Near-Field Scanning Nanophotonic Microscopy—Breaking the Diffraction Limit Using Integrated Nano Light-Emitting Probe Tip

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***Abstract*—We introduce a novel scanning "nanophotonic" microscope through monolithic integration of a nanoscale LED (Nano-LED) on a silicon cantilever. We review two recent trends of incorporating miniature light sources on the scanning probes for near-field scanning optical microscopy: one is to attach fluorephores at the tip to define a small light source, while the other is to integrate an LED and a nanometer aperture into scanning probes, based on silicon microfabrication techniques. The creation of Nano-LED combines the advantages of previous two approaches: no external sources are required and the reduction of the light source size directly leads to resolution improvement. Two types of Nano-LEDs have been successfully demonstrated utilizing nanofabrication and microelectromechanical systems technologies: 1) formation of thin silicon dioxide light-emitting layer between heavily doped p+ and n+ silicon layers created by a focused ion beam and 2) electrostatic trapping and excitation of CdSe/ZnS core-shell nanoparticles in a nanogap. We employed these probes into a standard near-field scanning and excitation setup. The probe successfully measured optical as well as topographic images of chromium test patterns with imaging resolutions of 400 and 50 nm, respectively. In addition, the directional resolution dependence of the acquired images suggests the size and shape of the light source. To our knowledge, these results are probably the first successful near-field images directly measured by such tip-embedded light sources. With the potential emission capability from near UV to IR and additional mass producibility, the nanophotonic microscope presents exciting opportunities in near-field optics, integrated circuit technology, nanomanufacturing and molecular imaging, and sensing in biomedicine.**

***Index Terms*—Fluorescence, LEDs, near-field scanning optical microscopy (NSOM), quantum dots (QDs), scanning probes.**

I. Introduction

NEAR-FIELD scanning optical microscopy (NSOM) overcomes the resolution limit of conventional microscopes by utilizing a miniaturized light source that tracks the sample surface [1]–[4]. A typical construction of the near-field scanning microscopy is shown in Fig. 1(a). A small light source located at the probe tip scans the surface, while the light source–sample interaction is recorded by a photodetector to form optical images. The photodetector is typically located under the sample with the inverted microscope configuration. Side-viewing microscopes are also commonly used to detect scattering light from the probe tip.

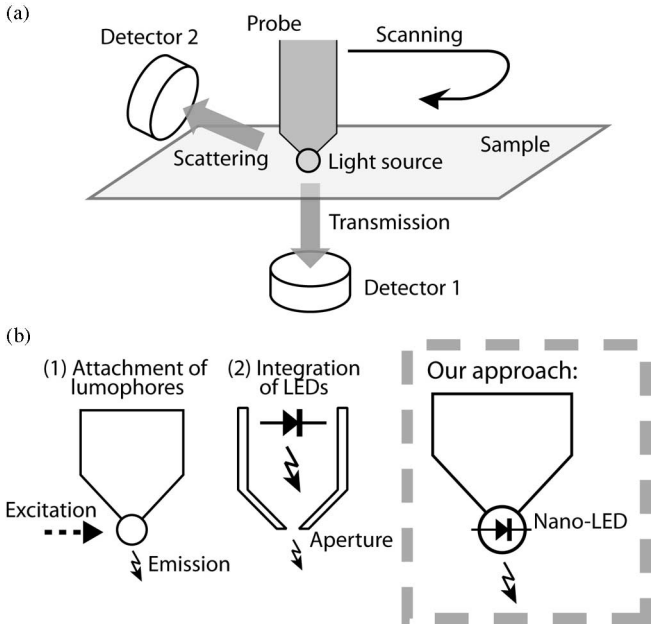

Fig. 1. Overview of the near-field scanning nanophotonic microscopy. (a) Schematic of a standard NSOM. (b) Integration of miniature light sources on the probe. Two conventional approaches include: (1) attachment of fluorescent light sources at the tip and (2) integration of an LED and an aperture. Our method is shown in (3) creation of Nano-LED at the tip.

