A Review of Metrology for Nanoelectronics

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***Abstract*—This paper highlights some new and old techniques that will have important metrology inroads for nanoelectronics beyond CMOS. Traditional electron microscopy techniques are envisioned to remain and play a core role at the nanoscale level, and others such as probing techniques and special holographic imaging will further be enhanced and provide more diverse capabilities. The paper presents metrology techniques for beyond CMOS as presented at the First Metrology for Beyond CMOS workshop hosted by the Focus Center Research Program Center of Functional Engineered Nano Architectonics, the National Science Foundation Nanoscale Science and Engineering Center for Nanoprobing, and the California Institute of Technology (CNSI).**

I. Introduction

THE push towards an ideal MOSFET device and architecture with respect to ultimate device density (devices per centimeters squared), power dissipation (in bits/Watt), and performance (in bits/second) continues as driven by the semiconductor industry's inherent need for economic prosperity. The MOSFET itself has been theoretically shown to operate at the fundamental limit of *kTlog2* as derived by the ideal MOSFET model by Swanson and Meindl in 1971 [1], [2]. However, for the industry to have any chance at reaching these ultimate limits with high yields and without economic exorbitance, materials and manufacturing processes must also be ideal, materials need to be pristine, defects completely eliminated, geometries patterned within atomic precision, and dopants positioned with 3-D atomic control. To achieve such excellence, technological development would be required in the following areas: 1) advanced materials; 2) sophisticated fabrication tools and processes; and 3) integrated metrology tools and modeling. This paper will focus on the role metrology technologies and tools will play as CMOS approaches the 32-nm node and beyond. This paper has been inspired by the Workshop on Metrology Beyond CMOS sponsored by Center of Functional Engineered Nano Architectonics Workshop, the Center for the Nanoprobing, and the California Institute of Technology [3].

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II. Metrology Needs

Metrology challenges have been elegantly summarized in the ITRS Metrology Chapter [4]. Some key challenging areas include: 1) general *in situ* metrology integration; 2) managing new material platforms such as silicon-on-insulator (SOI) and associated defects, critical dimension (CD), and film thickness; 3) interfacial properties of stack materials; 4) metrology on high aspect ratio structures; and 5) calibration of tools based on test structures. It seems these general challenges not only apply to the current CMOS platform, but are also highly correlated to most alternative nanoelectronic materials, devices, and architectures currently being assessed by the community. Two examples of these metrology aspects that are independent of technology solutions (materials, devices, architectures) are the following.

1) *Critical Dimension:* CD is of primary importance as the slightest variation can impact device capacitance and carrier density, influence electrostatics, vary threshold voltages, and alter photonic, thermal, and mechanical properties. Likharev [5] has shown that to ensure a 50-mV threshold voltage swing with a 5-nm gate length FET device, critical dimensions should be controlled better than 0.2 nm. Worst still is the sensitivity of channel thickness that requires CD control of 0.1 nm to keep within 50-mV threshold voltage variations. Alternative materials such as carbon nanotubes (CNTs) and carbon nanoribbons (CNRs) has been shown to also be sensitive to dimensional variations [6]. A change from a 2- to 4-nm tube diameter produces a 50% bandgap decrease for CNRs and over 100% bandgap decrease for CNTs. CD is also important for collective behavioral mechanisms such as magnetism. Nawate [7] shows how Pd particles with varying volumes possess nonlinear magnetic behavior. For example, 35 atoms, 55 atoms, and 65 atoms incur magnetic moments of 0.4, 0.3, and 0.4 μ_{B}, respectively.
2) *Quantum Size Effects:* Optical methods used to determine thickness, line edge roughness, and crystallinity are essential. However, as size reduces, quantum confinement effects alter critical optical constants used for calibration. For instance, Zhao [8] shows from first principle calculation that the bandgap and optical properties of Si nanowires are strongly influenced as a function of wire diameter due to quantum confinement and anisotropy in dielectric function being pronounced with wire diameters of less than 2.2 nm. Similar effects are seen with experiments performed on ultra thin SOI where quantum confinement effects alters photon energy critical points below 10-nm thickness [9].

This paper will review some techniques that could further advance metrology capabilities for existing CMOS and enable metrology for new materials, devices, and architectures as enabled by new state variables for information processing.

