaging. [3,4]

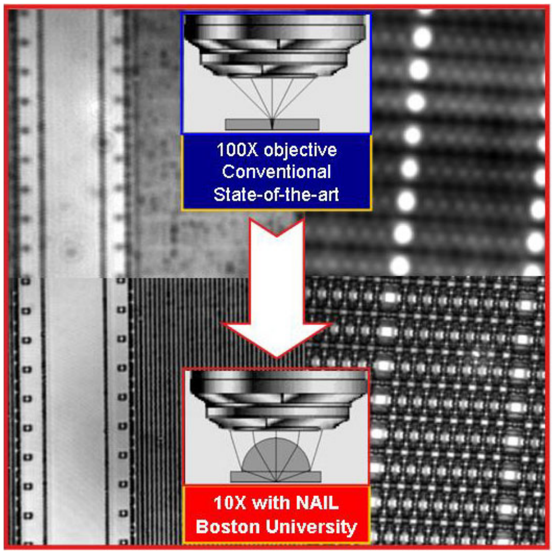

Fig. 1. Qualitative comparison of the images [2] displays the NAIL technique's striking improvement over state-of-the-art resolution.

While the BU team is working on the SIL microscopy, University of Rochester group develops a new technique to confine radiation to length scales much smaller than the diffraction limit of light. We use a sharp metal tip to localize optical radiation near the apex of the tip (Fig. 2(a)). The localized field provides a highly confined photon source for optical interactions with a sample in close proximity allowing for acquisition of a spectroscopic image with a spatial resolution defined by the sharpness of the tip. As part of the NIRT program and with funds from other NSF sources we unambiguously proved the feasibility of this imaging technique and recorded the highest resolution optical images of carbon nanotubes [5] demonstrating variations of the vibrational modes with a spatial resolution of 13nm. An example of these measurements is shown in Fig. 2 comparing a standard confocal image of carbon nanotubes with diffraction-limited resolution of ~300nm (b) to an image of the same sample area obtained with the tip technique (c) demonstrating a spatial resolution of 20nm. Note that the entire vibrational spectrum is recorded for each image pixel and thus it is possible to obtain multispectral images corresponding to various vibrational modes.

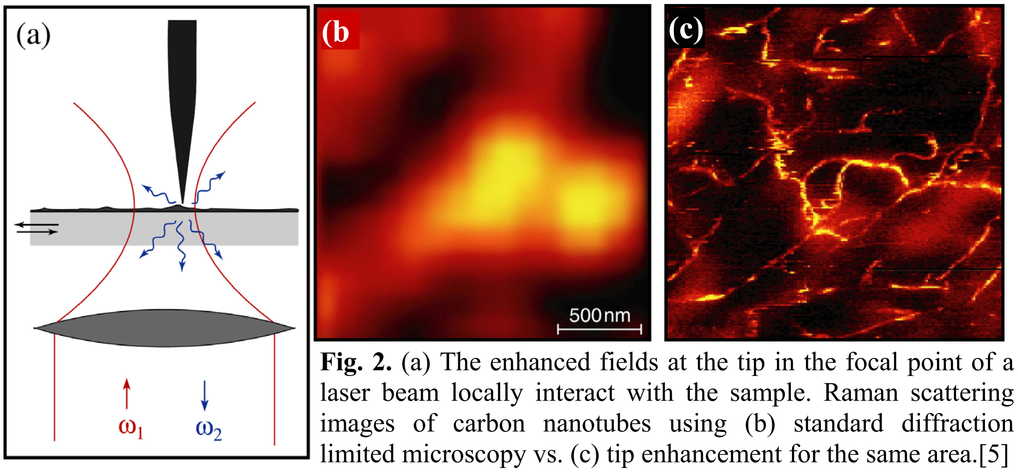

Fig. 2. (a) The enhanced fields at the tip in the focal point of a laser beam locally interact with the sample. Raman scattering images of carbon nanotubes using (b) standard diffraction limited microscopy vs. (c) tip enhancement for the same area.[5]

We are also applying the field-enhancement technique to optical lithography. This type of nanolithography is non-invasive since there is no mechanical contact between tip and surface. Furthermore, it relies on optical materials and techniques that have been developed by chip manufacturing industries over the past decades and hence it can be easily adapted to standard procedures. In our initial proof-of-principle experiments we first used a standard sharpened fiber tip to write a nanoscale pattern (see Fig.3). In the next step, the fiber probe has been replaced by a sharp metal tip and the photoresist has been exposed following the scheme by two-photon absorption using 150fs laser pulses at a wavelength of 800nm obtaining a feature size of 70nm.