The conventional approach of making a light source at tip has been made by using an optical fiber with a nanometer-sized aperture created through the metal coating [1]–[4]. The manual assembly process of the probe with an illuminating fiber has limited the throughput and large-scale operations of NSOM. In addition, an external light source is required for each probe, further increasing the system complexity. There have been several approaches to use alternative light sources instead of the fiber probes. Two major approaches are shown in Fig. 1(b). One approach is to attach a small amount of lumophore at the scanning probe tip [5]–[15], as shown in Fig. 1(b)-(1). The optical resolution of these systems is mainly defined by the diameter of the light source attached at the tip. This approach is similar to

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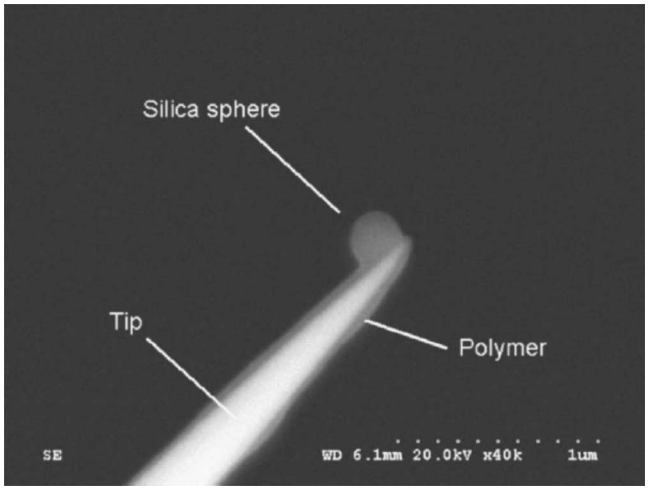


Fig. 2. Attachment of lumophores at the probe tip, where 200-nm-sized silica sphere covered with CdSe/ZnS QDs is attached to a scanning tip (From [5]. Reprinted with permission).

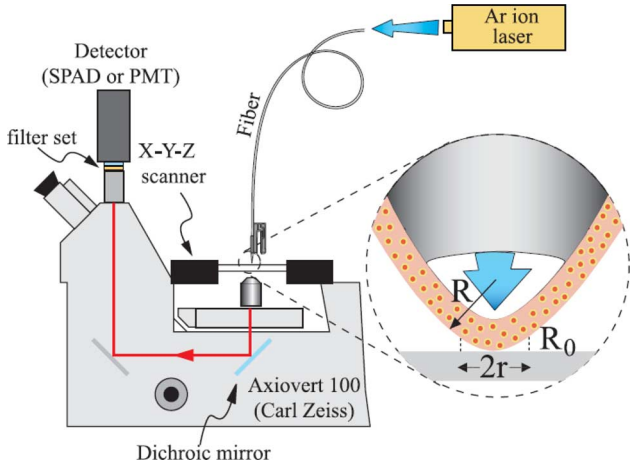


Fig. 3. Fiber-based excitation of the lumophores at the tip and the specimens under investigation (From [9]. Copyright (c) 2003, Blackwell Publishing Ltd. Reprinted with permission). CdSe QDs were attached at a fiber tip and excited by an Ar ion laser. Emission from the particles is used to excite fluorephores at the sample for imaging.

apertureless NSOM [16]–[19], which utilizes plasmonic effect of the metal coatings/particles at scanning probe tips. The lumophore is typically excited by an external laser light sources. The low signal intensity to the background excitation light has been the main problem.

Another approach is to integrate LEDs [20]–[22], as shown in Fig. 1(b)-(2). This approach is often associated with microelectromechanical systems (MEMS) technology and the light source is integrated onto a silicon micromachined scanning probes as the ones that can be found in atomic force microscopy (AFM). The light sources are integrated into the probes and no external light sources are required for these probes. Nanometer-scale apertures, as in typical fiber-based NSOMs, are used to define the size of the light source at the tip. Large energy dissipation at the probe tip causes thermal instability on the probe.

