# AN ASSESSMENT OF THE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY MATERIALS SCIENCE AND ENGINEERING LABORATORY

## FISCAL YEAR 2008

NATIONAL RESEARCH COUNCIL
OF THE NATIONAL ACADEMIES

# AN ASSESSMENT OF THE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY MATERIALS SCIENCE AND ENGINEERING LABORATORY

## FISCAL YEAR 2008

Panel on Materials Science and Engineering

Laboratory Assessments Board

Division on Engineering and Physical Sciences

NATIONAL RESEARCH COUNCIL
*OF THE NATIONAL ACADEMIES*

THE NATIONAL ACADEMIES PRESS
Washington, D.C.
**www.nap.edu**

**THE NATIONAL ACADEMIES PRESS 500 Fifth Street, N.W. Washington, DC 20001**

NOTICE: The project that is the subject of this report was approved by the Governing Board of the National Research Council, whose members are drawn from the councils of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine. The members of the panel responsible for the report were chosen for their special competences and with regard for appropriate balance.

This study was supported by Contract No. SB134106Z0011 between the National Academy of Sciences and the National Institute of Standards and Technology, an agency of the U.S. Department of Commerce. Any opinions, findings, conclusions, or recommendations expressed in this publication are those of the authors and do not necessarily reflect the views of the agency that provided support for the project.

International Standard Book Number-13: 978-0-309-12728-8
International Standard Book Number-10: 0-309-12728-9

Copies of this report are available from

Laboratory Assessments Board
Division on Engineering and Physical Sciences
National Research Council
500 Fifth Street, N.W.
Washington, DC 20001

Additional copies of this report are available from the National Academies Press, 500 Fifth Street, N.W., Lockbox 285, Washington, DC 20055; (800) 624-6242 or (202) 334-3313 (in the Washington metropolitan area); Internet, http://www.nap.edu.

Printed in the United States of America

THE NATIONAL ACADEMIES

*Advisers to the Nation on Science, Engineering, and Medicine*

The **National Academy of Sciences** is a private, nonprofit, self-perpetuating society of distinguished scholars engaged in scientific and engineering research, dedicated to the furtherance of science and technology and to their use for the general welfare. Upon the authority of the charter granted to it by the Congress in 1863, the Academy has a mandate that requires it to advise the federal government on scientific and technical matters. Dr. Ralph J. Cicerone is president of the National Academy of Sciences.

The **National Academy of Engineering** was established in 1964, under the charter of the National Academy of Sciences, as a parallel organization of outstanding engineers. It is autonomous in its administration and in the selection of its members, sharing with the National Academy of Sciences the responsibility for advising the federal government. The National Academy of Engineering also sponsors engineering programs aimed at meeting national needs, encourages education and research, and recognizes the superior achievements of engineers. Dr. Charles M. Vest is president of the National Academy of Engineering.

The **Institute of Medicine** was established in 1970 by the National Academy of Sciences to secure the services of eminent members of appropriate professions in the examination of policy matters pertaining to the health of the public. The Institute acts under the responsibility given to the National Academy of Sciences by its congressional charter to be an adviser to the federal government and, upon its own initiative, to identify issues of medical care, research, and education. Dr. Harvey V. Fineberg is president of the Institute of Medicine.

The **National Research Council** was organized by the National Academy of Sciences in 1916 to associate the broad community of science and technology with the Academy's purposes of furthering knowledge and advising the federal government. Functioning in accordance with general policies determined by the Academy, the Council has become the principal operating agency of both the National Academy of Sciences and the National Academy of Engineering in providing services to the government, the public, and the scientific and engineering communities. The Council is administered jointly by both Academies and the Institute of Medicine. Dr. Ralph J. Cicerone and Dr. Charles M. Vest are chair and vice chair, respectively, of the National Research Council.

**www.national-academies.org**

# PANEL ON MATERIALS SCIENCE AND ENGINEERING

ROBERT J. EAGAN, Sandia National Laboratories (emeritus), *Chair*
JOHN ALLISON, Ford Motor Company
EDMUND W. CHU, Alcoa Technical Center
DAVID R. CLARKE, University of California, Santa Barbara
KENNETH DAVIS, Arizona State University and Consultant
DELBERT E. DAY, Missouri University of Science and Technology
LISA DHAR, InPhase Technologies, Inc.
MARK EBERHART, Colorado School of Mines
RICHARD GAMBINO, State University of New York at Stony Brook
WILLIAM A. GODDARD III, California Institute of Technology
BRIDGETTE L. GOMILLION-WILLIAMS, The Dow Chemical Company
ELIZABETH G. JACOBS, Texas Instruments, Inc.
EDMUND H. MOORE, Air Force Research Laboratory
OMKARAM NALAMASU, Rensselaer Polytechnic Institute
THOMAS X. NEENAN, Warwick Effect Polymers
THOMAS P. RUSSELL, University of Massachusetts, Amherst
ISAAC C. SANCHEZ, The University of Texas, Austin
MARK L. WEAVER, University of Alabama
SHEREE WEN, WEN Technology and WENLAB USA
ROBERT M. WHITE, Carnegie Mellon University (emeritus)

*Staff*

JAMES P. McGEE, Director
ARUL MOZHI, Senior Program Officer
CY BUTNER, Senior Program Officer
LIZA HAMILTON, Administrative Coordinator
JONATHAN ELLSAESSER, Senior Project Assistant

# Acknowledgment of Reviewers

This report has been reviewed in draft form by individuals chosen for their diverse perspectives and technical expertise, in accordance with procedures approved by the National Research Council's Report Review Committee. The purpose of this independent review is to provide candid and critical comments that will assist the institution in making its published report as sound as possible and to ensure that the report meets institutional standards for objectivity, evidence, and responsiveness to the study charge. The review comments and draft manuscript remain confidential to protect the integrity of the deliberative process. We wish to thank the following individuals for their review of this report:

Katherine Faber, Northwestern University,
Katharine Frase, IBM Corporation,
Frank E. Karasz, University of Massachusetts, and
David C. Larbalestier, Florida State University.

Although the reviewers listed above have provided many constructive comments and suggestions, they were not asked to endorse the conclusions or recommendations, nor did they see the final draft of the report before its release. The review of this report was overseen by Alton Slay, Warrenton, Virginia. Appointed by the National Research Council, he was responsible for making certain that an independent examination of this report was carried out in accordance with institutional procedures and that all review comments were carefully considered. Responsibility for the final content of this report rests entirely with the authoring panel and the institution.

# Contents

# Summary

The Materials Science and Engineering Laboratory (MSEL) of the National Institute of Standards and Technology (NIST) works with industry, standards bodies, universities, and other government laboratories to improve the nation's measurements and standards infrastructure for materials. Its work is aligned with the mission of NIST, which is to promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life.

The MSEL consists of 149 technical staff, including 5 NIST Fellows, and 184 guest researchers, with total funding of $44.8 million at the time of this review. The MSEL is organized in four divisions: Ceramics, Materials Reliability, Metallurgy, and Polymers. A panel of experts appointed by the National Research Council (NRC) assessed the four divisions. Panel members visited these divisions and reviewed their activities.

As requested by the Director of NIST, the scope of the assessment included the following criteria: (1) the technical merit of the current laboratory programs relative to the current state of the art worldwide; (2) the adequacy of the laboratory facilities, equipment, and human resources, as they affect the quality of the laboratory technical programs; and (3) the degree to which the laboratory programs in measurement science and standards achieve their stated objectives and desired impact. In addition to these three criteria, the panel was asked by the Director of NIST to assess the projects within the laboratory conducted under the America COMPETES Act of 2007, which supports the President's American Competitiveness Initiative (ACI).[1]

On the basis of its assessment of the MSEL conducted in March 2008, the NRC's Panel on Materials Science and Engineering concluded that, for the selected portion of the MSEL programs reviewed, the staff, the projects, and many facilities are outstanding. The projects are clearly focused on the mission of MSEL. The facilities and equipment are rationally upgraded within budget constraints, with several facilities being unique; the funding provided through the America COMPETES Act of 2007 is being used effectively. Division chiefs and staff evinced high morale, attributable to several factors: clear definitions of expectations and of the processes for realizing them, strong support of the MSEL from NIST leadership and of NIST generally from the President and from the Congress (through the American Competitiveness Initiative and the America COMPETES Act), and positive feedback from customers.