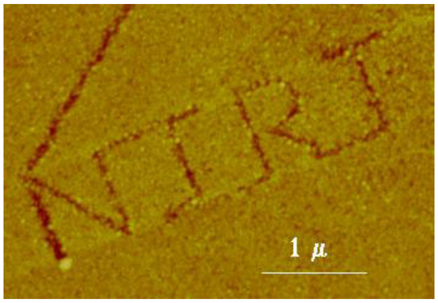

Fig. 3. AFM image of photoresist patterned by scanning a tapered fiber aperture on the surface with 100 pW of power from a GaN laser at λ=405 nm.

The ultimate goal in our program is to combine *SIL/NAIL techniques* with *tip-enhanced* near-field optical microscopy as depicted in the Fig. 4 and achieve spatial resolution in the order of 10nm with high collection efficiency as supported by theoretical studies. We are exploring both near-field optical lithography (Rochester) and electron-beam lithography (Mohanty-Ekinci at BU) techniques for fabrication of nm metal posts. Using an integrated nanofabrication technique with both hard and soft lithography, we developed a technique for creating metallic tips. Preliminary nanotips are 1μm high, sub-10 nm wide pillars of metals (Au, Al and Cr) on silicon substrate (Fig.4). We are also exploring the use of co-polymer templates for the fabrication of high-density arrays of nanotips in collaboration with NIRT at UMass Amherst (**Tuominen** and **Russell**).

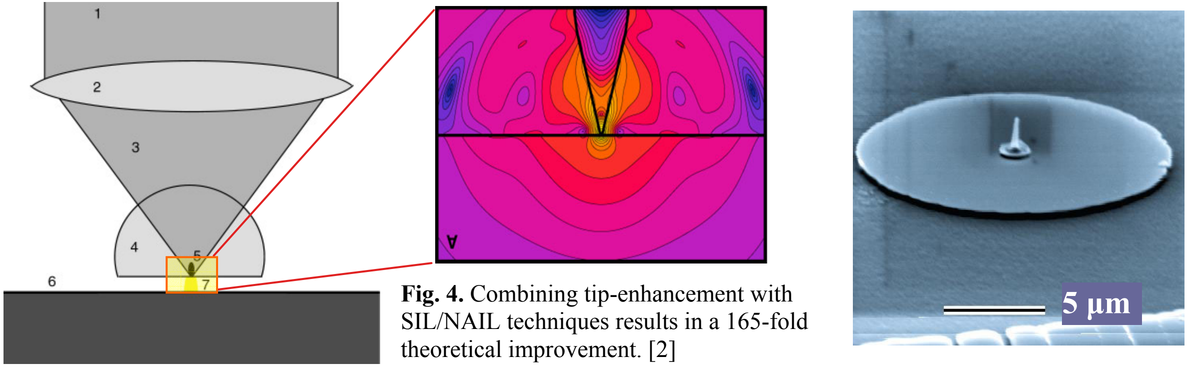

Fig. 4. Combining tip-enhancement with SIL/NAIL techniques results in a 165-fold theoretical improvement. [2]