We have developed a novel NSOM through monolithic integration of a nanoscale LED (Nano-LED) on a silicon cantilever [23], [24], as shown in Fig. 1(b)-(3). This method combines the advantages of earlier described approaches: the resolution is associated with the size and position of created light source and no external sources are required. In this paper, we start from reviewing fist two approaches, followed by illustrating our new approach and its benefits.

II. Integration of On-Tip Light Sources: Two Conventional Approaches

A. Attachment of Lumophores at the Tip

In this approach, a small amount of lumophore is attached at the scanning probe tip.

First, fluorescent markers are used to modify the tip of a scanning probe. Then the tip modified with fluorephores mechanically approaches the sample, controlled with nanometer-scale precision of scanning microscopy. Fig. 2 shows an example of particle attachment to a scanning probe [5]. A silica sphere coated with CdSe/ZnS core-shell quantum dots (QDs) is glued to a probe tip. Similar attempt for attaching a fluorescent bead on a force sensing cantilever was made by Kan *et al.* [6]. Other approaches for adding lumophores at tip have also been made. Wang *et al.* have used polymerization of photosensitive epoxy precursor containing colloidal CdSe–ZnS nanoparticles to fabricate a fluorescent fiber tip [7]. Cuche *et al*. have deposited fluorescent oxide nanoparticles on a standard optical tip through low-energy cluster beam deposition. The tip was validated through successful NSOM imaging [8].

In this approach, both the lumophores at the tip and the specimens under investigation need to be excited by an external source in a two-step process [9], as shown in Fig. 3. First, the lumophores at the tip are excited by an Ar ion laser to emit fluorescent light. Then, this emission excites the fluorephores at the sample for observations. Fluorescence energy transfer (FRET) occurs due to the proximity of the tip and sample configuration, which warrant the donor–acceptor distance to be smaller than 5–10 nm. In this case, lumophores at the tip work as the donors while those of the sample work as the acceptors. The same group used a reversed configuration to perform a single-molecule FRET imaging [10]. A single CdSe/ZnS nanoparticle on the substrate was used as the donor, while a fluorescent dye attached at the fiber tip worked as the acceptor. Similar reversed configuration for FRET has also been used with an AFM tip by Vickery and Dunn [11].

The common idea to these studies is to reduce the number of molecules acting in the imaging. Michaelis *et al.* [12] exploited the narrow zero-phonon spectral lines of the terrylene molecules at low temperatures. By tuning the laser frequency, they excited one of the several terrylene molecules attached at the end of an optical fiber to achieve imaging with a single molecule. Chevalier and coworkers coated a fiber-based NSOM probe with a diluted solution of QDs to attach a very small number of QDs in [13] and [14]. The key is to improve the resolution to the order of the single molecule. However, in many cases, lumophores tend to be randomly attached, and the effective distribution of the lumophores for imaging is less clear. In addition, due to

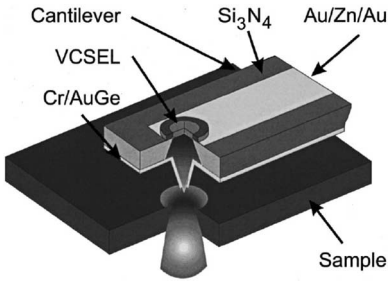


Fig. 4. Integration of laser diodes, such as VCSEL, with micromachined cantilevers (From [20]. Reprinted with permission).