Since the previous NRC assessment of the laboratory,[2] the MSEL has won internal NIST competitions to fund promising projects, terminated projects of relatively low impact, hired a few new staff and some postdoctoral temporary staff through the highly competitive NRC-administered National Academies Research Associateship Program, and upgraded experimental capabilities. The result is an organization that is strong in staff, facilities, and focus.

The relatively new MSEL program planning and evaluation process provides a framework for the MSEL to assess ongoing and new projects with regard to risk and impact. The inclusion of an assessment of risk and steps to mitigate it, along with a process to "authenticate" the need—that is, to identify and verify the need of customers—is noteworthy.

---

[1] See Domestic Policy Council, Office of Science and Technology Policy, 2006, *American Competitiveness Initiative,* Washington, D.C. "America COMPETES Act" is the short title for the America Creating Opportunities to Meaningfully Promote Excellence in Technology, Education, and Science Act of 2007 (Public Law 110-69).

[2] National Research Council, 2005, *An Assessment of the National Institute of Standards and Technology Measurement and Standards Laboratories: Fiscal Years 2004-2005,* Washington, D.C.: The National Academies Press.

The MSEL technical staff is strong and productive as measured by publication in refereed journals, output of products (such as standard reference materials [SRMs] and standard reference databases [SRDs]), awards, and strong demand by customers (U.S. industry) for advanced measurement services and standards. Staff and managers participate in and are elected by their peers to lead major technical societies, which validates the quality of the staff and enhances the reputation and stature of NIST.

Funding for various programs under the America COMPETES Act started in fiscal year (FY) 2007, so it is early to assess its impact. The projects receiving this funding are well conceived, and the first results are positive. The Ceramics Division, which is responsible for enhancing the NIST capability in the National Synchrotron Light Source (NSLS) at the Brookhaven National Laboratory, is effectively applying the funding to perform unique measurements that have already resulted in new discoveries. The enhanced research capability is significant enough that it should be managed in a way that brings broader participation from NIST in establishing priorities for the portion of beam time that NIST controls. The Metallurgy and the Materials Reliability Divisions share in executing a program to develop standards and test protocols related to the hydrogen economy—specifically, to the safety of pipelines to transport hydrogen under high pressure. A flexible research and test facility is under construction at the NIST facility in Boulder, Colorado, that holds promise of greatly enhancing the capability of NIST to study this complex problem. A related project to measure hydrogen content rapidly in order to assess materials compatibility is solid but small. The project to build the first broadband coherent anti-Stokes Raman scattering microscope is off to a good start in the Polymers Division.

Issues and opportunities for improvement are cited in the division chapters. Issues that are common to varying degrees among the divisions include the following: a large fraction of the staff is retirement-eligible, and limited hiring resulting from constrained budgets may threaten capabilities; some equipment is antiquated and some facilities are in need of upgrade, notably in Boulder. Several projects beneficially incorporated modeling and simulation, but more is needed.

In future NRC panel assessments, project presentations should briefly include the size, start date, and expected duration of a project as well as indicating linkages to other projects, if applicable, as an adjunct to the statement of partnerships and other evidence of authentication that was frequently provided. Providing data that compare MSEL capabilities with those of other laboratories would be helpful as well. Trend data for 5 years would help the panel in evaluating progress in projects and assessing the strength of programs.

# The Charge to the Panel and the Assessment Process

At the request of the National Institute of Standards and Technology, the National Academies, through its National Research Council, has since 1959 annually assembled panels of experts from academia, industry, medicine, and other scientific and engineering environments to assess the quality and effectiveness of the NIST measurements and standards laboratories, of which there are now nine,[3] as well as the adequacy of the laboratories' resources. In 2008, NIST requested that five of its laboratories be assessed: the Building and Fire Research Laboratory, the Manufacturing Engineering Laboratory, the Materials Science and Engineering Laboratory, the NIST Center for Neutron Research, and the Physics Laboratory. Each of these was assessed by a separate panel of experts; the findings of the respective panels are summarized in separate reports. This report summarizes the findings of the Panel on Materials Science and Engineering.

For the FY 2008 assessment, NIST requested that the panel consider the following criteria as part of its assessment:

1. The technical merit of the current laboratory programs relative to the current state of the art worldwide;
2. The adequacy of the laboratory facilities, equipment, and human resources, as they affect the quality of the laboratory technical programs; and
3. The degree to which the laboratory programs in measurement science and standards achieve their stated objectives and desired impact.

In addition, because NIST has begun to receive increases in funding through the President's ACI and the America COMPETES Act of 2007, the Director of NIST also requested that the assessment panels specifically examine and review the progress of all of the FY 2007-funded initiatives relevant to their respective laboratories and comment on these program growth areas explicitly in their reports.

The context of this technical assessment is the mission of NIST, which is to promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve the quality of life. The NIST laboratories conduct research to anticipate future metrology and standards needs, to enable new scientific and technological advances, and to improve and refine existing measurement methods and services.

To accomplish the assessment, the NRC appointed a panel of 20 volunteers whose expertise matched that of the work performed by the MSEL staff. Each panel member was also assigned to one of four review teams whose members' expertise matched that of the work performed by the staff in the four MSEL divisions being assessed: Ceramics, Materials Reliability, Metallurgy, and Polymers.[4]

The Materials Reliability Division review team held a 1-day meeting at the Materials Reliability Division facility in Boulder, Colorado, on March 13, 2008. The other three review teams individually visited their respective MSEL divisions at the NIST facilities in Gaithersburg,

---

[3] The nine NIST laboratories are the Building and Fire Research Laboratory, the Center for Nanoscale Science and Technology, the Chemical Science and Technology Laboratory, the Electronics and Electrical Engineering Laboratory, the Information Technology Laboratory, the Manufacturing Engineering Laboratory, the Materials Science and Engineering Laboratory, the NIST Center for Neutron Research, and the Physics Laboratory.

[4] See http://www.msel.nist.gov/ for more information on MSEL programs. Accessed August 12, 2008.

Maryland, on March 19, 2008. During their 1-day meetings they attended presentations, tours, demonstrations, and interactive sessions with the MSEL staff. Immediately following the division review team meetings, the entire panel assembled for a day-and-a-half meeting at the NIST facilities in Gaithersburg on March 20-21, 2008. During that time the panel attended interactive sessions with the laboratory managers, and also met in a closed session to deliberate its findings and to define the contents of this assessment report.

The panel's approach to the assessment relied on the experience, technical knowledge, and expertise of its members, whose backgrounds were carefully matched to the technical areas within which the MSEL activities are conducted. The panel reviewed selected examples of the standards and measurements activities and the technological research presented by the MSEL. It was not possible to review the MSEL programs and projects exhaustively. The examples reviewed by the panel were selected by the MSEL. The panel's goal was to identify and report salient examples of accomplishments and opportunities for further improvement with respect to the following: the technical merit of the MSEL work, its impact with respect to achieving its own definition of its objectives, and specific elements of the MSEL's resource infrastructure that are intended to support the technical work. These highlighted examples for each MSEL division are intended collectively to portray an overall impression of the laboratory, while preserving useful suggestions specific to projects and programs that the panel considered to be of special note within the set of those examined. The assessment is currently scheduled to be repeated biennially. While the panel applied a largely qualitative rather than a quantitative approach to the assessment, it is possible that future assessments will be informed by further consideration of various analytical methods that can be applied.

The comments in this report are not intended to address each program within the MSEL exhaustively. Instead, this report identifies key issues and focuses on representative programs and projects relevant to those issues. Given the necessarily nonexhaustive nature of the review process, the omission of any particular MSEL program or project should not be interpreted as a negative reflection on the omitted program or project.

The report's Summary first highlighted issues that apply broadly to several or all of the divisions or to the laboratory as a whole. Then, after this chapter on the charge to and approach taken by the panel, individual chapters present observations specific to the respective laboratory divisions. Comments on the progress of the programs funded under the America COMPETES Act are followed by overall conclusions.

# Ceramics Division

## SUMMARY

The Ceramics Division has programs that are well defined and focused on its mission; it has developed several state-of-the-art facilities, some of which are unique; it has a fine, productive staff; and it has an enthusiastic and effective leadership team that vigorously evaluates the technical performance of the staff. The MSEL-wide project evaluation process, while burdensome in its early implementation, has been effectively employed by the Ceramics Division to redefine its project portfolio. The division's aggregated research capabilities support materials characterization, particularly in x-ray metrology and synchrotron-based methods, and those located in the state-of-the-art Advanced Measurements Laboratory (AML) that are among the best. Proactive performance evaluation has addressed personnel issues to enhance the capability of the Ceramics Division. The staff is very productive, as measured by publications in refereed journals and the delivery of many standard reference materials and phase-equilibrium diagrams. The programs integrate theory and modeling with experimental studies, where feasible, to create a balanced portfolio.

