Tip-Based Nanofabrication: An Approach to True Nanotechnology

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ABSTRACT

True nanotechnology, defined as the ability to reliably and repeatably fabricate nanostructures with controlled differences in size, shape, and orientation at precise substrate locations, currently does not exist. There are many examples demonstrating the capability to grow, deposit, and manipulate nanometer-sized features, but typically these techniques do not allow for controllable manufacturing of individual structures. To bridge this gap and to unlock the true potential of nanotechnology for defense sensing applications, the Defense Advanced Research Projects Agency (DARPA) launched the Tip-Based Nanofabrication (TBN) research program with the intent of achieving controlled manufacturing of nanostructures using functionalized AFM cantilevers and tips. This work describes the background, goals, and recent advances achieved during the multi-year TBN program.

Keywords: nanotechnology, tip-based fabrication

1. INTRODUCTION

Nanotechnology, and the revolutionary advances in technology that it promises, are of great interest throughout industry, academia, and the Department of Defense (DoD). Loosely defined, nanotechnology refers to the science and engineering of features with sub-micron dimensions. While there is an almost-infinite list of possible geometries and materials, quantum dots, nanotubes, and nanowires have attracted significant attention due to their promise of revolutionizing the state of the art. These nanoengineered materials are key enablers for a multitude of civilian and defense sensing applications including, but not limited to, optical, biological, and IR sensors and quantum computers.[1-3]

The controlled manufacturing of nanostructures will ultimately be required to fully take advantage of nanotechnology. Controllable nanomanufacturing is defined as the automated, parallel fabrication of individual structures with control over position, size, shape, and orientation at the nanometer scale. There have been numerous demonstrations of the capability to grow, deposit, and/or manipulate nanostructures in recent years,[4] but these approaches generally suffer from significant deficiencies when viewed against the above-stated definition of controlled nanomanufacturing. Toward the goal of realizing true nanotechnology, the Defense Advanced Research Projects Agency (DARPA) initiated the Tip-Based Nanofabrication (TBN) research program in 2008. The objective of the TBN program is to initialize and mature hardware, capabilities, and technologies for the fabrication of nanostructures using parallel tip arrays; while the focus is on developing controllable manufacturing techniques using functionalized AFM cantilevers and tips, other methods and materials are also being explored, including optical and bio-inspired approaches. An additional goal of the program is to enable the *in situ* detection of the nanostructure position, size, shape, and orientation thereby allowing the structures to be repaired or remanufactured as needed.

This work describes the recent advances achieved by the performers on the DARPA Tip-Based Nanofabrication research program. At the end of the multi-year program, the resulting technologies are targeted for insertion in proposer-specified devices and applications. The background of the TBN program is presented in Section 2 followed by a description of the TBN requirements and goals in Section 3. The approaches and recent achievements of the TBN teams are highlighted in Section 4 followed by a discussion of future directions in Section 5.

2. TBN PROGRAM BACKGROUND

Currently, the controlled manufacturing of heterogeneous, individual nanostructures has not been realized. While there have been numerous demonstrations of the capability to grow, deposit, and manipulate nanometer-sized features, these

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methods generally do not allow precise control over the size, shape, position, and orientation of individual structures. For example, dense, aligned carbon nanotube forests can be fabricated using pre-growth lithography for substrate position control.[5,6] Methods such as this, however, do not provide the ability to grow individual nanotubes at user specified substrate locations with precise control over their orientation and dimensions. There also exists the potential to create large arrays of quantum dots with high uniformity,[7,8] but again, there is no ability to manufacture patterned arrays of heterogeneous quantum dots and no method to repair the nonuniformity that typically arises from the growth process. There are examples for capturing, manipulating, and placing individual nanowires into arrangements needed for device construction,[9,10] but these generally rely on very challenging methods for manipulation, metrology, and repair as well as a nearby cache of devices with suitable sizes and shapes. While these emerging approaches are indeed exciting, they do not appear to provide a path to controlled nanomanufacturing.

