

Report on Japan-US Symposium on Tools and Metrology for NanoTechnology

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Submitted by

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Executive Summary

The first Japan-US Symposium on Tools and Metrology for NanoTechnology was held on Cornell University campus in late January (Jan. 21-24) of 2003. The symposium was sponsored by Ministry of Education, Culture, Sports, Science and Technology (MEXT) of Japan and the National Science Foundation (NSF) of United States. The objective was to take a critical look at the innovations needed in tools for creation, analysis, measurement, and repeatability-verification in the wide array of areas where nanotechnology has a strong role. The symposium employed discussion and debate, guided by advanced research work at the nanoscale conducted in a variety of disciplines: biology, chemistry, electronics, mechanics, materials, optics, etc. and in their cross- and inter-disciplinary offshoots, as the mechanism for arriving at the conclusions reported here. All these disciplines employ a number of approaches, besides the top-down and bottom-up approaches, that are now recognized as a very essential ingredient in success of nanotechnology. By bringing together the leading practitioners of the field in a lively debate, the symposium focused on identifying the necessary tools and techniques that need to be developed in order to take the research ideas to manufacturing. The symposium identified some of the critical directions, whose smooth passage is essential to the success of nanotechnology, as:

- 1) Fabrication and synthesis techniques for ordered as well as custom patterns of increasingly small and increasingly complex structures in a variety of materials
- 2) Measurement and visualization of the key characteristics of the experimental structures
- 3) Verification of reproducibility of the experiments
- 4) Should the experimental direction have a larger-scale potential, development of tools and methods for scaling up production to economically feasible levels

The *abstract digest and the talks on the enclosed CD* give a broad view of the lively presentation, discussion, and debate of the symposium. This information is also available at the NNUN web-site (<http://www.nnun.org>) and it provides the underlying rationale for the directions identified and the recommendations made by the symposium.

The symposium makes the following recommendations:

1. *The symposium recommends that a strong effort be placed in development of tools, methodologies, underlying science and engineering, and practical techniques to solve the process-related problems of reproducibility, size and three-dimensional metrology, and of stability of processes and structures at the smallest dimensions because these are common foundation issues for most practical applications of nanotechnology that are likely to reach manufacturing.*
2. *The symposium recommends that a strong effort be placed in development of standards and references with atomic-scale multi-dimensional precision and in development of technology that allows reproducible and large-scale positioning and control of properties of active and passive uses of nano-scale elements (nanotubes, ...) should their applicability is clearly demonstrated.*
3. *The symposium recommends that an attempt be made to evaluate and find mechanisms that will lead to development and availability of tools that are affordable to university researchers. An effort in this direction by National Laboratories will have a widespread impact.*
4. *In order to facilitate the success of applications of nanotechnology that are critically dimension dependent, the symposium recommends that effort also be placed in developing the advanced tools needed for definition, measurement and reproducibility testing of small dimensions in both planar and vertical dimensions.*
5. *The symposium suggests that high-speed ab initio simulation capability and the experimental data visualization capability be developed across the wide variety of disciplines in order to fundamentally understand, and to provide guidance of experimental optimization and materials and process developments.*
6. *To better facilitate graduate training as well as pursuit of research objectives, the symposium suggests that funding agencies consider a larger time overlap in research funding and graduate student lifetime (~5 years).*
7. *The symposium suggests that the evolution of an interdisciplinary field is in itself an interesting history in making, and that it be studied for ethical and sociological content and for deriving lessons for the future.*
8. *The symposium suggests that we, as a society, continue to find ways to build on the momentum of children's inherent interest in sciences. This needs fostering and creation of incentives for education and learning and of the excitement of sciences for the students and the teachers. The symposium recommends that this requires a continuous hands-on and laboratory-based K-12 and undergraduate teaching.*

Report

The challenges for scientists and engineers working in the area of nanotechnology are extraordinarily complex. The subject area is broad and the challenges are quite often interdisciplinary. The scientific work is at a dimensional scale where tools for creation, characterization, and visualization are limited. New techniques for fabrication, synthesis and characterization must be developed to effectively explore the realm of nanotechnology. Furthermore, for attractive applications, scaling up of these techniques for large-scale production use requires significant improvements in speed, reproducibility, and control. Development of tools and metrology for fabrication, characterization, and production are therefore an important barrier to progress of nanotechnology.