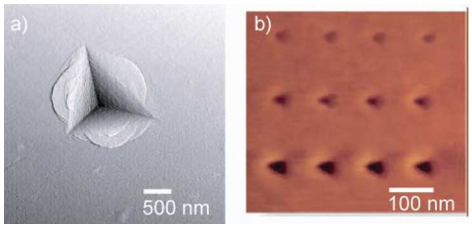


Fig. 1. Elasticity at the nanoscale. In (a), AFM image of a typical indentation in Zr–Cu–Al metallic glass left by a nanoindenter is shown. However, one can extend capabilities of AFM to measure elasticity. In (b), a specially designed cantilever and tip indents a thin carbon film, allowing elasticity to be found on much more local scale than previously possible.

III. Scanning Probe Microscopy

Over a decade ago, the atomic force microscope (AFM) emerged as a leading method to probe topology of structures at nanoscale dimensions [10]. Since then, scanning probe microscopy has become not only a topographic imaging tool with sub-Angstrom resolution, but its abilities have been extended to include measuring intrinsic mechanical and electrical properties of materials. For example, several conducting AFM probes have been integrated into the same platform by many groups (and commercialized by companies like Multiprobe, Inc.), enabling parametric analysis of even the smallest transistors in industry whose contacts are $>$ 50 nm. Also, the special way (near field "tunneling") in which a scanned probe couples to electromagnetic radiation has been used to extract local information about plasmons for emerging plasmonic devices [11]. Similar to those examples, two approaches to scan probe metrology include: 1) extending the capability of current scanned probe techniques to replace current metrology tools and 2) using new features of the scanned probe microscope to characterize material properties such as elasticity (elastic modulus).

Typically, quantitative measure of elasticity is performed by a nano-indenter, a tool which presses into a surface while accurately measuring the depth into the surface and the downward applied force. Although known as a nanoindentor, the tool typically has only micron spatial resolution. Veeco, working in conjunction with Dow Chemical, has now pioneered a true "nano" indenter by using an AFM with a special cantilever and a calibrated tip size and geometry [12]. Using a similar theory of the nanoindentor applied to an AFM, they are able to quantify the elastic modulus of a variety materials including polymers, carbon films, and semiconductor samples at much smaller length scales than previously possible (see Fig. 1).

Instead of indenting a surface, one can also study mechanical properties by driving the AFM tip so that it taps on the surface. Normally in this "tapping" mode, one measures average impulse. However, Solgaard [13] has shown that during the time the tip makes contact with the surface (repulsive part of the impulse), information about the elasticity or "hardness" of the surface may be extracted. Instead of looking directly at the time response of the tip, Solgaard extracts the elasticity information by looking at the frequency response of the tip. Higher harmonics of the drive frequency are generated during the repulsive impulse which can uniquely determine the "hardness" or elasticity of a surface at the nanoscale.

Using a specially fabricated cantilever with two bending modes—a normal flexural mode and an additional (stiffer) torsional mode designed to couple to high frequencies—and a four-quadrant photodetector to decouple to the two modes, elasticity measurements were made on a variety of materials such as a polymer thin films, carbon nanotubes, and silicon oxide. Unlike the nanoindentor method, the tapping technique does not permanently damage the surface. However, quantitative measurements of the elastic modulus are currently not possible.

The AFM is also commonly used to probe electrical characteristics of samples by applying a voltage to a conducting tip and measuring current. However, from the resistance map it is difficult to characterize typical semiconductor parameters of interest such as a doping profile or types of dopants (p or n). For instance, native oxides exist on the samples making contact difficult and unpredictable, significant work function differences between the conductive tip and sample mask the underlying behavior of the sample, and complicated current spreading leads to difficult interpretation resistance map. Many of these issues can be surmounted by pulling the tip away from the surface and applying an ac signal to capacitively couple to the sample beneath, a technique called scanning capacitance microscopy (SCM). Similarly to the nanoindenter, the original SCM was developed and commercialized over 50 years ago. Currently, several companies are integrating high frequency measurement into AFM with the goals of local, quantitative measures of dopant profiles for metrology and qualitative maps of dopant positions useful in failure analysis. In addition, such a capability could also be used to probe and profile quantum confinement effects in nanostructures in particular band-gap variance and leakage currents.

These tools are currently in development specifically for the semiconductor industry and have particular relevance to next generation silicon products. Since CD, electrical, and mechanical characteristics are fundamentally intertwined at the nanoscale, the measure of each is critical and most likely will also be for beyond CMOS. In particular, within the immediate semiconductor industry these techniques could be applied to characterizing a range of materials from low-k dielectrics to complex material stacks and associated interfaces for beyond CMOS electronics.