Recently, there have been significant efforts that have become the building blocks towards controlled manufacturing. Perhaps the most notable and successful is the IBM Millipede program.[11] The goal of this effort was to create a topographic data storage technology using large arrays of AFM cantilevers to form and detect indentations thermally. To date, parallel fabrication, detection, and modification of indentations with spacing of 18 nm between tracks, 9 nm within a track, and depth of 1 nm have been demonstrated. In addition to the Millipede program, the scientific community has decades of experience with macroscopic instruments for manipulation of AFM tips and has demonstrated the ability to extract signals and information from surfaces and interfaces through tip interactions.[12-14] Specifically, functionalized AFM cantilevers and tips have the ability to manipulate environments on the surface of a substrate at the sub-micrometer scale which can, in turn, be used to create high temperatures, high electric and magnetic fields, as well as rapid temporal and spatial variations of all of the above and more.[15-17]

Building upon the successes of IBM and the research community, there is a distinct opportunity for exploring the use of functionalized, parallel AFM tips as nanomanufacturing tools to enable the fabrication of nanostructure arrays. The Tip-Based Nanofabrication program attempts to capitalize on this prior work with the goal of bridging the gap toward true, controlled nanomanufacturing, essentially creating a machine shop at the nanoscale.

3. TBN PROGRAM GOALS

3.1 Program Requirements

The main objective of the Tip-Based Nanofabrication program is to develop hardware, capabilities, and technologies for controlled nanomanufacturing in order to develop a framework for delivering capabilities of much broader use. The TBN program also addresses a key aspect of nanofabrication: the ability to detect and repair defects. The tips not only serve as a fabrication tool, but also as a sensor thereby allowing a "measurement mode" as well as a "fabrication mode" of operation. The ability for the system to provide measurement *in situ* is an essential component so that the nanofabrication process may be scalable to true manufacturing.

Within this focus, the program has requirements specific to both the fabricated nanostructures and the manufacturing tool itself. Throughout the tenure of this multi-year program, progress toward meeting these requirements is measured by predetermined periodic milestones. Success in meeting the end-of-program goal of controlled nanomanufacturing will directly enable many important applications, and the capabilities developed for this subset of nanostructures should be extendable to many other structures, materials, and treatments.

3.2 Measuring Progress

Control over size, position, and heterogeneity of features are critical parameters for achieving the TBN program goals. The ability to fabricate a specific structure with specific dimensions is essential to unlock unique dimension-dependent properties in devices. This capability is generally absent in most existing nanostructure fabrication methods, and is regarded as a key goal of this program. The first milestone for measuring progress is feature size control of a dimension (length, diameter, radius, orientation) which starts at 10% and eventually decreases to 1% by the end of the program essentially excluding many of the presently-available methods for uncontrolled growth. Heterogeneity and position control are also important parameters for making real nanodevices and by the end of the TBN program, variations of at least two different parameters (such as size, shape, or orientation) to an accuracy of 1% is required at controlled locations to within 5 nm.

The tools necessary for controlled fabrication of nanostructures will require significant modification over instruments commonly used today. The efficacy of the nanomanufacturing tools developed on this program is assessed through fabrication rate, height sensing, and tip wear. Although automation is not required in the early stages of the program, the fabrication rate of at least 60 features per minute per tip was chosen to drive development of 2-axis scanning platforms with integrated sensing to enable high throughput. Sensors are also critical for detecting height above the surface; by the end of the program, the height of the tip above the surface must be sensed to an accuracy of 2 nm. In any localized manufacturing method, the stability of the geometry and characteristics of the tools is important; for tip-based methods, the size and shape of the tip is likely to be important for maintaining control of shapes and positions. The goal for wear and reliability on the TBN program is to achieve tip shape variations of less than 1% in height and 3% in radius after one million operations.

4. TBN APPROACHES

Success of the TBN program will usher in a new class of nanomanufacturing processes to realize practical nanoscale devices. Working toward this goal are ten teams funded under the TBN program, each with a distinct and innovative approach to controlled nanomanufacturing. These approaches range from STM or AFM based systems to laser-assisted optical approaches to fabrication using CVD and DPN. Table 1 presents a list of the main TBN performers and their initial approaches which is followed by a description of their more recent published accomplishments.

Table 1. The Tip-Based Nanofabrication performers and their proposed approaches.