The funds appropriated under the America COMPETES Act of 2007 have been effectively used to enhance the capability of the three NIST beam lines in the National Synchrotron Light Source, a U.S. Department of Energy (DOE) national user facility, at the Brookhaven National Laboratory in New York. Noteworthy accomplishments include fielding the most advanced synchrotron detectors and performing, for the first time, interfacial structure measurements on commercially important high-dielectric-constant gate oxides. Several industrial partners are involved in the research.

The division has made good use of the exceptional capabilities in the AML, where it has 11 modules comprising two laboratories focused on nanomechanics (using atomic force microscopy [AFM], scanning tunneling microscopy [STM], and nanoindentation) and x-ray metrology, using a custom-built parallel beam diffractometer. Both require exceptional stability of the measurement environment.

Other noteworthy accomplishments include the first combinatorial measurements of metal high-dielectric-constant oxide structures and the release of the first nanoparticle reference materials. There are also important, ongoing projects to provide a spectrum of standard reference materials, standard test methods, and standard reference databases. These projects may not appear in refereed publications, but they do appear in thousands of laboratories worldwide as essential elements of research and production activities.

## TECHNICAL MERIT RELATIVE TO STATE OF THE ART

The staff of the Ceramics Division are experienced, knowledgeable, and recognized as leading authorities in their respective fields. Evidence of the quality of the research being designed, organized, and conducted by the staff in the division includes the large number (185) of peer-reviewed publications in the past 18 months; collaborative relationships, both long and short term, with outside organizations such as the American Ceramic Society, other government agencies, and the National Cancer Institute (NCI); and national and international awards (e.g., election as fellows in technical societies and members of the National Academy of Engineering) received by staff members for their outstanding research accomplishments.

The division is engaged in a wide range of important research topics, including the analysis, compilation, and dissemination of crystallographic and phase-equilibrium databases that serve thousands of materials engineers and scientists throughout the world. These topics also include modern and timely research fields such as nanodevices for measurement and standards, nanoparticles for medical applications, and synchrotrons for measuring the nanoscale structure of solids. The fact that all three NIST synchrotron beam lines are being used to capacity is strong evidence of the usefulness, capability, and demand for these state-of-the-art facilities.

No major weaknesses were detected in the overall research program. The division staff is effectively evaluating the research projects on a timely basis and eliminating struggling projects in favor of projects in areas where future growth is more likely to occur.

## ADEQUACY OF INFRASTRUCTURE

The FY 2008 technical portfolio of the Ceramics Division totals $13,030,000, with $830,000 supporting NIST Fellows' projects. Technical work is grouped in four topical areas: nanomechanical properties, structure determination methods, functional properties, and synchrotron methods.

The capabilities of the Ceramics Division to characterize materials by x-ray and synchrotron radiation, scanning probe microscopy, and other analytical techniques are, in aggregate, state of the art, and in several cases they are unique. Eleven laboratory modules in the AML—a state-of-the-art facility with Class 10,000 to Class 100 air quality, temperature control ranging from 0.25 $^{o}$C down to 0.01 $^{o}$C, and exceptional vibration control—provide x-ray metrology and nanomechanics laboratories and facilities needed to prepare standards required by U.S. industry to develop materials and devices based on nanotechnology. The capabilities on the beam lines in the NSLS at the Brookhaven National Laboratory incorporate unique equipment designed by NIST to provide measurement capabilities used by NIST scientists as well as many industrial and university-based collaborators. These noteworthy measurement capabilities have been directly applied to fulfilling the mission of the Ceramics Division to develop analytical techniques and SRMs.

The division has worked effectively within its budget to develop its experimental capabilities, but some of the equipment (e.g., furnaces and x-ray diffraction equipment used in phase-diagram studies) has exceeded what would generally be considered its useful life.

The division has an effective approach to assessing its staff and taking appropriate actions to address performance issues. The staff who presented their work to the panel were enthusiastic and articulate in describing their generally outstanding work. The Ceramics Division should increase efforts to compete more effectively for NRC postdoctoral students, both for the immediate capability that they bring and for their potential as future permanent staff.

The productivity of the staff is outstanding, as measured by publications in refereed journals with high Institute for Scientific Information impact factor ratings. Moreover, the demand for the SRMs supplied by the division, including new reference materials developed for the emerging nanotechnology field—such as for gold nanoparticles—is evidence of the impact of the staff's productivity. Recognition in the form of an invited paper in *Science*[5] and the 2006

---

[5] S.J.L. Billinge and I. Levin, 2007, "The Problem with Determining Atomic Structure at the Nanoscale," *Science*, Vol. 316 (April 27): pp. 561-565.

Outstanding Paper Award in *Measurement Science and Technology*[6] is noteworthy. The refereed publications were appropriately supplemented by practice guides, such as *Fractography of Ceramics and Glasses*.[7]

## ACHIEVEMENT OF OBJECTIVES AND IMPACT

The Ceramics Division is meeting the NIST mission, vision, core competencies, and many of its core values. The division has suffered from limited hiring. It needs to compete more effectively for postdoctoral students in the NRC-administered National Academies Research Associateship Program. Measurement science, rigorous traceability, and the development and use of standards are, taken together, a core competence of the Ceramics Division. The division demonstrates customer focus to promote U.S. innovation and industrial competitiveness.

### Nanomechanical Properties Group

The Nanomechanical Properties Group conducts five projects, all initiated between 2006 and 2008. Two of these make effective use of NIST's comprehensive collection of state-of-the-art atomic-resolution testing capabilities in the AML: two scanning probe microscopes with capabilities including STM and AFM, two instrumented indenters with unprecedentedly small noise and thermal drift, and an optical interferometer with 1-nanometer-depth resolution.

One of the AML-related projects has resulted in a highly precise prototype microfabricated cantilever beam array for calibrating commercial atomic force microscopes. This array has been employed by five vendors of atomic force microscopes and by users of these microscopes in industry, academia, and government. The other AML-related project has focused on nanoindentation for measurements of fracture and viscoelastic properties of materials and structures—including fracture toughness measurement in thin, amorphous films.

A large component of one exciting project, focused on nanoscale-particle property measurements and standards, was initiated at the request of the National Cancer Institute in 2006. The NCI is engaged in an initiative to make use of the special properties of nanoscale particles to radically change the way that cancer is diagnosed, treated, and prevented. The NIST contribution, which makes very effective use of NIST core measurement competence, involves the development of measurements and standards for the physical characterization (with respect to size, surface area, charge, agglomeration, stability, and purity) of nanoscale particles of interest to the NCI program. The Ceramics Division-led NIST team has so far completed the first reference materials for biomedical applications, released in January 2008. These reference materials are for particles of 10, 30, and 60 nanometer diameter. Computational approaches are also under development in the Ceramics Division for specific particle properties.

Two very recently initiated projects—Mechanical Reliability Measurements for Microelectromechanical Systems (MEMS) and Piezospectroscopy Measurements and Standards—appear to be well planned and oriented toward important applications. The former employs the design of a macroscale test structure in a novel manner for microscale structures, and the latter addresses nanoscale stress mapping with a NIST-built Raman optical probe.

---

[6] R.S. Gates and J.R. Pratt, 2007, "Prototype Cantilevers for SI-Traceable Nanonewton Force Calibration," *Measurement Science and Technology*, Vol. 17, no. 10, pp. 2852-2860.

[7] National Institute of Standards and Technology, 2007, *NIST Recommended Practice Guide: Fractography of Ceramics and Glasses*, Gaithersburg, Maryland.

## Structure Determination Methods Group

The portfolio of the Structure Determination Methods Group contains a balanced set of projects that were initiated during three separate decades. The database work is among the best, and the diffraction metrology work and standards work are excellent.