Tools enable nanotechnology development and exploration. These tools, which have largely had a research focus, have limited manufacturing use. The techniques made possible by these tools are limited to a relatively small set of materials, structures, and devices. This in turn limits the materials, structures and devices that can be produced with favorable cost and in adequate quantities. Advances in nanotechnology however, continue to enable the development of new tools for the study and fabrication of other nanostructures. An example of this is scanned probe instrumentation that have been applied extensively to probe-based writing and characterization. These tools employ a variety of nanostructures as tips or other sensors and actuators for highly sensitive measurements. Lastly, the advances of nanoscience and nanotechnology provide the economic and technical driving force to enable the development of production scale tools necessary to support migration of this technology into the commercial sector. This is obviously a complex set of interactions involving a very broad section of science and engineering, and by bringing together key scientists and engineers from these areas to explore the role of tools and metrology for nanotechnology, the symposium was successful in crystallizing the needs in tools and metrology for nanotechnology.

There were a total of 38 presentations (15 min. for talk + 5 min. for questions and answers), and each session had an extra half an hour or more for discussion following a number of presentations in a given subject area. Dr. E. Gulari (Acting Assistant Director of Engineering at NSF), Mr. T. Maruyama (Deputy General Manager of MEXT), and Dr. T. Noda (Deputy Director General of NRNCJ) provided the United States and Japan background of national interest and effort in nanotechnology. The symposium also provided time for the participants to hear about Cornell centers that have significant effort in nanotechnology and for visits and discussion of research being conducted in the centers.

The broad range of areas in which nanotechnology plays a critical role was divided into four major themes with a distribution over 35 presentations. These themes were:

- Polymers, Organics, Particles and Synthetic Chemistry Research and Techniques
- Materials Research and Techniques
- Chemical and Physical Tools and Metrology
- Mathematical and Other Tools, Manufacturing Needs, and Other Perspectives

Among the characterization, fabrication and synthesis tools discussed were the scanning probe based atomic force microprobes, scanning tunneling microprobe, and other near-field scanning probe instrumentation such as those for Raman measurements. The wide array of charged particle based tools – transmission electron microscope, the STEM, focused-ion beam tools, and electron beam tools such as those for lithography were discussed at length. Among the neutral beam tools discussed were those employing neutrons (as used in neutron spectroscopy), near-field tools, imprinting and optical stepping tools (including those employing ultra-violet radiation), and methods for self-assembly. The limitations of these, possible mechanisms for improving their performance were discussed and are summarized later in this document.

Among the issues discussed in the context of calculation and visualization needs were, the importance of obtaining structural information at the atomistic scale and the atomistic information for reaction paths. This approach is critical to answering questions of what the experimental techniques are actually observing, and for correlation of properties and structures. The issues with such approaches discussed were those related to finding efficient methods for treating nanoscale objects containing $>10^4$ atoms and efficient methods for sampling the vast phase space for global total energy minimum and for finding the reaction path. The possible solutions for these is an order n calculation, efficient parallel computation and development of algorithms through intuition and trial and error. Strong computing and intelligent minds are indispensable for the computation and visualization.

In the specific issues related to high resolution chemical and electrical research down to atomic scales, a number of issues and possible solutions were identified. A significant range of the living and organic high-resolution characterization has to be accomplished in a controlled environment. Environmental chambers and stages are needed with new correction techniques for achieving high resolution. The use of low voltages also requires additional aberration correction. In-situ imaging of soft materials requires uncommon techniques such as cryo-TEM, cryo-SEM and freeze fracturing. There is no universal solution to the issues, but at the least, the research community needs access to a few TEMs and SEMs with such capabilities. Such tools also require significant technical expertise, and there are less of such technically qualified individuals today than in the past because of limited career opportunities. Development of qualified individuals is a very important requirement for this field to advance since these technical approaches do not have other alternatives.

The most significant tool for definition for of small dimensions currently is electron-beam lithography. Speed and cost are important factors in electron beam lithography usage, and while the tool is reasonable for research purposes, it is inadequate in its present form for manufacturing. Manufacturing will require both speed and control improvements. Resolution and registration can likely be improved by closed-loop control of the systems.

Focused-ion beam and small angle neutron scattering are two techniques that were discussed in the symposium. Focused-ion beam, like electron-beam lithography, is a suitable tool for university environment because it provides rapid fabrication and characterization capability. But, its issues are similar to those of electron-beam lithography and, in addition, those due to ion-based contamination. Modification of column for a larger window in acceleration voltage and

beam intensity will require further work. The small angle neutron scattering has been applied for reciprocal space imaging of soft colloids and complex fluids in multiple modes. The facilities for users are somewhat limited and all these tools are quite expensive.