We are also developing high-resolution imaging methods to study thermomechanical behavior of NEMS and materials on nanometer length scales on a wide range of systems including electronic components, and nanocomposite materials. Photothermal and photoacoustic microscopy- powerful tools for probing vibrational and thermal properties on the nanoscale- also suffer from limited spatial resolution imposed by the optical diffraction limit. The Laser Acoustics Lab (Murray) at BU developed a novel high-resolution photoacoustic and photothermal microscopy system using a GHz modulated DFB laser source for material excitation and phase-locked optical interferometry for detection of the resulting surface displacement in the fm range. [6] The excitation laser modulation frequency is scanned over the range of interest and the time domain signals are subsequently reconstructed allowing us to separate (temporally) various acoustic modes generated in the system. NAIL/SIL based probes will be incorporated into this system with the ultimate goal of studying the thermomechanical behavior of materials on nm length scale. Meanwhile, Ekinci's group is incorporating SIL imaging techniques in detection of NEMS displacements using optical interferometry. We have also fabricated and measured a series of nanomechanical beams with resonance frequencies reaching 1 GHz, fabricated metallic nanowires, [7] and studied high-temperature superconductivity in the nanometer scale. [8]

In addition to collaborating with US universities and other NIRT teams, we have a strong *international collaboration* including scientists from Switzerland, Austria, Turkey and Germany. We have already fabricated samples for thermal imaging at Swiss Federal Institute in Lausanne (EPFL) and we are currently working on spectroscopy of quantum dots in collaboration with researchers from ETH Zürich (Imamoglu) and University Linz, Austria (Bauer).

On the educational front, Ekinci has developed and taught a new graduate course titled *Topics in Nanotechnology* covering scanning probe microscopy, nanomechanics, and nanoelectronics. The enrollment of 2 Electrical Engineering, 2 Physics, 3 Mechanical Engineering and 5 Manufacturing Engineering students is a manifestation of the interdisciplinary nature of nanotechnology education. Our NIRT group supports a number of graduate and undergraduate students as well as high school students. We are working with the GK-12 program at BU (PI: Goldberg) for dissemination and outreach. Under the GK-12 program, high school teachers will work with researchers in the summer, and then continue their activities together within the existing GK-12 program, thereby bringing new research material into the classroom through new curriculum and new module development.

References

[1] For further information: http://ultra.bu.edu and http://www.optics.rochester.edu/workgroups/novotny/ . Please forward inquiries to *selim@bu.edu* , *goldberg@bu.edu* and *novotny@optics.rochester.edu*

[2] B. B. Goldberg, S. B. Ippolito, L. Novotny, Z. Liu, and M. S. Ünlü, "Immersion Lens Microscopy of Photonic Nanostructures and Quantum Dots," *IEEE J. Selected Topics in Quantum Electron.*, vol. 8, no. 5, pp. 1051, 2002. (invited)

[3] S. B. Ippolito, S. A. Thorne, M. G. Eraslan, B. B. Goldberg, Y. Leblebici, and M. S. Ünlü, "High spatial resolution subsurface thermal emission microscopy," IEEE/LEOS Annual Meeting, Tucson, AZ, October 2003. (invited)

[4] S. Thorne, S. B. Ippolito, M. Eraslan, B. Goldberg, M.S. Ünlü, and Y. Leblebici, "High Resolution Backside Thermography using a Numerical Aperture Increasing Lens," Proceedings of 29th International Symposium for Testing and Failure Analysis, 2-6 November 2003, Santa Clara, CA

[5] A. Hartschuh, E. J. Sanchez, X. S. Xie, and L. Novotny, ``High-resolution near-field Raman microscopy of single-walled carbon nanotubes," *Phys. Rev. Lett.* 90, 95503, 2003. (Also see NSF News Tip of April 16, 2003: http://www.nsf.gov/od/lpa/news/03/tip030416.htm)

[6] T.W. Murray, O. Balogun, and N. Pratt "High Frequency Laser Based Acoustic Microscopy Using a CW Generation Source," 16th World Conference on Nondestructive Testing, Montreal, Canada 2004

[7] P. Mohanty, R A. Webb, "High-field Measurement of Electron Decoherence in Metallic Nanowires: Switching off Magnetic Impurity Spins," *Phys. Rev. Lett.* 91, 066604 (2003)

[8] P. Mohanty, J. Wei, V. Ananth, P. Morales, and W. Skocpol, submitted to *Physica C* (invited)