IV. Alternative Probing Techniques

As research marches forward beyond CMOS, alternative probing techniques are being developed. One example that has driven the innovation of alternative probing technique is research in strongly correlated electron materials (SCE). These materials are a broad array of materials that show interesting behaviors based on their strong electron–electron interactions, material examples are: 1) colossal magnetoresistance maganites; 2) high temperature superconductors; 3) Mott insulators; 4) heavy electron compounds; and 5) organic conductors [14]–[20]. These materials show functionality associated with many phases and most of these phase transitions are present at low temperature $Tc < T_{\mathrm{room}}$. These materials have complex electric and magnetic interactions which manifest in different phases and associated phase transitions (many, in fact, below

room temperature). The challenge for the community is to determine whether complex states can be controlled to enable new device functionality to compete with familiar FET technologies. To do so, new metrology techniques must be developed to characterize device performance parameters such as energy, phase switching speed, and size dependence for SCE materials. As a result, and among other applications, some alternative probing techniques have been realized that include piezoresponse force microscopy (PFM), electric field microscopy (EFM), and variable temperature magnetic field microscopy (VT-MFM).

PFM is based on the detection of local piezoelectric deformation in a ferroelectric sample induced by an external electric field [21]–[24]. Linear coupling between the piezoelectric and ferroelectric parameters infers that the domain polarity can be determined from the sign of the field-induced strain. Application of a uniform electric field along the polar direction results in the elongation of the domain with polarization parallel to the applied field and in the contraction of the domain with opposite polarization. A problem of low sensitivity of a static piezoresponse mode has been circumvented by employing a dynamic piezoresponse imaging method based on the voltage modulation approach, which allows sensitivity to be increased by three orders of magnitude. Scanning force microscopy provides a unique opportunity for controlling the ferroelectric properties at the nanoscale and direct studies of the domain structure evolution under an external electric field, which cannot be matched by previously available techniques. A conductive probing tip can be used not only for domain visualization but also for *in situ* modification of the initial domain structure. Application of a small dc voltage between the tip and bottom electrode generates an electric field of several hundred kilovolts per centimeter, which is higher than the coercive voltage of most of ferroelectrics, thus inducing local polarization reversal. One of the most important applications of PFM is local piezoelectric spectroscopy, i.e., measurements of local hysteresis loops at the 10-nm level. The vector PFM approach in conjunction with the local switching experiments can be used to analyze the effect of grain crystallographic orientation on the local hysteresis loop parameters. Application of piezoresponse force microscopy to ferroelectrics has opened new possibilities not only for high-resolution imaging of domain structures, but also for quantitative characterization and control of ferroelectric properties at the nanoscale.

Another approach in better understanding strongly correlated electron systems is based not only on magnetic field imaging, but on temperature *dependent* magnetic field imaging. This is possible by developing variable temperature, otherwise known as low temperature magnetic field microscopy (developed by de Lozanne [25] among others). While the heart of such an instrument is still a microfabricated piezoresistive cantilever sensor, there are four substantial improvements: *in situ* lateral positioning of the cantilever tip over the sample, external optical access to aid in positioning, a longer probe in order to reach a superconducting magnet inside a standard Dewar, and a better heat sink to allow the MFM to reach lower temperatures. Such an instrument is ideally suited for exploring microscopic phase evolution in materials with low-temperature magnetic transitions such as the manganites and for examining micromagnetics in patterned magnetic devices at low temperatures and especially systems aiming for ultimate device density beyond CMOS that operate at or near the superparamagnetic limit [26], [27].