Scanning probe tools have found extensive usage because of the cost and the atomic scale manipulation capabilities that they have provided. However there remain specific issues with these tools also. Tip stability, preparation, cost, and quality, need to improve for reproducible fool-proof routine usage. The low scanning speed leads to low throughput. The tools are still inadequate for chemical analysis and need to be merged with other methods. When the sensing probes are brought near the samples, properties can change, and therefore adequate work needs to be done to match characterization with measurements and what they mean.

Tip characteristics are vital in all scanning probed tip approaches. Simple measurements such as dimensions or surface characteristics (profiles, e.g.) are strongly dependent on tip morphology and characteristics. Reliability and trust in measurements depends on the use of right tips and proper de-convolution of data. Thus, the use of tips requires a significant knowledge and sophisticated techniques in measurement and analysis. Hence, protocols and standards for x, y, and z scales measurement and analysis are indispensable. Multi-probe tools clearly would be useful in a number of cases, such as in four-point measurement of nanostructures as well as parallel operation for speed.

Nanotubes have found much discussion in electronics and sensors and as standards. Size control, chirality control, placement control, contact technology, achieving p-type and n-type tubes, drift and hysteresis, integration of such tubes as transistor elements and integration of such tubes as wiring elements are all research and manufacturing related problems. Finally, whether nanotubes are capable of overcoming CMOS limits in the way electronic functions are achieved is a large area of debate.

Micro-and Nano-machining is an important discipline in nanotechnology. Many scanned probe instruments are examples of nano-machines. Nanomachining, e.g., is part of sophisticated microsystems in which there are MEMS and NEMS elements in a wide variety of applications. These require two- and three-dimensional fabrication techniques, electronic circuit technology that is compatible with MEMS processes, controllable deposition and growth of new materials (nanotubes, e.g.), high performance actuators and energy sources, integration of functional materials, and development of complex integrated systems. In fabricating such structures, stiction of moving parts during the process of creation and use, lithography in three-dimensions, packaging, deep and high aspect ratio etching and other patterning, deposition of thick stress-free films, filling of trenches and large aspect ratio regions without voids, low temperature bonding, as well vapor-phase based deposition processes and low cost prototyping are issues in achieving success in such nano-machining based applications.

Nanotechnology employs a variety of techniques. All such techniques require standards and protocols for the processes and for characterization of processes and of results achieved. To make broad advances possible, new techniques are needed. In particular, low temperature dry bonding is a critical one in three-dimensional integration and in systems employing multi-functional integration. Bonding does not have a stable low temperature or room temperature process, commercial tooling, and requires materials science-based research as well as

development of industrial tools that could be affordable to academics. Lithography also requires development of reproducible techniques capable of achieving the dimensions at high speed and low cost. Imprinting is one such approach and will require clean environment for flattening of organic surfaces, high aspect ratio etching of bilayers, and development of nano-scale alignment techniques. Self-assembly, as a technique, also has interesting usage in manufacturing. It has the desirable attribute of low cost, but in practice, while it exhibits good short-range order, it has poor long-range order. So, defect density of self-assembly is high. Size distribution and registration effects on patterned surfaces are both a feature and an issue with general purpose applications. E-Beam lithography has been the mainstay for creation of nanoscale dimensions. Speed, cost, and closed-loop control for dimension and registration are issues that need addressing in e-beam techniques. So, for nearly all the techniques of nanotechnology, there are considerable developments needed in order to bring the promise to fruition.

Below is a short list, by categories, of the rationale and recommendations of the symposium.

Process and Technology Related Issues

The symposium observed that a number of important problems remain in the variety of currently researched techniques for nanotechnology.

- Bottom-up technology, self-assembly, e.g., has issues with defects when long-range order (>10 nearest neighbors) is considered
- Control of size and size variations, and in the case of three-dimensional objects, variations in the third dimension, are a central issue in reproducibility and manufacturing
- Measurement of three-dimensional small objects: size and their variation, and measurements along the third dimension remains an issue
- Many long-employed techniques have difficulties due to energies as well as transport limits. As an example, electroplating at 20 nm gaps is very difficult
- Small dimension patternin encounters difficulties due to pattern collapse because of the softness of materials employed
- High aspect ratio pattern transfer in bilayers (such as those employed in imprinting) has difficulties at small dimensions because of similar transport and stability issues as those in the problems described above
- Custom nano-scale patterns, and transfer of these patterns, will be a critical element in most general nanotechnology applications. This is a very slow process, has problems with reproducibility, control within a range, and verification of dimensions. While electron beam technology has been a mainstay for custom applications, it remains slow for manufacturing.