Organization	Principle Investigator	Approach
California Institute of Technology	Michael L. Roukes, Professor of Physics, Applied Physics, and Bioengineering	Tip-Based Large-Scale Manufacturing of Functional Nanowire Circuits
Carnegie Mellon University	David S. Ricketts, Department of Electrical and Computer Engineering	Tip Directed Field-Emission Assisted Nanomanufacturing
Cornell University	Clifford Pollock, School of Electrical and Computer Engineering	Nano Optical Tether System for Precision Nanowires
Case Western Reserve University	Mohan Sankaran, Department of Chemical Engineering	Tip-Assisted Gas Phase Deposition on Insulating/Conducting Substrates
Lawrence Livermore National Laboratory	James J. De Yoreo	Bio-Mediated Synthesis and Thermally-Driven Reactivity to Achieve nm-Scale Templating
Northwestern University	Chad A. Mirkin, Department of Chemistry	Scanning Probe Epitaxy: Directed Synthesis of Nanostructures by a Moving Tip
Purdue University	Xianfan Xu, School of Mechanical Engineering	Nanoscale Bowtie Antenna Array for Controlled, Parallel Synthesis of Nanowire
University of California, Berkeley	Costas P. Grigoropoulos, Mechanical Engineering Department	Nanofabrication by Tips Coupled with Lasers
University of Illinois, Urbana-Champaign	William P. King, Department of Mechanical Science and Engineering	Carbon Nanotube Synthesis via Nanoscale Thermal Processing with Diamond Probe Tips
Zyvex Labs	John Randall	Atomically Precise Manufacturing

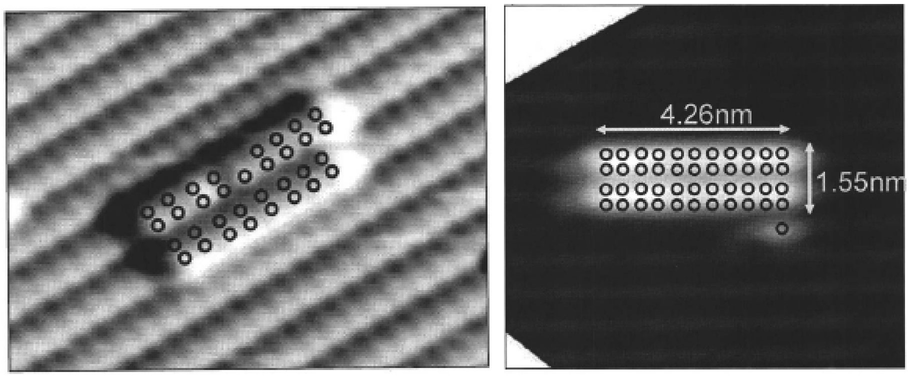

Figure 1. Passivated Si(1 0 0) 2 x 1 surface where H atoms have been removed by the STM tip. The circles indicate where the individual atoms have been removed. Image courtesy of John Randall, Zyvex Labs.

Scanning Tunneling Microscopy (STM) is commonly used to characterize nanoscale structures and its capabilities are being leveraged for nanomanufacturing by several of the TBN teams. The approach explored by Zyvex is to fabricate nanostructures with atomic precision. Si(1 0 0) 2 x 1 surfaces with a monolayer of H atoms are patterned using a process called H-depassivation lithography.[18] H atoms are individually removed from the surface when current from an STM probe excites an Si:H bond, stripping the H atom from the surface and leaving unsatisfied bonds. Figure 1 shows the passivated surface where the H atoms have been removed by the STM tip, exposing the unsatisfied bonds.[19] At these bonding sites, selective deposition of metals, oxides, semiconductors, and dopants can be accomplished by passing a precursor gas through the ultra high vacuum environment.[20] By applying this method sequentially, the Zyvex team will ultimately build nanostructures with atomic precision.

STM functionality is also employed by the Carnegie Mellon team. Their approach combines the resolution and control of STM with the multimodal chemistry capabilities of Chemical Vapor Deposition (CVD). Their Tip-directed, Field-emission Assisted Nanomanufacturing (TFAN) method starts with a silicon substrate coated with adsorbed precursor, such as disilane (Si_2H_6). An electron beam emitted from an STM tip then hits the surface, cracking the H atoms on the surface and leaving the Si behind. Then an additional precursor layer is absorbed on the Si sites, and the process is repeated. In this process, nanowires are built by stacking layers of silicon, the width of which is controlled by the magnitude of the field emission current.[21] The STM tip can be programmed to move across the surface to create user-defined patterns in virtually any configuration. This process can also be adapted for direct writing of oxides, other semiconductors (III and IV), and metals.[22] Carnegie Mellon is also extending the capability of this nanofabrication technique by scaling to microelectromechanical systems (MEMS)-based arrays of multiple STM tips, as shown in Figure 2. The probes are fabricated using post-CMOS MEMS processing, enabling the arrays to be integrated with circuits to achieve individual control and nanometer resolution.[22]