The group's mission is focused on SRMs, SRDs, computational tools, and x-ray, electron, and neutron diffraction. Core competencies lie in x-ray metrology; x-ray, electron, and neutron-based structure measurements; structure determination computational tools; and phase-equilibrium and crystallography determination. Customer engagement is strong, as demonstrated by SRM distribution through vendors with instrumentation packages, SRD licensing and distribution agreements, workshops, and the collaborative partnership with the American Ceramic Society on phase-equilibrium diagrams.

The community demands that NIST continually improve SRMs; some 700 are sold each year. NIST certified the amorphous fraction of a 50:50 mixture of silicon and SRM 676a (alumina powder) to an accuracy that is considered to be the best; this is a noteworthy accomplishment. X-ray reflectometry can be used to measure the thickness and interfacial roughness of thin films repeatably. Many semiconductor industry partners demand this degree of measurement. The new work on measuring local structure is an excellent new start, and that work has been published in *Science*.[8] The long-term work on ceramic phase-equilibrium data and crystallographic databases is among the best.

## Functional Properties Group

The focus of the Functional Properties Group is measurement science, standards, and technology pertaining to the functional properties of advanced materials and devices, including ceramics and nanomaterials. The Functional Properties Group is a leader in three areas: combinatorial measurement methods, nanocalorimetry measurements, and thermoelectric measurements and standards. Of the three projects in these areas, one is almost mature—Combinatorial Measurement Methods for Advanced Complementary Metal Oxide Semiconductor (CMOS) Devices, a project that started in 2004.

There are only two or three combinatorial thin-film synthesis laboratory facilities in the United States and a similar number in Japan. The NIST facilities are among the best of these and will be further enhanced by an atomic-layer deposition tool destined for the Nanofabrication Facility (located in the AML). For example, the material currently used for the CMOS is reaching its fundamental materials limit, and thus new materials are required by the $750 billion semiconductor industry. Semiconductor customers (Semiconductor Manufacturing Technology Consortium [SEMATECH], Intel Corporation, Micron Technology, Qualcomm, NIMS Japan, and IMEC, Belgium) are partnering with NIST to address this matter.

Beyond the qualification of partners, the impact of the projects is measured by public recognition (*Science Watch*), invited NIST presentations, awards, effective partnership with users, SRM sales, database royalties, customer surveys, technology transfer to users, and patents (to a lesser degree). Regularly scheduled internal and external assessments of the Ceramics Division project portfolio by independent evaluators and customers or potential customers identified in the MSEL project management process are critical to authenticating projects'

---

[8] S.J.L. Billinge and I. Levin, 2007, "The Problem with Determining Atomic Structure at the Nanoscale," *Science*, Vol. 316 (April 27): pp. 561-565.

impact.  An excellent opportunity exists for the Functional Properties Group to develop novel, combinatorial-compatible measurement methods.  This quality work should be continued in order to help address these types of materials issues within the semiconductor industry.

### Synchrotron Methods Group

Three mature projects that began in 1993 are found in the Synchrotron Methods Group, and the impact from this effort is expected to continue to increase when new facilities are constructed within the next several years.  Support for synchrotron-related work was enhanced by funding through the America COMPETES Act, as discussed in the chapter on this funding.

## CONCLUSIONS

The Ceramics Division is conducting high-quality research on a broad range of topics that strongly support the mission and core interests of the MSEL and NIST.  The staff is using the financial resources and equipment at its disposal in an efficient manner.  The research is widely recognized as being of high quality and in many instances is judged to be among the best.

# Materials Reliability Division

## SUMMARY

The theme of the Materials Reliability Division, located in Boulder, Colorado, is the reliability of structures—from bridges to single cells. The organization of the division lines up well with its work and helps focus on the emerging areas of nanoscale reliability and cell and tissue mechanics. The division has effectively built on its core competency of mechanical property measurements and extended the applications to small sizes (nano) and biological areas. The projects are clearly focused on the mission of the MSEL, and the division has engaged its NIST colleagues in Gaithersburg, Maryland, in several projects.

The quality of the technical staff is generally excellent, and the division's laboratories are well equipped, with a few exceptions. The division programs should add enhanced modeling and theory expertise. The recent transfer of staff to augment the theoretical and modeling capability is good. Reliability testing is only part, albeit an essential part, of the science and application of reliability engineering needed by U.S. industry. The division has made excellent progress in recruiting staff by transferring two individuals from Gaithersburg, but there remains an imbalance between experimental expertise and the modeling commonly used in industry. A more visible program of three-dimensional finite-element modeling of thermal and electrical stresses is desirable. The division should increase its efforts to access expertise in other divisions at NIST and/or to add staff.

In general, the physical infrastructure facilities of the Materials Reliability Division are dated, but overall the laboratories are well equipped. The ratio of technician support to professional support seems to be very low for laboratories of this caliber and size (a situation not unique at NIST to this division).

The productivity, as measured by publications and products, for example, Charpy test specimens, is very good. Staff members have won awards, and an appropriate number of meetings were hosted. The division is a good example of executing the MSEL project evaluation process to focus its effort on projects that fulfill the mission of the MSEL.

The division has done a good job of developing new and important areas of research beyond its traditional focus. It has reached out to establish a customer base in areas such as biomaterials and has worked to reinvigorate its traditional core competency in large-structure reliability programs.

## TECHNICAL MERIT RELATIVE TO STATE OF THE ART

The technical merit of the work in the Materials Reliability Division is high, whether in the historic role of supplying Charpy test specimens or at the new frontier of exploring the application of stress measurements to biological systems. The ability to address a diversity of projects attests to the quality of the staff and the management of the division. Generally, the staff has augmented its capability by forming partnerships in the areas in which it needs help. The area of medical device reliability would benefit from added expertise in reliability analysis. In general, the technical work reviewed was excellent and in some cases unique. Theory and modeling could enrich the experimental program. Steps have been taken to augment the staff; continued additions are warranted.

## ADEQUACY OF INFRASTRUCTURE

The equipment associated with the structural materials projects of the division is often old, but it has been updated with modern electronics. The basic test equipment has some unique capabilities that were designed and constructed by NIST staff—for example, very low temperature test cells. A hydrogen test facility under construction, while not reviewed in detail, appears to be very versatile and will be a significant contribution to the work relating to the technologies targeted by the ACI and the America COMPETES Act (see the chapter below on this funding). The Nanoscale Reliability Group has constructed a unique atomic force microscope for the measurement of mechanical properties at the nanoscale and has established good teaming with industry and universities.

## ACHIEVEMENT OF OBJECTIVES AND IMPACT

The technical work of the division is grouped in three topical areas: structural materials, cell and tissue mechanics, and nanoscale reliability, as discussed below.

### Structural Materials Group

The Structural Materials Group has core competencies in mechanical testing on a macroscale and in developing standard measurement techniques for materials and properties that are critical to the nation's infrastructure. This group manages the Charpy Standard Reference Material and Verification Program, which sells several thousand units a year. All of the structural materials work is clearly focused, and the payoffs from success are clear. Good customer ties were shown—for example, in joint planning with DOE and the U.S. Department of Transportation (DOT) on hydrogen and on pipeline safety. The mature (historic) Charpy Test Sample Program has an element of science in computational models and crack tip measurements. The impact of the work is high and clear for the specific projects and more generally as it relates to understanding the failure of large, complex structures. It is a vital role for NIST to play.

This group maintains equipment capable of tensile, fatigue, and fracture impact analysis and crack tip opening analysis (CTOA) in the range of 1 N to 4.4 MN over a temperature range of liquid helium to 1000 $^{o}$C. The equipment has been installed in the laboratory for an extended period of time, in some cases having been in service since the 1960s, although periodic electronic and other upgrades have kept most of the equipment in state-of-the-art condition. Some areas, such as nondestructive evaluation, have been diminished through attrition of staff. However, this laboratory and its personnel remain fully capable as a valuable, unbiased resource for responding to critical national infrastructure testing needs, such as the investigation of the collapse of the World Trade Center's Twin Towers and now the hydrogen pipeline safety effort as one of the ACI-related programs.

The Pipeline Safety project is a basic data-gathering and measurement activity that has as a clear customer the pipeline industry. It is an indispensable standards activity. A strength of the activity is the continued development of the fracture toughness test, based on the optical measurement of the crack tip angle, as a measurement tool for evaluating pipeline safety. This laboratory operates with a relatively small permanent staff, but it has done a good job of using postdoctoral, cooperative, and other guest researchers to supplement labor needs. In addition, the laboratory is highly collaborative in much of its work, leveraging its mechanical testing expertise with others laboratories in the MSEL and other agencies, such as DOT, for funding. There is a need for extending the strain-rate capabilities for the high-speed fracture mechanics testing of pipelines to remain competitive. Data from these tests are essential for those who accurately model pipeline rupture under burst conditions.