The symposium recommends that a strong effort be placed in development of tools, methodologies, underlying science and engineering, and practical techniques to solve the process-related problems of reproducibility, size and three-dimensional metrology, and of stability of processes and structures at the smallest dimensions because these are common

foundation issues for most practical applications of nanotechnology that are likely to reach manufacturing.

Standards and References: Critical Dimensions and Nanotubes

The need of critical dimension is a very broad issue for nanotechnology. Electronics requires a control that is factors of 10 smaller than the smallest dimension. This means that atomic scale control is required and references are required with atomic precision in x, y, and z direction. Nanotubes have been posited as possible option for a future generation of electronics. Such use of nanotubes requires exact positioning for active devices and wires for millions to billions in order to replace current approaches.

The symposium recommends that a strong effort be placed in development of standards and references with atomic-scale multi-dimensional precision and in development of technology that allows reproducible and large-scale positioning and control of properties of active and passive uses of nanotubes, if their applicability is clearly demonstrated.

Research Tools

The symposium discussion often focused on the cost of research – in tools and in longer-term support needed in graduate training. Tools are quite often derived from semiconductor industry. Such tools usually have a high cost, and the need exists for finding mechanisms that will lead to low-cost tools. AFM and STM were often cited as tools that have had a large impact in nanotechnology research.

A very large and practical application of nanotechnology has been in electronics. However, electronics faces a number of serious problems at nanoscale. Non-destructive characterization and testing in the midst of the nearly 450 steps process (~1,500 hours of machine and human effort) requires a variety of essential measurements, e.g., (a) sub-1 nm resolution of 5 nm dimensions; measurement of thin-film properties (thickness, real and imaginary index, etc.) in structures consisting of multiple stacks of thin films; measurement of stress at nm-scale resolution, etc. A very significant direction of research is three-dimensional integration. This approach will benefit immensely from a wafer-scale room temperature covalent bonding technique.

The symposium recommends that an attempt be made to evaluate and find mechanisms that will lead to development and availability of tools that are affordable to university researchers. Making this one of the goals of National Laboratories was one suggestion.

In order to facilitate the success of applications of nanotechnology that are critically dimension dependent, the symposium recommends that effort also be placed in developing the advanced tools needed for definition, measurement and reproducibility testing of small dimensions in both planar and vertical dimensions, and in particular, with an emphasis on non-destructive measurement tools.

Theory, Modeling, Simulation and Visualization of Theoretical and Experimental Information

The experimental nanotechnology implementations are quite often time and resource intensive. The theoretical underpinnings of molecular (large and small), inorganic and organic condensed matter interactions, and those of living objects, are strongly desirable for us to develop a strong intuitive and comprehensive understanding. Visualization of the necessary data, theoretical or experimental, is essential to development of this understanding.

The symposium suggests that high-speed ab-initio simulation capability and the experimental data visualization capability be developed across the wide variety of disciplines in order to fundamentally understand, and to provide guidance of experimental optimization and materials and process developments.

Time Period of Academic Research Grants

Graduate research time-scale and funding time-scale was recognized by discussion as being at odds with each other. Research funding is usually at a much smaller time scale than the lifetime of a graduate student.

To better facilitate graduate training as well as pursuit of research objectives, the symposium suggests that MEXT and NSF consider a larger time overlap in research funding and graduate lifetime (~5 years).

Culture

Nanotechnology is unique among the scientific frontiers in its breadth and interdisciplinary content. In addition to the complexity of advanced technology at the limits of dimensional scales of atoms that we can visualize, there are complexities that arise from the language and approaches that are the culture of different fields – from within sciences such as biology, chemistry, physics, or zoology, to within engineering of chemical to electrical to mechanical. This difference, and the approaches that the different disciplines value in their publication and information dissemination in for-profit and professional publications, leads to interesting conflicts of culture, speculations of uses of nanotechnology, and at times ethical issues.

The symposium suggests that this evolution of an interdisciplinary field is in itself an interesting history in making, and that it be studied for ethical and sociological content and for deriving lessons for the future.

Educational and Societal

Although not a central theme of the symposium, training and developing methods that encourage graduate students to stay in the field appeared as an important subject of discussion in the symposium. Participants from Japan also voiced increasing concern in encouraging students to pursue careers in engineering and science.

The symposium suggests that we continue to find ways to harness the inherent interest of children in sciences by building on its momentum through hands-on laboratory-based learning, and encourage and develop the momentum through 12 and undergraduate education through continued emphasis on science as a practical endeavor.

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