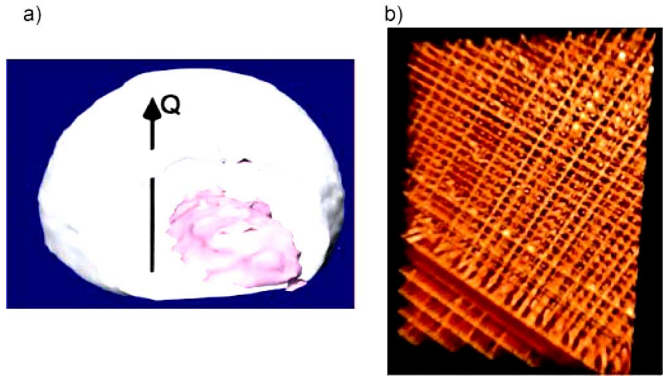


Fig. 2. X-ray tomography. (a) When coherent X-rays (produced from a synchrotron) are incident on a crystalline particle (lead on a silicon substrate), diffraction pattern contains series of Bragg points corresponding to lattice. Each Bragg point also has structure which contains detailed information about shape of particle (inset). In addition, asymmetry between Bragg points contains information about distortions or strain fields in lattice. Algorithms can then be used to reconstruct a real space image of both shape and distortions in lattice to nanometer accuracy, where purple region demonstrates strain induced in lead particle by substrate. (b) More typical tomographic image of metal interconnects of a microprocessor is demonstrated from a laboratory-sized tool. Here, tomography image is reconstructed from differences in X-ray scattering from metal and dielectric, nondestructively producing an intricate and useful image with sub-50 nm resolution.

V. X-Ray and Electron Microscopy

Instead of relying solely on new technologies such as scanning probe microscopy, mature technologies such as X-ray, neutron, and electron scattering have made consistent advances to keep pace with metrology challenges brought to the community by new research problems and existing challenges facing the semiconductor industry.

When one shines a beam of particles, say photons, the coherence of the beam determines the type of information one can extract. Traditionally, incoherent X-ray scattering (needed for nanoscale metrology) has provided valuable statistical information such as surface or interface roughness. However, with the evolution of more coherent X-ray sources local information about structures can also be gathered. Unlike visible light, X-rays have the disadvantage of very small elastic scattering cross section in materials (index of refraction nearly 1 for most materials), thus making refractive optics difficult. Diffractive optics are becoming a popular alternative, with spatial resolution now reaching below 30 nm [28].

However, a limited scattering of X-rays allows the study of both buried interfaces and intrinsic material composition. Measuring the diffraction off the lattice or other periodic structures (such as lithographic features) gives details of shape and even distortions in the lattice (strain fields) can be observed. Although X-rays only weakly scatter, heavier materials scatter more photons than lighter materials so image contrast can be achieved. In fact, one can imitate 3-D images of a medical CT scan by rotating the sample in the beam, producing a tomographic image. In Fig. 2, 3-D images are shown of IC copper interconnects and

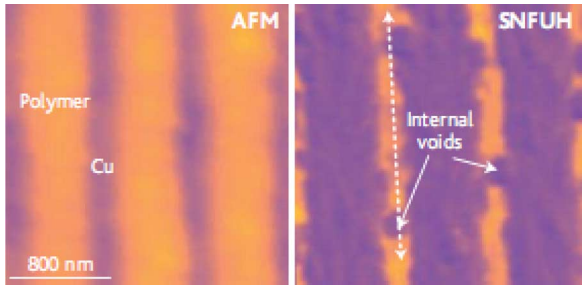
strain fields [29] in metal particles reconstructed from various algorithms. Additionally, material composition can also be determined through inelastic scattering of the X-rays on the inner shell electrons (X-ray fluorescence provides the fingerprint for atoms and molecules). Reflected X-rays at glancing incidence can provide a measure of critical dimensions and roughness that make up the building blocks of nanoelectronics. This provides an encouraging route forward to beyond CMOS where more complex 3-D structures are envisioned and where tolerances would be a greater concern.

X-ray scattering also provides a means to study dynamics at this length scale. Using image contrast, electromigration of a buried copper interconnect can be monitored and recorded as a function of time, with limited damage as compared with electron optics (given the time scale needed to observe the electromigration event) [30]. One can also measure how the diffraction pattern of the X-rays evolve in time. In the future, free electron lasers should be capable of producing coherent X-ray pulses at nanosecond timescales, allowing foray into both fast and high resolution imaging with dynamic X-rays [29].

Beams of more strongly interacting particles such as electrons can also provide valuable local information of material composition, shape, and structure. Compared to X-rays or scanning probes, the probe size (beam) of the scanning transmission electron microscope (STEM) can be made much smaller, on the order of 0.2 nm. (Although the electron wavelength is on the order of 0.002 nm, the beam size is limited by aberration in the electron optics.) Similar to X-ray imaging, the narrow beam (from a nearby field emission tip) can be rastered across the sample to gain a 2-D elemental map of the sample with single atom sensitivity. Mass contrast is provided by elastic scattering proportional to atomic number (Z); however, current practice is limited to elements heavier than arsenic $(Z = 33)$ for single atom detection (lighter elements can still be imaged by averaging over a few dopants). Additionally, inelastic scattering (spectroscopy) can provide elemental resolution with a spatial resolution of 0.3 nm.