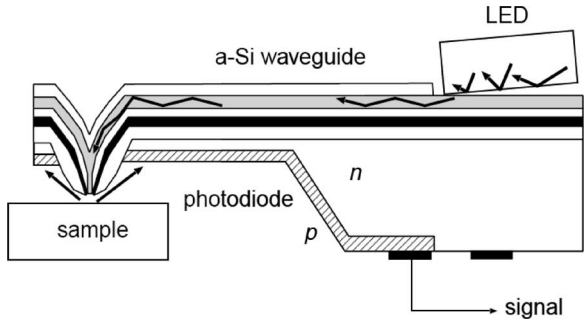


Fig. 5. Coupling of LED to a scanning probe integrated with waveguide, aperture, and photodiode (From [21]. Reprinted with permission).

the rather inefficient two-step excitation configuration, the low signal intensity relative to the background excitation light can also present problem for imaging. Precise control of the light source–sample distance, i.e., donor–acceptor distance, is therefore a critical issue in obtaining efficient energy transfer, and thus, a better optical signal. We recently developed a nanoscale stamping technique of fluorescent colloidal QDs [15]. Nanoparticles were deposited on silicon probe tips with a force sensing quartz tuning fork as the stamp pad. Nanoparticle attachment and manipulation has broad applications not only for near-field imaging, but also for QD-based nanoelectronics [25], [26], magnetometry at the nanoscale [27]–[29], and biochemical sensors [30], [31].

B. Integration of LED

The light sources can be integrated into the probes and no external light sources are required for these probes. This approach has been made in attempt to fabricate totally integrated scanning optical microscopes for optical data storages [20], [21] or nanoscale patterning tools [22]. LEDs or laser diodes are integrated with micromachined cantilevers. As shown in Fig. 4, Heisig *et al.* integrated a vertical-cavity surface-emitting laser (VCSEL) in a GaAs cantilever and performed optical imaging [20]. The aperture was made by thermal metal evaporation process, which is same as typical fiber-based optical probes. GaN LED was also assembled with aperture-based silicon scanning probe to perform patterning of photoresist [22].

Nanometer-scale apertures, as in typical fiber-based NSOMs, are used to define the size of the light source at the tip. Focused ion beams (FIBs) have been a popular technique to define apertures on silicon-based micromachined scanning cantilevers [32], [33] as well as on standard fiber probes [34]. Minh *et al.* demonstrated the combination of oxidation and selective etching for aperture definition [35]. The tip is formed by nonuniform oxidation of silicon and the aperture is created by selective etching of SiO_2 in a buffered-HF solution. As shown in Fig. 5, Sasaki *et al.* used this technique and integrated a waveguide, photodiode, and LED into a scanning probe to perform optical imaging with a 200 nm resolution [21].

The light sources used in these probes were made by standard fabrication techniques of LEDs or laser diodes, and were significantly larger than were required for subwavelength resolution near-field scanning. Although additional optical elements such as microlenses can be used to assist focusing the incident light [22], the apertures made through metal films essentially define the optical resolution. In addition, large energy dissipation at the probe tip causes the thermal instability on the probe.

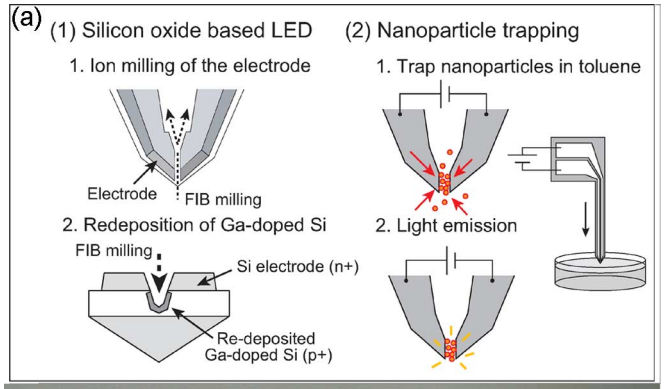
III. Nano-LED on Scanning Probe Tip

We have developed a new approach for creating a Nano-LED to work as a scanning light source [23], with the first successful optical scanning imaging using such tip-embedded light source [24]. With the miniature light source created at the tip, components such as waveguides and apertures previously considered essential in NSOM probes are no longer needed. Similar attempt has also been considered with organic LEDs (OLEDs) on silicon cantilevers [36], [37]. This method combines main benefits associated with the other two approaches examined in this paper. First, creation of "apertureless" small light sources will directly lead to resolution improvement. Second, the light source is built-in on tip and a compact system can be made without any external light sources.