Other TBN teams are taking a chemistry-based approach with techniques including tip-based Dip-Pen Nanolithography (DPN) and Chemical Vapor Deposition (CVD). The University of Illinois Urbana-Champaign uses doped silicon atomic force microscope (AFM) cantilevers with wear-resistant ultrananocrystalline diamond (UNCD) tips with radii as small as 15 nm.[23] The tip is thermally controlled by integrated heater-thermometers on the microcantilever, controlled by an NPN back-to-back diode.[24] Using these heated tips, nanostructures can be fabricated in multiple ways. One is to dip the tip into a polymer-nanoparticle ink composite, heat it, then move it across a substrate. As the ink melts, it flows off the tip and deposits nanoscale (less than 100 nm) features onto a substrate. This flexible technique has been demonstrated with a wide range of polymers with organometallic molecules or metallic, semiconducting, or magnetic nanoparticles on a variety of substrates, including gold, mica, and silicon oxide. After writing, the nanoparticle features are functional, or may be further modified by oxygen plasma treatments. The left side of Figure 3 shows one example of this capability. A polyethylene-based ink was used to deposit CdSe/ZnS quantum dots onto the substrate; their functionality as quantum dots is demonstrated by their fluorescence.[25] The heated cantilever tips have also demonstrated local thermal reduction of graphene as shown on the right side of Figure 3. The team has shown the ability to tune the topographical and electrical properties of the graphene and produce variably conductive nanoribbons as small as 12 nm on epitaxial graphene films. Reliable nanofabrication of reduced graphene structures has enormous potential for a variety of devices ranging from sensors to mechanical resonators.[26]

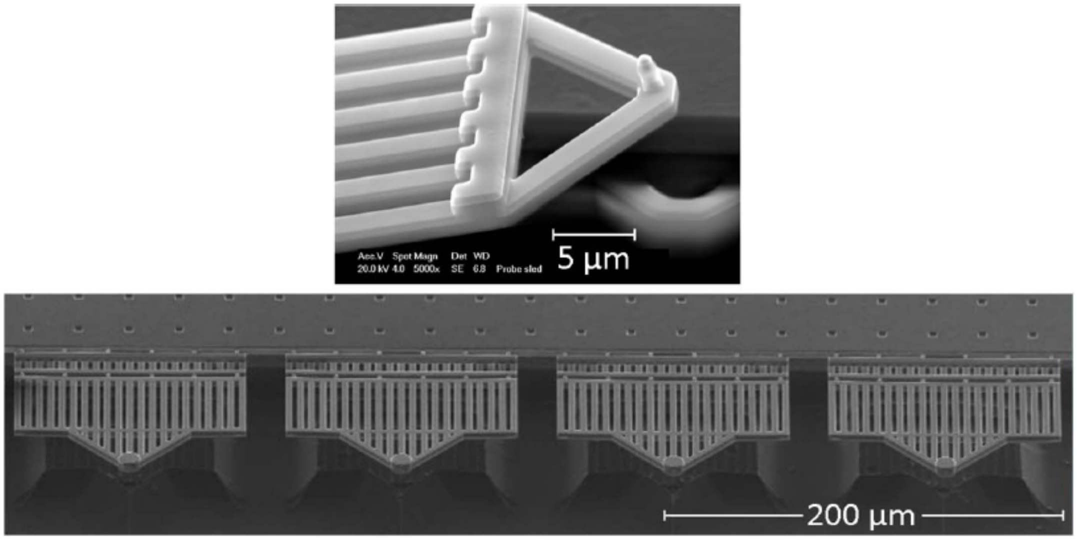

Figure 2. Top: CMOS actuator with integrated STM tip. Bottom: Array for writing parallel features (before addition of tips). Image courtesy of David S. Ricketts, Carnegie Mellon University.