Although electrons and X-rays employ the same elastic and inelastic scattering principles to measure material properties, they have very different interaction cross sections which provides advantages for each. Due to the ease of focusing electrons compared with X-rays, the spatial resolution of a STEM is currently unrivaled by X-rays. However, for larger sample volumes (micron or larger samples) which require less resolution ($\sim$ 30 nm), X-rays may provide significant advantages in terms of speed and volume of data collection. Similarly, because of the lack of interaction of the X-ray, they are commonly thought as less damaging than electron beams. However, if one normalizes the damage events by the number of useful (elastic) imaging events, electron beams actually tend to damage materials less [34].

As expected for two rival techniques, both are being investigated. With X-rays, the key technical challenges include focusing the X-rays to a smaller beam for finer resolution. Also, there is significant work to be done to understand coherent scattering from X-rays and dynamic evolution for time-dependent studies of nanostructures. For electron beams, pushing the forefront in atomic scale tomography and angstrom level resolution are some of the goals for the next generation STEM. Amazingly, to get to atomic scale tomography one must be able to tilt the sample with angstrom level precision, posing a significant challenge. Also, further correcting the lens aberration of the electron beam should allow more resolution and contrast to the images.



(a) (b)

Fig. 3. SNFUH imaging of copper low-dielectric interconnect system. (a) Typical AFM (topography) image shows periodic polymer and copper features. Copper lines are about 60 nm wide and polymer one is around 200 nm. (b) Phase image of SNFUH that clearly reveals surface elastic contrast and subsurface voiding in copper lines.

VI. Alternative Metrology Methods on Horizon

Alternative metrology methods on the horizon are numerous. Such examples include scanning near-field ultrasound holography (SNFUH), employing selective electrochemical deposition (SED) for detection of point defects, and exotic bio-based metrology using biomimetics.

Employing ultrasounds for metrology possesses desirable functionality such as nondestructive, noncontact, subsurface imaging and potential high volume throughput. Shekhawat and Dravid have pioneered scanning near-field ultrasound holography (SNFUH) with improved spatial resolution (10–100 nm) and depth information [35]. In SNFUH, acoustic waves are launched on both the probe tip and the sample at slightly different megahertz frequencies. The interference of these two waves forms a surface acoustic standing wave. This wave is altered by subsurface features such as voids, and the change in its frequency is monitored by the AFM cantilever. Shekhawat and Dravid show that this technique can be used for hard and biological materials (contact mode for hard materials and the noncontact mode for biological materials) which is desirable for various hybrid structures potentially used for beyond CMOS solutions. An example applicable to semiconductor industry is the detection of voids in copper lines as shown in Fig. 3. The direct, nondestructive observation of a void in an opaque material had previously seemed to be a nearly impossible task.

CNTs are represented as a candidate material for emerging beyond CMOS devices in the ITRS roadmap. Since single-walled CNTs operate in the ballistic regime, a single point defect can considerably vary the electrical characteristics of CNT-based devices. New metrology tools that are able to detect CNT defects have been proposed. Selective electrochemical deposition (SED) is one potential solution to efficiently and

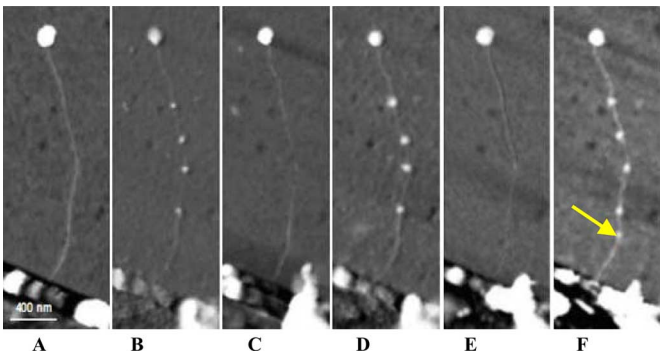


Fig. 4. SED results on CNT that is 1.8 μm long. (A) Pristine CNT. (B) Pristine CNT and nucleated nickel dots by SED methods operating for 10 s. (C) Reverse SED showing removal of metal dots. (D) Regrowth of nickel dots on defect sites. (E) Cleaning of SED grown dots using an acid etch which results in added defect as shown in (F) with yellow arrow.