A. Design and Fabrication of Nano-LED

We have tested two types of Nano-LEDs created at the silicon probe. The main body of the scanning probe for both designs is made by wet-etching of single crystal silicon wafers. Since the probe body retains the flat top surface of the original wafer, several methods for creating LEDs can be easily employed. We have utilized a series of nanofabrication and MEMS technologies to form the light-emitting probe tip on silicon, as shown in Fig. 6: 1) formation of a thin silicon dioxide layer buried between a phosphorus-doped n+ silicon layer and a gallium-doped p+ silicon region, created locally at the tip by an FIB and 2) electrostatic trapping and excitation of semiconductor nanoparticles, such as CdSe/ZnS core-shell QDs at the probe tip.

1) Silicon-Oxide-Based Nano-LED: A silicon on insulator (SOI) wafer with a 2-μm-thick device layer was used. The device layer was prepatterned before the etching of the probe body and utilized as the LED electrodes. The device layer was doped by phosphorous oxychloride for 50 min at 900 °C. The probe tip and the electrodes were trimmed with an FIB so that a 150-nm-wide gap on the electrode tip converges to the very tip of the probe body. This milling process induces redeposition



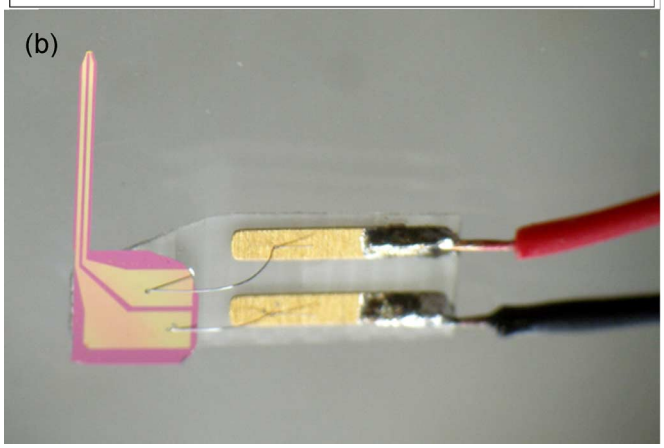


Fig. 6. Design and fabrication of Nano-LEDs on the probe tip. (a) Two methods of Nano-LED integration. (1) Silicon probe is directly doped with FIB and (2) CdSe nanoparticles are trapped at the FIB-created nanogap. (b) Photograph of the nanophotonic scanning probe.

of gallium-doped p-type silicon, which eventually creates a p-n junction [38] with a thin silicon dioxide layer as the light emitter [39]. The LED was typically driven at 4–8 V and emitted light centered at 560 nm.

2) Electrostatic Trapping of Semiconductor Nanoparticles: Additional semiconductor nanoparticles at probe tip were used as the light source. The same probe as in Section III-A1 was used. The electrodes were then immersed in the toluene solution of CdSe/ZnS core-shell QDs to trap the nanoparticles. When the voltage (typically 5–10 V) was applied, the nanoparticles were polarized and attracted to the gap along the electric field gradient. With the QDs trapped at the nanogap, driving voltages of the LED was typically 10–20 V, larger than the ones without QDs. Electroluminescence from the Nano-LED with trapped QDs is shown in Fig. 7.

One advantage of nanoparticle-based LED [40], [41] is that the emission wavelength can be tailored by changing the diameter of the deposited nanoparticles. Emission spectra were measured for the two types of the LEDs (Fig. 8). For the cases with electrostatically trapped CdSe nanoparticles in case 2), we found several small emission peaks that were not found in case 1). The peaks did not correspond to the designed emission peak (560 nm) of QDs, possibly due to the aggregation of particles sintered to form larger particles [25] at the emitting locations.