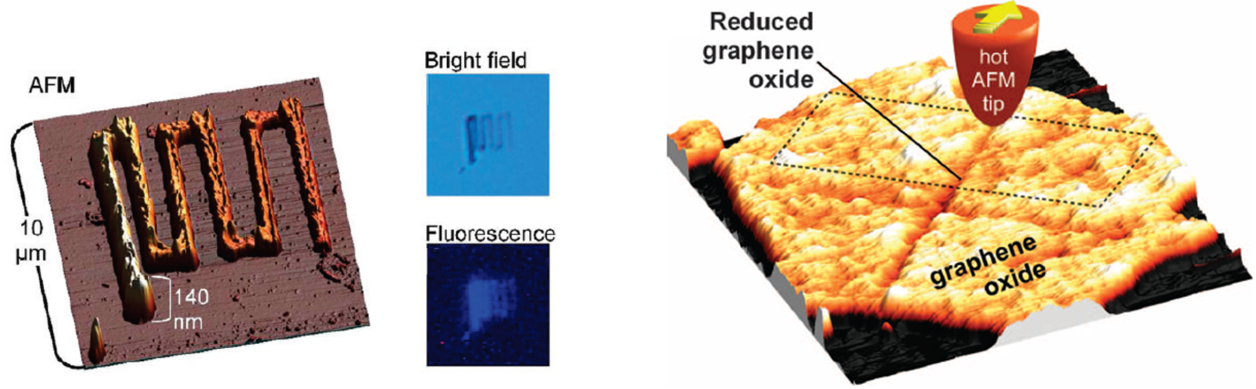

Figure 3. Left: AFM images of a polyethylene-based ink for writing CdSe/ZnS quantum dots. The fluorescence images demonstrate the functionality of the CdSe/ZnS nanoparticles as quantum dots. Right: Local thermal reduction of graphene oxide is accomplished by heating the graphene surface to 330 °C with an AFM tip. The x-shape is formed by bringing the tip across the surface at a rate of 2 µm/s. Image courtesy of William P. King, University of Illinois, Urbana-Champaign.

Northwestern is pursuing a dip-pen based nanolithography approach, with arrays of tips to fabricate nanoscale features with varying dimensions. The team first pursued polymer pen lithography, a scanning probe lithography method that uses soft elastomeric tip arrays with as many as 11 million pyramid-shaped pens. The ink delivery is dependent on time and force, so features can be printed with varying sizes to as small as 100 nm.[27] Recently, the team has moved towards hard-tip, soft-spring lithography, which involves an array of hard silicon tips on a polydimethylsiloxane (PDMS) backing as shown in Figure 4. Using this method, arbitrary patterns with feature sizes less than 50 nm can be formed over a 1 cm^2 area. The team recently demonstrated printing sub-50 nm features of poly(ethylene glycol) (PEG) and 16-

mercaptohexadecanoic acid (MHA) onto a hexamethyldisilazane-coated Si surface and a thermally evaporated polycrystalline Au surface, respectively.[28]

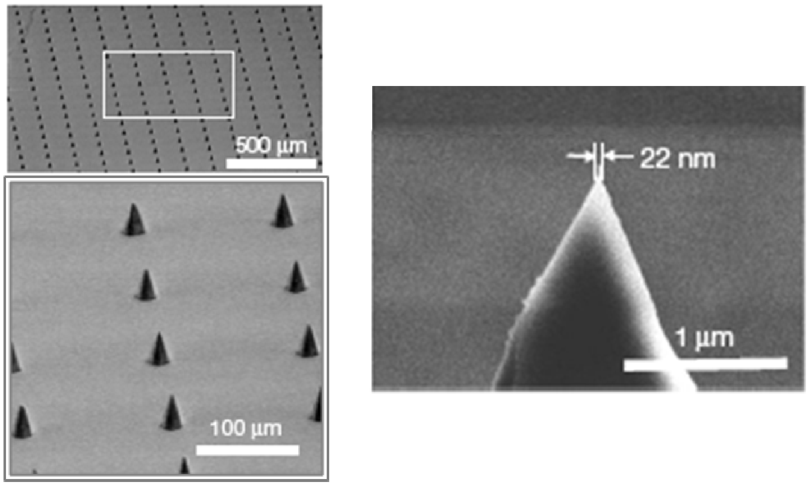

Figure 4. Left: SEM image of the Si tips 150 mm apart on SiO_2/PDMS/glass. Right: Close up of one of the Si tips with a diameter of 22 nm. Image courtesy of Chad A. Mirkin, Northwestern University.