Supplying Charpy test SRM samples is a large and important component of the work of the Structural Materials Group. This group has established itself as providing SRMs for Charpy testing to meet the ASTM (formerly known as the American Society for Testing and Materials) standards. This is the most successful SRM program at the MSEL. The Structural Materials Group supported the revision of two International Organization for Standardization (ISO) standards in FY 2007.

## Cell and Tissue Mechanics Group

One of the reasons why NIST has a history of success is that it has maintained a research program at the leading edge of developing fields where measurement methods and, eventually, standards will be central. In the rapidly developing field of tissue and cell engineering, NIST has a vital role in indentifying the crucial parameters that will need to be measured.

The Bioreactors project has established a strong and promising collaboration with a University of Colorado research group for investigating the effect of different types of forces on the histology, growth morphologies, and various tissue expressions under realistic in vitro tissue growth environments. The research being performed is elegant, conceptually simple, and likely to provide important insights into tissue growth under conditions pertinent to actual body strains. This is excellent basic research, although it is premature to judge whether it will also lead to a set of instruments that can measure tissue response characteristics that will be of essential value to the burgeoning field of tissue culture for biomedical replacements. Bringing in a biomedical industry partner could add value.

The Single Cell Mechanics BioMEMS project is one among the worldwide activities working to establish methods for the in vitro measurement of cell deformation and mechanics. Many of these activities are focused on using the mechanical response of cells as a tool for assessing disease progression and the viability of cells. The more sophisticated projects are those that combine the two- and three-dimensional response of cells to deformation with complementary mechanics-based models of the deformation to elucidate the deformation mechanisms. The current force-displacement measurements at the Materials Reliability Division using a uniaxial straining MEMS device designed and fabricated at NIST facilities is a promising approach. The reliability is limited by the lack of strain measurements in other directions as well as by any mechanics modeling to relate the measurements to the underlying histological structure of the cells. Complementary efforts and tools reside in the Ceramics Division. The Materials Reliability Division should expand interdivisional collaborations.

The single-cell tester used in this research was conceived by NIST and was fabricated by the NIST in-house MEMS facilities. The first of its kind, this tester has the potential to play a key role in increasing the understanding of tissue formation, disease progression, and disease treatment and may impact drug discovery.

Medical Device Reliability is one of the projects in the biomaterials area. Medical applications represent a significant sector of the U.S. health industry. Current unacceptably high failure rates of implantable medical devices such as pacemakers, cardiac defibrillators, and neural transmitters point to the urgent need to establish standards and to develop measurement methods to ensure quality, reliability, and consistency and so to minimize the need for repetitive procedures to remove recalled devices from the human body.

NIST is working with the International Electronics Manufacturing Initiative Medical Electronics team to address short- and long-term reliability issues with medical devices. The division has established programs to develop new measuring tools, explore methods to improve reliabilities, and develop accelerated testing standards. Teamed with companies and with the National Institutes of Health (NIH), the Cell and Tissue Mechanics Group has gathered critical data on the next generation of devices and identified key exposures of failure mechanisms. The target of this work is to reduce the failure rate to less than 0.1 percent (current levels are an order-of-magnitude higher). Although the group has organized workshops with important industry players, the division researchers would benefit from collaborating more directly with experts who can help establish more direct links between the current technical characteristics of devices and their reliability and performance. The division should apply a more focused and specific plan to address reliability issues and to include experts well versed in reliability.

The Cell and Tissue Mechanics Group has noted that parylene (coatings used in implanted probes) degrades in the presence of electrical fields and has communicated this important observation to the Food and Drug Administration.

Overall the Cell and Tissue Mechanics Group has excellent staff who are employing innovative approaches.

### Nanoscale Reliability Group

Strain engineering is an important new area of semiconductor technology. As devices have scaled down from micrometers (1,000 nm) to 45 nm today and 20 nm in the next few years, perfectly induced localized strains in device structures can enhance electron and hole mobility by more than 30 percent, leading to faster devices. Strain metrology, therefore, is indispensable for the U.S. semiconductor industry.[9]

The metrology of strains at the nanoscale is extraordinarily difficult, and no group has yet measured strains in microprocessors. The Nanoscale Reliability Group has developed its techniques using geometrically simpler structures (doped nanowires) where measurement of the stress state is less complex.

The group used transmission electron microscopy (TEM) and scanning electron microscopy (SEM) together with commercially available software tools to acquire diffraction data and lattice images with high spatial resolution (10 nm for diffraction, 0.2 nm for imaging), and then measured the changes in lattice parameters across nanowires. These data were used to produce two-dimensional strain maps in gallium nitride/indium gallium nitride (GaN/InGaN) nanowires. A goal of the work is to develop three-dimensional strain mapping, which would be a significant advance. A challenge using this approach is to interpolate the results of these measurements to actual devices.

One of the successes of the Nanoscale Reliability Group has been the use of atomic force microscopy to study localized strain on the nanoscale. The quality of the research is comparable with that of leading groups in this area, and the contributions in the area of contact resonance AFM imaging are at the forefront of such work. Complementary work using tip enhanced Raman spectroscopy provides a suite of capabilities for developing metrology at the nanoscale.

---

[9] A. Diebold et al., 2008, “Update Presentation on Metrology Roadmap,” *International Technology Roadmap for Semiconductors*, Spring 2008, U.S. Metrology Technical Working Group.

There is considerable overlap with research on nanomechanics at the NIST Gaithersburg site. The establishment of Communication Working Groups within the MSEL is a good step toward strengthening the collaborations between the different AFM groups.

Another of the strengths of the Nanoscale Reliability Group has been the evaluation of material properties at small length scales. This is a crucial technical and intellectual endeavor because many of the mechanical properties of metals and polymers are different at small lengths. For example, there is the well-known indentation size effect, wherein the hardness of many metals and polymers increases as the indentation load, and hence the size of the hardness impression, is decreased. The question arises as to whether other related mechanical properties of concern to the microelectronics industry, such as fatigue strength, are also different at small length scales and, if so, why. Few groups other than the NIST group at Boulder are tackling these important questions. One of the difficulties is that it is necessary to do both the testing and the evaluation at the same dimensions. One approach, taken both at NIST and at the Max Planck Institute in Germany (by a NIST researcher working there), is to use cyclic Joule heating to differentially strain and hence fatigue the interconnect lines while monitoring their microstructural changes with complementary electrical measurements. This work has quantified the combined effect of mechanical constraint and dimensional scaling, which were shown to dramatically alter the fatigue failure resistance of aluminum lines. Although aluminum lines are increasingly being replaced in microelectronics by copper, it is expected that this measurement approach can be readily transferred to the investigation of the fatigue of copper interconnects in devices.

The focus on nanoscale strain metrology is understandable, given the huge importance of the microelectronics industry. The group may also wish to consider the strain metrology associated with polymer electronics devices and displays. There are two distinct challenges. On the one hand, polymer-based electronics will be much larger than current silicon-based electronics, and so the strain metrology has to be performed over much larger dimensions without loss of precision. On the other hand, even though polymers generally exhibit larger compliances than those of metals and semiconductors, the strain incompatibilities with nonpolymeric components, such as interconnects and chips, are larger, and so the local strains that have to be quantified will be larger. In addition, the recent use of Green’s function calculations to perform lattice displacement mapping around a germanium (Ge) quantum dot in silicon (Si) provides an opportunity for the group to develop capability in finite-element modeling, plasticity theory, and mechanics analysis. An active recruiting program to add staff and backfill for anticipated retirements is desirable.

## CONCLUSIONS

The projects reviewed are focused on the mission of the MSEL and build effectively on the historic strength of the Materials Reliability Division in mechanical testing for reliability. Augmenting the experimental capability with modeling and simulation expertise by the transfer of staff and hiring will greatly enhance the strength of the program.

The division has developed several unique measurement tools and devices that have potential to strengthen the competitive position of U.S. industry. The recently enhanced focus at NIST on obtaining patents may be beneficial and should be used to explore the best approach for commercializing these developments.