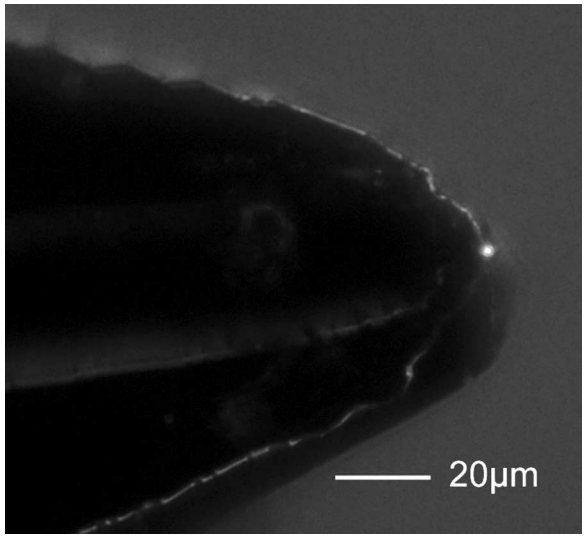


Fig. 7. Electroluminescence from QDs trapped to the nanogap at the probe tip.

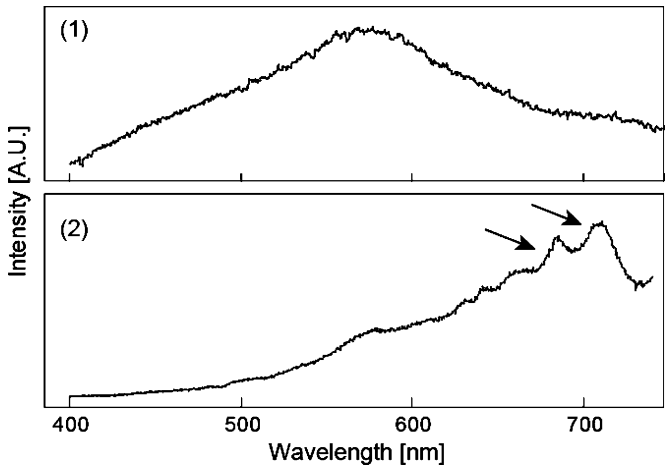


Fig. 8. Emission spectra of the two types of Nano-LEDs: (1) silicon-dioxide-based LED. (2) QD attached LED. Small additional emission peaks are found in (2), while both (1) and (2) have a common broad peak around 580 nm.

B. Imaging With Nano-LED on Probe

The probes were tested in a standard NSOM setup (Veeco Aurora) shown in Fig. 9(a). The silicon probe was attached to a quartz tuning fork as introduced by Karrai and Grober [42] and controlled in shear-force feedback to perform simultaneous topographic and optical imaging. A 30-nm-thick chromium pattern made on a glass substrate was used as a sample. Fig. 9(b) and (c) are the topographic and optical images taken with the type-1 probe. The imaging was successful to reveal several characteristics of the Nano-LED and the tip. Multiple imaging of an edge in the topography in Fig. 9(b) is thought to be caused by relatively low aspect ratio of the probe tip.

Directional dependence of optical resolution was found in the optical image in Fig. 9(c), suggesting the actual size and shape of the Nano-LED at the probe. The bandwidth for 20%–80% intensity change in each triangle edge is also shown in Fig. 9(c). Fig. 9(d) is a topographic profile at a triangle edge