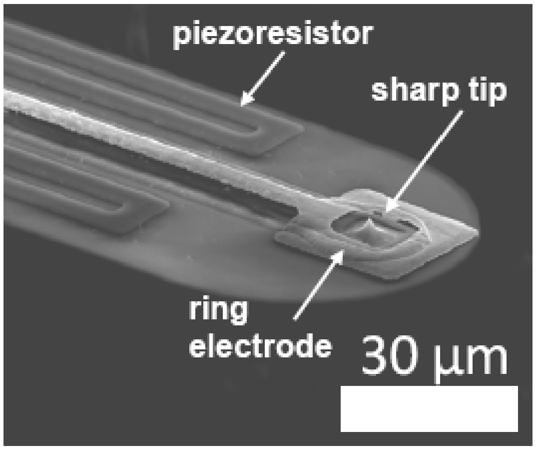

Figure 5. SEM showing the Case Western nanotorch with integrated electrodes and microchannels for local CVD of Si quantum dots. Image courtesy of Massood Tabib-Azar, University of Utah.

The Case Western Reserve University team has created a "nanotorch," shown in Figure 5, in which microchannels and electrodes are integrated into an AFM tip that generates localized microplasma.[29] The plasma is formed by flowing reactive gases through the microchannel and applying a potential difference between the microchannel walls and the tip. The resulting electric field creates a localized plasma region where the active species is deposited. Gases such as Ar, SF_6, CHF_3 may be used for etching, or SiH_4 may be used to deposit Si quantum dots. By utilizing different gases, silicon structures may be deposited, etched, and patterned with features as small as 1 nm.[30]

Optical-based TBN methods incorporate innovative structures within the nanofabrication tool itself such as optical antenna arrays and laser-assisted deposition. The approach explored by the Purdue team uses nanoscale bowtie aperture arrays to focus a laser spot for nanolithography capable of sub-100 nm features.[31] Concentrated radiation results from directing a femtosecond laser beam through the bowtie onto a silicon substrate in a chamber with flowing silane gas.[32] As the laser scans, as many as 100 silicon lines 100 to 250 nm wide are written. Figure 6 shows a schematic and SEM image of the bowtie aperture array. Using focused ion beam (FIB), the bowtie arrays are milled out of a gold film 1.25 microns thick. The dimensions of the bowtie are optimized in order to maximize laser transmission.[33]

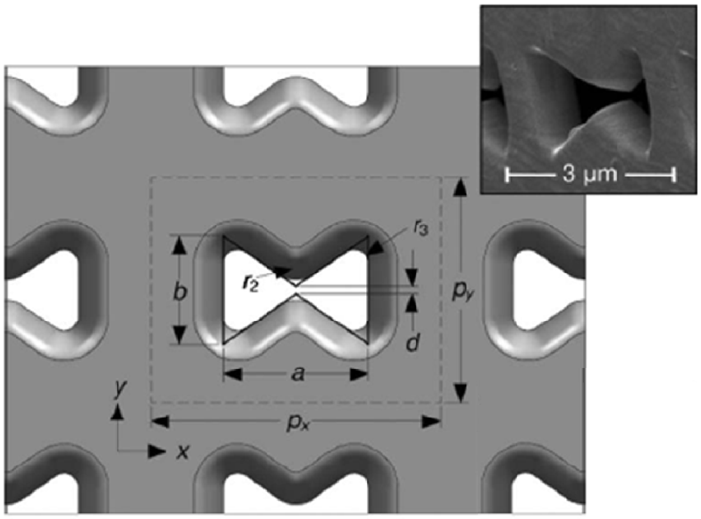

Figure 6. Schematic of Purdue's nanoscale bowtie antenna array. Inset: SEM image of bowtie taken at 52°. Image courtesy of Xianfan Xu, Purdue University.

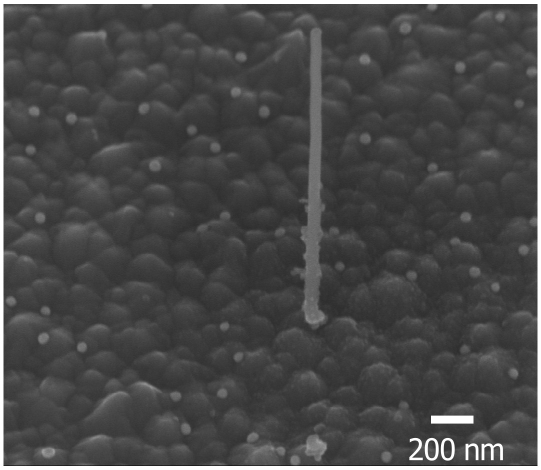

Figure 7. A single Si nanowire among Au nanoparticles on an amorphous Si substrate fabricated by laser-coupled tips. Image courtesy of Costas P. Grigoropoulos, University of California, Berkeley.