# Metallurgy Division

## SUMMARY

The panel reviewed 20 of the Metallurgy Division’s projects and engaged the division staff in discussions of the MSEL project evaluation process. Overall, the programs reviewed are well conceived and executed, responsive to NIST’s mission, and of high scientific and technical quality. The morale of the division is generally high, which may be the result of the division’s recently improved infrastructure and facilities, increased capital spending, stable core competencies, and positive outlook toward future opportunities.

The division’s staff level has been steady from FY 2006 to FY 2008, a plateau after the preceding 10 years of decline. The division uses postdoctoral fellowships to recruit new hires. It should also consider initiatives that would attract more senior people. A related area of concern is a decline in knowledge and a rise in technology gaps resulting from the retirement of some senior staff. The division should develop a plan for knowledge capture and/or a succession plan either to mentor junior researchers or to hire senior researchers in critical technical areas.

Many of the Metallurgy Division’s staff members are recognized both internally and externally, as is evident by the awards that they have received. The division has continued to maintain exemplary visibility through the organization of workshops at NIST, publications, and conference presentations. Overall, the staff has evinced a high level of commitment and dedication.

## TECHNICAL MERIT RELATIVE TO STATE OF THE ART

The Metallurgy Division maintains a capable and engaged workforce devoted to achieving the goals of NIST. It is part of the NIST mission to increase innovation and industrial competitiveness by advancing measurement science, standards, and technologies in order to enhance economic security and improve the quality of life. The division maintains a diversity of activities including both fundamental sciences and applied studies in support of these goals.

The technical merit of the Metallurgy Division is high relative to the state of the art. In general, the quality of the research is excellent. Some examples of outstanding research are provided below.

Particularly noteworthy are efforts to standardize and improve hardness measurement, to develop atomistic modeling in support of the development of nN-level force standard for very small loads, to develop fundamentals-based quantitative models of microstructural evolution in materials for the extraction of kinetic data from experiments, and to develop a fundamental science-based understanding of the mechanisms leading to tin (Sn) whisker formation in electronic devices. Major accomplishments include the development of SRMs for hardness testing and a complete revision of the ASTM standards for hardness testing, which accomplish the goal of improving hardness as a measure of materials properties and quality control. The nN-level force standard based on gold nanowires is cutting edge. NIST continues to lead the development of thermodynamic and kinetic databases, which will have broad applicability in all areas of materials engineering. Modeling efforts directed toward sustainable energy sources and hydrogen storage are timely and necessary. The formation of tin whiskers on lead-free surface finishes has evolved into a major reliability issue in microelectronics. Research at NIST has revealed the mechanisms for whisker formation, which could lead to inhibiting it, thereby extending the service lifetimes of future components.

The in situ, x-ray stress measurement capability for sheet metals undergoing deformation was exceptional. This type of measurement, which has never been done before in sheet metals, represents a breakthrough in deformation materials metrology. NIST should continue to develop this exciting technology so that it can be applied more broadly to solve practical engineering problems.

## ADEQUACY OF INFRASTRUCTURE

The Materials Science and Engineering Laboratory's commendable capital investment is welcomed by the division staff and will enhance the laboratory's capabilities. The new tunneling characterization probe instrument adds to an already impressive magnetic thin-film fabrication and characterization capability. The strain measurement enhancement of the Kolsky bar technology will provide novel high-speed direct strain measurements, which are important and will be of interest to a broad range of researchers. The Lorenz microscope will provide state-of-the-art imaging capability for the Magnetics Group.

The division should assess its computing needs and develop a strategy for the future. The division's staff appears innovative and willing to deal with difficult problems. One of these, as mentioned, is the decline in staff associated with staff members' not being replaced upon retirement. An effort should be made to hire experienced individuals and not to rely entirely on NRC postdoctoral fellowships.

The staff expressed some concerns that traditional measurement expertise may disappear with future retirements. Such expertise gaps should be identified and a strategy developed to maintain important core competencies. An effort should be made to capture this experience through mentoring.

## ACHIEVEMENT OF OBJECTIVES AND IMPACT

The Metallurgy Division is doing well in terms of meeting its objectives of establishing standards and providing technology for supporting mainline U.S. industry (e.g., automotive, aerospace, and magnetics industries). However, it appears to have a gap in its ability to develop technologies that may be disruptive. The division should consider incentives for initiating projects that have high risk but also could have high payoff. There may be opportunities for it to accomplish this by working more closely with small and medium-sized businesses. An example would be in magnetics, where spin torque devices are a potential opportunity that should be explored, as well as magnetic sensors for biomedical applications. Small Business Innovation Research (SBIR) programs may be an important way to facilitate such efforts and should be fully explored. The current project selection process is a positive development, but it probably needs to be fine-tuned to ensure that high-risk, disruptive technologies are not filtered out.

In general, the materials profession lags other fields (e.g., biology, astronomy, and others) in effective use of Web technology for knowledge dissemination and collaboration. The Metallurgy Division is highly proficient at disseminating knowledge through publications, presentations, and in particular at workshops such as the diffusion workshops. It has an excellent opportunity to leverage the division's existing activities and to be a leader in the effective use of Web and information technology (cyber infrastructures) to disseminate information (databases and models). The current Web-accessible databases for thermodynamics and diffusion are excellent leading-edge examples that could be expanded. There are currently only limited sets of thermodynamic data available; however, these could be easily expanded and serve as best practices by other groups within the MSEL and within other government-supported research laboratories.

The Metallurgy Division has played an important technical role in a number of high-visibility failure analyses (e.g., analysis of the World Trade Center collapse, the sinking of the *Titanic*, and the failure of naval structures). The work on the *Titanic* has led to related studies of the Ellis Island Ferry and USS *Arizona* wrecks in efforts to establish the practicality of salvage and a timetable for preservation activities. Considering the vast number of aging wrecks populating the nation's waterways, the technical merit of these activities relative to the state of the art is quite high. These examples could showcase for laypersons and national leaders the Metallurgy Division's expertise and the national need for experts in metallurgy. However, the efforts referred to here largely involved full-scale structural analysis coupled with metallurgical insights and are not tied in with other computational efforts; this appears to be a missed opportunity. The impact of these efforts could be increased by identifying underlying themes of research and how they tie in with other division research areas. This would allow the Metallurgy Division to capitalize on the high visibility that such projects provide.

The use of computational materials science tools to incorporate and disseminate information and knowledge is a growing trend within the materials profession. Strengthening this activity within the MSEL and making these models available to the broader research community offer important means of increasing the impact of the MSEL.

There are good examples of cross-group and cross-division collaboration in the MSEL, and there is an excellent collegial atmosphere of cooperation. However, many of the division's major research projects are disconnected. The Metallurgy Division could increase the impact of its research through better integration of its existing expertise into some cross-group projects that would demonstrate the impact of this combined expertise. An example might be an integration of efforts in thermodynamics, kinetics, and evolution of microstructure in Pb-free electrodeposits, with (currently nonexistent) efforts at using this information to predict the key properties of solders and, in turn, structural analysis of electrical interconnects. This would make use of all of the impressive expertise in thermodynamics, phase transformations, property development, and full-scale structural analysis. Other examples might be in the use of thermal barrier coatings for improving the performance of turbine blades for power generation or in the development of magnetic sensors.

A high level of innovation was evinced within the Metallurgy Division. In particular:

- The development of high-resolution three-dimensional chemical imaging (demonstrated in multilayer stacks) is highly innovative and the first known use of this technique on crystalline structure compensating for diffraction effects. This research has significant potential for development as a high-throughput evaluation tool for crystalline nanostructures.
- The measurement of stress by means of multidirectional x-ray diffraction during sheet metal forming is a highly innovative first use of this technique. It provides a new approach for characterizing of the evolution of yield surfaces required for the simulation of sheet metal forming.

- The use of depth-resolved x-ray microbeam stress measurements to measure stresses within dislocation cells is a highly creative and unique research.

## CONCLUSIONS

The Metallurgy Division represents a unique, high-quality research effort in the development of measurement science and materials standards in selected areas of critical importance for American competitiveness. The division could enhance its effectiveness by identifying common themes to better coordinate its activities and by increasing its focus on the entrepreneurial, highly innovative sectors of the U.S. economy.