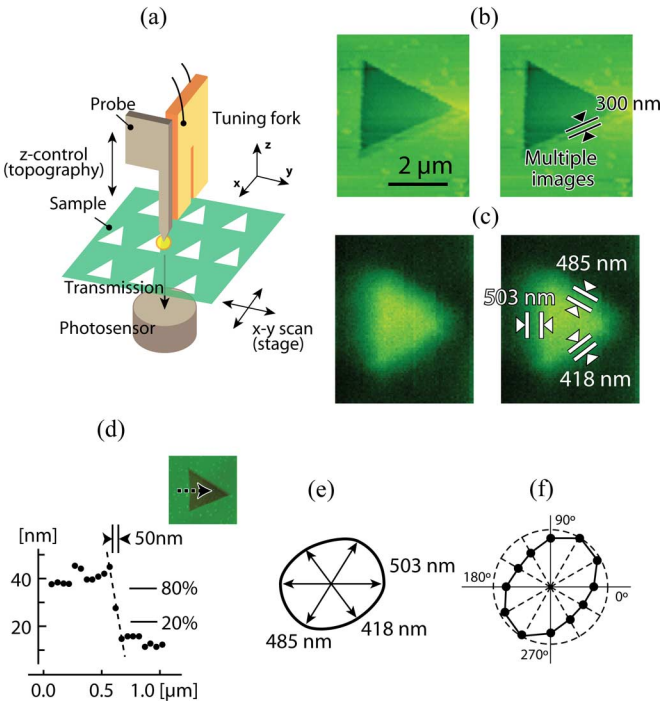


Fig. 9. Near-field scanning nanophotonic microscopy. (a) Schematics of the experimental setup. (b) Topographic images. Multiple images of an edge are visible. (c) Optical images. Directional dependence of the resolution was identified. (d) Topographic profile of an edge of a triangle pattern. (e) Nano-LED shape is estimated from the optical image. (f) Polarization of the Nano-LED measured with a rotating polarizer.

with the bandwidth for 20%–80% change, suggesting spatial topographic resolution to be 50 nm. On the other hand, multiple imaging of an edge was occasionally found as shown with a 300-nm-wide separation in Fig. 9(b), suggesting a relatively low aspect ratio of the probe tip. Fig. 9(e) shows an ellipsoidal shape of the Nano-LED estimated from the directional resolution dependence in the optical image. Since the sample has 30-nm-thick profiles rather than a complete flat plane, the topographic measurement may have acted as an artifact in the optical images. If we consider rather a large difference in the resolutions of optical and topographic measurements (400 and 50 nm, respectively), we can conclude that topographic interference in the optical image is less significant. Fig. 9(f) is the Nano-LED polarization measured with a rotating polarizer placed in front of the detector. Polarization measurement is important in applications such as thin-film characterization [43] or optical data recording [44]. The polarization in Fig. 9(f) was found to have a significant correlation with the Nano-LED shape in Fig. 9(e). Both the shape and the polarity of the Nano-LED are thought to be highly confined by the geometry of the nanogap created by the FIB. Similar geometrical confinement of the polarization was also found in nanoscale apertures at the probe tip [35].

IV. Conclusion

We have reviewed two major approaches of using alternative light sources for the NSOM. We have also introduced our recent progress in creating a Nano-LED at the scanning probe tip, which combines the advantages of the described two approaches. The Nano-LED does not require any external light source and the resolution is directly related to the light source size, which can be further improved in the design and the fabrication procedures. Optical and topographic resolutions of 400 and 50 nm, respectively, were demonstrated. The images can be used to interpret the shape and the size of the Nano-LED at the probe front.

Compared to the topographic resolution of 50 nm, the relatively coarse optical resolution of 400 nm can be further improved through reduction in the size and precise positioning of the LED at tip. For dimension and position control, several deposition techniques can be considered, as shown in [7]–[14], including our recent work on nanoscale stamping technique [15]. To better control the emission wavelengths, multilayer of charge transporting structures can be configured to obtain optimal hole–electron recombination at the QD light source, as demonstrated in [41].

With the potential emission capability from near UV to IR, the mass-producible, self-illuminating Nano-LED on-chip will open many exciting opportunities in biomedical and industrial applications, including near-field microscopy of subcellular structures, direct material patterning, ultrahigh-density data storage, and compact "light-on-chip" biosensors and biochips.

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