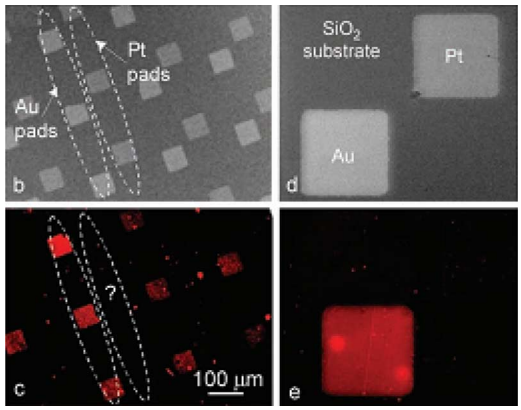


Fig. 5. Organic/Inorganic metrology of specific interactions between engineered peptides and noble metals. Top images [(b) and (d)] show optical images of microfabricated metal pads. (c) and (e) show fluorescent images (as a result of attaching steptavidin-conjugated quantum dots to the peptides) when peptide immobilization takes place which verifies self-assembly nature on only Au pads.

quantitatively measure defect densities in individual SWNTs and SWNT circuits [36], [37]. A sequence of electrochemical potentials applied to a SWNT can selectively nucleate metal deposition at the sites of highest chemical reactivity. As SWNT defect sites are more sp^2 bonded lattice, appropriate deposition reactive rather than the pristine potentials can decorate these sites with high selectivity. Fig. 4 shows SED results on a CNT that is 1.8 μm long. The figure shows the pristine CNT and nucleated nickel dots by the SED methods operating for 10 s. These depositions can be easily removed by a reverse SED process. Repeatability is shown in Fig. 4(d) and cleaning is performed using an acid etch which results in an added defect as shown in Fig. 4(f). Such a technique is far superior to CNT point defect detection and quantification using Raman spectroscopy and scanning tunneling microscopy. Using this SED method, Collins has quantified CNT defect density to be one per 4 μm [36]. To put these results into context, this equates to about one site per 10^6 carbon atoms or, for a dilute network, 10^{12} surface atoms. This number can be compared to Si crystals that typically have oxygen impurities of one per 10^{13} and silicon vacancies or interstitials of one per 10^{12} atoms.

A growing area of interest and rapid progress revolves around bio-specific self-assembly interactions for beyond CMOS processing and metrology applications. Such techniques usually target specific binding of various elements to target sites (such as a defect or certain element/material) so that quantitive information can be extracted for analysis and feedback (metrology). Impressive results have come from peptide engineering, specifically using gold-binding peptides. Sarikaya was one of the first to show quantitatively that a genetically engineered peptide has metal-specific binding and recognition sites [38]. By using steptavidin-conjugated quantum dots (QD) attached to the peptides, a metrology capability is possible to identify at which material sites the peptides preferentially assemble. This is a simple example of using a bio-based element to specifically attach on a metal. Such a capability would enable a metrology capability to possibly detect (and quantify) contaminants or defects within a crystal lattice. Fig. 5 shows an example of the direct self-assembly of the peptides attached with QDs where they selectively attach onto gold preferentially. Such a result is significant in terms of biding the biological and inorganic domains in terms of specificity (Au versus Pt) and quantitative path towards measurement (QD fluorescence). The mechanism by which a peptide binds selectively to a metal surface rather than an oxide and specifically to Au rather than Pt is not well understood; however, polar moieties on the gold-binding peptide and the physical conformation of the peptide may contribute to the specific absorption. Similar research by Belcher and coworkers [30] has also generated great interest where the realization of highly specific bio-self assembly methods via bio-engineering could one day be used for practical metrology applications.

VII. Conclusion

In our quest to continue scaling and progress by keeping with Moore's law, technology development and processing greatly depends on manufacturing process perfection, material predictability, defects elimination, and spatial feature control with atomic precision. In this spirit, we have provided a snapshot of unique metrology technologies for emerging nanoelectronics ranging from probing techniques, electron microscopy, ultrasound holography, and selective electrochemical deposition, among others. Multiple tools using different physical phenomena will be required for process control of future nanoelectronics, as done similarly with today's CMOS processing. Nevertheless, new techniques will require us to take into account nanostructures such as CNTs, QDs, single monolayers and be compatible to both organic and inorganic components in a nondestructive manner.

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