# Polymers Division

## SUMMARY

The Polymers Division has done outstanding work with an energetic and highly motivated group focused on customers and collaborations to achieve its objective of providing critical measurement solutions with a high impact. In addition, the division has done exceptionally well in executing several projects in areas of critical importance to industry (electronic materials, biomaterials, and industrial polymers), homeland security (passport security with the U.S. Department of State and the U.S. Government Printing Office), the U.S. Department of Justice (body armor), consortia (SEMATECH, the Semiconductor Research Consortium, and others), and academia. The division has been very proactive in engaging faculty and graduate students in its research portfolio and in attracting them in large numbers through the NRC-NIST postdoctoral fellowships. This has proven to be a very effective recruiting mechanism.

Energy is a significant contemporary problem; NIST in general and the Polymers Division in particular can make important contributions to the measurement science and technology related to organic photovoltaics (OPVs) and energy storage devices. Efforts in this area are slightly below an effective critical mass and should be increased if the Polymers Division is to have significant impact in this area.

The presentations to the panel did not indicate how the recommendations of previous NRC panels have been addressed, and for many of the programs presented, a plan for the future was not clearly laid out. For example, it is not clear how the Matrix-Assisted Laser Desorption Ionization (MALDI) program has been reoriented to address the previous panel's comment that "the work in the laboratory on mass spectrometry . . . (MALDI-mass spectrometry) of polymers has been very relevant. It is now time to consider whether the 'low-hanging fruit has been picked.' The laboratory may wish to consider whether the most effective use of resources is to continue these programs at their current levels."[10] Even presentations showing breakthrough results did not clearly articulate a vision for the future.

## TECHNICAL MERIT RELATIVE TO STATE OF THE ART

The polymer projects in all six of the Polymers Division's groups demonstrate outstanding technical performance in most areas, with examples of state-of-the-art accomplishments, and strike a good balance between exploring the frontiers of measurement science and technology and in transferring the know-how to U.S. industry for economic impact, strengthening U.S. competitiveness. The researchers have been publishing papers in journals, presenting at workshops, and giving invited presentations to conferences, industry, and academia. They also have received several noteworthy awards, including the Presidential Early Career Award for Scientists and Engineers, American Chemical Society fellow, and NIMS young scientist awards.

The division's programs are highly leveraged with cooperative research and development agreements, or CRADAs (e.g., the Electronics Materials CRADA with Intel Corporation),

---

[10] National Research Council, 2005, *An Assessment of the National Institute of Standards and Technology Measurement and Standards Laboratories: Fiscal Years 2004-2005,* Washington, D.C.: The National Academies Press, p. 75.

interagency agreements (e.g., Biomaterials NIH Research Project Grants [R01s], Exploratory/Developmental Grants [R21s], National Institute of Dental and Craniofacial Research/NIH grants), visitors, sabbaticals (e.g., Intel, IBM, Seoul National University), and synergistic collaborations (e.g., SEMATECH in electronics materials).

NIST-developed measurement methods can have far-reaching consequences in transforming U.S. industries and competitiveness. The Polymers Division should more aggressively cultivate relationships with analytical equipment companies, both as customers and collaborators, in order to define an easy path for technology to reach the marketplace.

## ADEQUACY OF INFRASTRUCTURE

This division of 106 people with a budget of $14 million is pursuing 22 projects with a great deal of success. However, given the resources available, the division should either obtain additional resources or consider deemphasizing programs that require substantially more resources in order to offer a reasonable probability of success. A return on investment and/or impact assessment should be done, investment should be made, and resources should be placed in those areas where a significant impact can be made realistically. The fuel cell effort is an example. While this problem is important, it is a complex problem that requires a significant investment of personnel and other resources. The level of effort currently underway is insufficient to have substantial impact. This is not a criticism of the technical level of the current effort, but rather a realistic assessment of the potential outcomes. Either more investment needs to be made or the efforts of the program should be redirected elsewhere.

The depth and breadth of the staff are impressive, as is the division’s continuing ability to attract high-quality staff, associates, and postdoctoral fellows. The objectives and the vision for building a diverse workforce will be challenging in the absence of significant hiring.

Several group leaders are serving also as principal investigators for projects. While these leaders seem to be doing an effective job of carrying out both leadership and technical functions, this may not prove to be an effective long-term strategy.

There was not apparent within the division a sufficient computational materials science effort to complement the outstanding experimental accomplishments. In several areas, theory and simulations to interpret the experimental measurements could likely provide additional predictive power. Hiring a knowledgeable senior computational scientist or NRC postdoctoral fellows with theoretical expertise may be an effective way to initiate an effort in this direction.

## ACHIEVEMENT OF OBJECTIVES AND IMPACT

### Electronic Materials Group

The Electronic Materials Group has done an outstanding job of developing measurement methods for the use of polymeric materials for the electronics industry. The group has extensively collaborated with major industrial customers (Intel, IBM, and Advanced Micro Devices), consortia (e.g., SEMATECH), and academia. The effort is currently targeting opportunities to develop similar methods for emerging electronics areas (flexible electronics, OPVs, and energy). Its efforts in dimensional metrology with Intel and other collaborators using critical-dimension small-angle x-ray scattering (CD-SAXS) can complement, validate, and augment existing destructive and nondestructive pattern shape measurement technologies.

The high-quality, important, fundamental studies on understanding the materials sources of line-edge roughness are of significance in driving the electronics industry down Moore's curve. An example is the development of measurement methods delineating the rheological basis for residual stress in patterns printed by nanoimprint lithography. It is important to apply this technology for sub-25 nm CMOS and non-CMOS patterning applications.

The integrated suite of measurement capabilities, including x-ray techniques coupled with spectroscopy, serves as an example of how this group has been able to illustrate the importance of conjugated plane tilt and side chain interdigitation in explaining the serendipitous observation of high conductivity in pBTTT (poly(2.5-bis(3-quaterdecylthiophene-2-yl)-thieno[3.2-b]thiophene), a thiophene-based copolymer.

### Nanostructured Materials Group

The Nanostructured Materials Group has 14 people working on a large number of projects that do not seem to be related. For example, the templated assembly of block copolymers, fate of nanoparticles in biological systems, and fuel cell membrane efforts have little in common. Nevertheless the projects, such as the block copolymers work, appear to be well thought out and tightly coupled with industry and likely to have a broad impact. The measurement methods developed for gold and titanium dioxide nanoparticles and their aggregation and characteristics in biological systems are rigorous.

### Processing Characterization Group

The 19 people in the Processing Characterization Group focus on nanotube metrology, directed assembly, and microrheometry. In the area of microrheometry, significant resources are to be deployed in the future in addressing measurement needs of complex fluids with rheology, spectroscopy, microscopy, and microfluidics. The Nanotube Metrology Program is outstanding and has made noteworthy progress since 3 years ago when demonstrated concepts during the previous NRC assessment were only at the idea stage. The integrated approach employing key techniques in dispersion, separation, and standards development with nanotubes is an example of the depth and breadth that this group brings to bear on addressing important measurement problems.

### Biomaterials Group

The Biomaterials Group has made impressive progress in the past 3 years. The project on dental materials has been well refocused and reorganized to be of high impact by concentrating on improving the understanding of the fundamentals of composite shrinkage, stress development, tooth and composite interphase, and interphase stability under bacterial challenge in order to develop clinically relevant information related to shrinkage and microleakage. There is an opportunity for this group to find a pathway for biomarker materials to become available to U.S. industry and academia, as they can have a high impact on biomedical research. The Quantum Dot Program is exemplary in this regard in terms of quality of work.

The Protein Preservation project systematically examined the relationship between material properties and protein stability using a model system and found that there is a correlation between local glass dynamics and protein stability in glass. This group has developed a benchtop surrogate for neutron backscattering using steady-state fluorescence techniques.

The major emerging industry in regenerative medicine will continue to raise multifactorial problems in materials science. There will be a growing and continuing need for standards and metrology that address the needs of this industry, with NIST uniquely positioned to play a large role. Progress is being made in this regard and, in particular, there has been close cooperation with ASTM to provide leadership in the development of reference scaffolds for tissue engineering.

### Characterization and Measurement Group

The Characterization and Measurement Group has enjoyed great success in its Machine-Readable Travel Documents project, in which it tackled an important problem for the U.S. Department of State and carried it out remarkably well. This group has the capabilities needed to address challenging technical problems quickly and well.

The group is performing a study of the ballistic resistance of polymeric materials by examining compromised body armor. The results obtained using a folding test apparatus designed by the group motivated the U.S. Department of Justice’s National Institute of Justice to include folding protocols in the draft of its current ballistic armor standard. The group should pursue opportunities for beneficial follow-on projects.