The team from the University of California, Berkeley is working on nanofabrication using laser radiation coupled to sharp tips. In this approach, the laser energy is confined in the optical near-field on a catalyst particle. The laser energy is used to controllably grow single semiconductor nanowires, even out of plane as shown in Figure 7.[34,35]

Meeting the TBN program metrics for position and size control requires the nanofabrication system have some metrology methods for determining size and location of the nanostructure with exquisite precision. The Cornell team has built a tool capable of precise wafer-scale metrology called the Nanometrology Optical Ruler Imaging System (NORIS) as shown in Figure 8.[36] A frequency-stabilized, external cavity single mode diode laser creates a beam that is diffracted through a microfabricated metal thin-film quasiperiodic aperture array.[37] The resulting optical diffraction pattern is projected onto the metrology area and is sampled using an CMOS imaging array. The ruler's position is calculated using Fourier transform cross-correlation methods.[36] This high precision ruler has shown positioning within 17 nm over wafer scales.[38] This metrology tool has the potential for wide-spread use among all tip-based nanofabrication approaches as it can accurately measure the size and position of virtually any nanostructure.

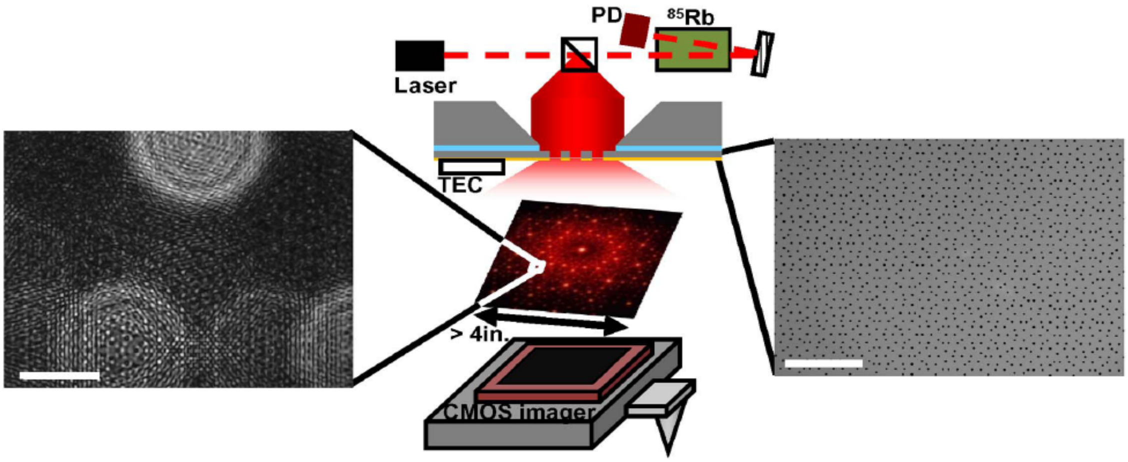

Figure 8. Cornell Team's Nanometrology Optical Ruler Imaging System (NORIS). A thermally-stabilized Ti/Au aperture array is illuminated by a frequency stabilized laser producing a diffraction pattern on a sample. The CMOS imager measures the diffraction pattern to precisely determine nanostructure size and location. Image courtesy of Clifford Pollock, Cornell University.

5. FUTURE DIRECTIONS

Successful completion of the TBN program will require increased precision in size, position, and heterogeneity. Further work to increase the capabilities of TBN as a nanomanufacturing tool will also involve scaling to tip arrays with integrated electronics and sensors for increased automation. Control over the size and position of the nanostructures enables a wide range of applications. Each team's nanomanufacturing technique has yielded the capability to reliably fabricate nanoscale structures with the end goal focused on producing a specific device. Examples include single electron transistors, single molecule chemical sensors, quantum dot IR sensors and emitters, high density interconnects, Kane qubits, and many more. The approaches to nanomanufacturing being explored in the TBN program are unique and there is great potential for the realization of truly revolutionary devices and new capabilities.

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