The project on Thermal Properties at the Nanoscale has developed new measurement techniques for nanothermomechanometry. One application to carbon fullerenes ($C_{60}$) was mentioned to the panel. It has promising potential for other applications.

### Combinatorial Methods Group

The Combinatorial Methods Group has done an outstanding job of retaining the best of an open-source consortium model while expanding the underlying core strengths of the group to probe complex interfaces and thin films. The group has accomplished impressive developments of a library of microfluidic surface-initiated grafted copolymers with systematic gradients in chemistry, architecture, and molecular weight. It has also developed an impressive microfluidic technique to determine copolymer reactivity ratios.

## CONCLUSIONS

The Polymers Division is doing an effective job in building an outstanding research team. The team is talented, energetic, and motivated to have an impact on polymer measurement science and technology. The division should review its programs to ensure a critical mass per program by either providing more resources or trimming the number of programs. Building some computational work is important to complement the outstanding experimental accomplishments. In several areas, theory and simulations to interpret the experimental measurements could likely provide additional predictive power. Energy is a significant contemporary problem, and NIST in general and the Polymers Division specifically can make important contributions to the measurement science and technology related to OPVs and energy storage devices.

# Programs Funded Under the America COMPETES Act

## SUMMARY

The funding under the America COMPETES Act started in FY 2007, so it is early to assess its impact. The projects benefiting from the funding are well conceived, and the first results are positive. The Ceramics Division is responsible for enhancing the NIST capability in the National Synchrotron Light Source at the Brookhaven National Laboratory. It is effectively applying the funding to attain new capability such as interfacial structure measurements on high-dielectric-constant gate oxides. The enhanced research capability is significant enough that it should be managed in a way that brings broader participation from NIST in establishing priorities for the portion of beam time that NIST controls. The Metallurgy and the Materials Reliability Divisions share in executing a program to develop standards and test protocols related to the hydrogen economy—specifically, to the safety of pipelines to transport hydrogen under high pressure. A flexible research and test facility is under construction at the NIST facility in Boulder that promises to greatly enhance the capability of NIST to study this complex problem. A related project to measure hydrogen content rapidly in order to assess materials compatibility is solid but small. The project to build the first broadband coherent anti-Stokes Raman scattering microscope is off to a good start in the Polymers Division.

## CERAMICS DIVISION

A strong core competence of the Ceramics Division is in the area of x-ray measurements on materials, in which the use of a synchrotron as the x-ray source represents a very important class of measurement technique. Synchrotrons provide a source of x-ray radiation that is intense, bright, collimated, tunable, and polarized—all characteristics not available from laboratory x-ray sources. This technique offers uniquely accurate subnanometer-resolution measurements of the electronic, chemical, and spatial structure of advanced materials, including semiconductor electronic materials, polymeric materials, nanotubes, catalysts, and biomaterials.

The Synchrotron Methods Group within the Ceramics Division focuses on three important technical areas: the development of synchrotron measurement methods, material structural determination using synchrotron measurements, and synchrotron beam-line operations at the National Synchrotron Light Source, a DOE national user facility at the Brookhaven National Laboratory in New York. NIST's Synchrotron Methods Group operates three beam lines (experiment stations) that have x-ray wavelengths appropriate for measurements on materials spanning the entire Periodic Table of the Elements. The NIST group is widely recognized as a pioneer and leader in the development and application of synchrotron-based techniques, and opportunities to use the NIST beam lines are in high demand. Although the facility operates full 24-hour days, industry and academia applicants seeking facility measurement opportunities exceed capacity by nearly 2 to 1.

The expertise of the NIST team lies principally in the area of specialized detectors. The funding under the America COMPETES Act has been directed toward several specific capability upgrades:

- A state-of-the-art analyzer and multi-element detector for near-edge x-ray absorption spectroscopy (NEXAFS) capability was installed in 2007 on beam line U7A. In addition, funding has been committed to the NIST Electronics and Electrical Engineering Laboratory in Boulder, Colorado, for the development of a detector to provide unprecedented NEXAFS sensitivity and selectivity. This work is in progress and slated for completion in December 2009. The application of this capability is in the surface chemistry and molecular structure of materials, including self-assembled monolayers, deoxyribonucleic acid (DNA), proteins, other biological materials, organic and molecular electronics, polymer surfaces and interfaces, catalysts, and nanotubes.

- A new detector for extended x-ray absorption fine structure capability was installed in 2007 on beam line X23A2. The application of this capability is in measuring local atomic and electronic structures from nanolayer thin films to bulk materials and from crystalline to highly disordered materials of higher molecular weight (beginning with titanium on the Periodic Table).
- A new, high-throughput endstation for x-ray standing wave spectroscopy capability was installed in 2007 on beam line X24A. This line is used for the lower- and middle-molecular-weight materials.
- A major project, in excess of $1 million capital, was begun in partnership with Sandia National Laboratories to implement a new variable kinetic energy x-ray photoelectron spectroscopy (VKE-XPS) capability on beam line X24A for high-spatial-resolution measurements. The new surface measurement capability will be completed in 2008, and enhanced depth measurements, to less than 1 nanometer, will be completed in 2009. A scientific program using the high-throughput VKE-XPS was initiated with SEMATECH to study next-generation transistor gate layer structures.
- A novel three-dimensional VKE-XPS chemical microscope is under development, also for beam line X24A. This capability will provide strategic chemical and structural insights in nanotechnology applications such as transistor gate-stacks, organic electronics, MEMS lubrication, self-assembled-monolayer templates, and catalysts. The project is a Phase II SBIR collaboration slated for delivery in the summer of 2009.
- Owing to the unexpected flat funding under the America COMPETES Act in 2008, two projects were put on hold: the Synchrotron Enhanced Scanning Tunneling Microscopy project and the Modernized High-Resolution Diffraction Beamline project.

An exciting and very timely development is the DOE announcement that a new, billion-dollar beam line—NSLS II—will be built on the Brookhaven site, with groundbreaking in 2009 and occupancy starting in 2012. The x-ray radiation source will be 10,000 times brighter than the current NSLS, with the potential for major enhancements in measurement capabilities. The existence of the NIST ACI Synchrotron project enables NIST to plan construction of two new state-of-the-art beam lines in the new facility. The NIST team has already begun responding to this exciting and unique opportunity to maintain leadership in synchrotron-based materials measurements.

The capability emerging from the enhanced funding is broadly applicable to NIST programs; therefore, it may be desirable to establish a formal process for prioritizing the projects that are performed during the time that the facility is under NIST control. (The DOE user-facility process that competitively selects projects determines who uses the beam lines the remainder of the time.)

## MATERIALS RELIABILITY DIVISION

The hydrogen pipeline safety project is part of a coordinated NIST program to develop the infrastructure for a hydrogen economy. The Metallurgy Division is focusing on research and development test methods to look at issues such as developing hydrogen-resistant alloys and looking at supersaturation effects on alloys. The Materials Reliability Division has taken responsibility for measuring the mechanical properties, in the presence of hydrogen, of existing and new high-strength pipeline steels. The purpose is to ascertain whether, and at what pressures, existing gas or petroleum pipelines in the United States could be used for shipping hydrogen around the country in preparation for a possible hydrogen fuel economy. This is an essential material property measurement activity for which the Materials Reliability Division is well equipped based on its past and present experience in bulk material property mechanical testing and pipeline testing. The facility that the division needs for extending its capabilities to testing in hydrogen is under construction and will give NIST at Boulder an unrivaled facility for testing pipeline steels under realistic hydrogen transportation conditions.

## METALLURGY DIVISION

One project in the Metallurgy Division modestly funded through the America COMPETES Act is focused on competitiveness relative to the hydrogen economy. Since the project is at an early stage, an assessment of it may be premature; however, in general it is off to a good start. It is the Hydrogen Distribution (hydrogen embrittlement of steels) project, which has a sound technical approach and is a worthwhile activity. NIST has the required expertise to meet the project's objectives. The level of coordination between MSEL divisions on this project is excellent. The tie between this project and increasing American competitiveness is somewhat tenuous, but the transportation of gaseous hydrogen is clearly important to the success of the hydrogen economy.

A second project receiving funding through the America COMPETES Act is an effort to develop a rapid high-throughput measurement of hydrogen content for assessing novel materials for hydrogen storage. This Hydrogen Storage project is technically sound and has demonstrated the feasibility of a new measurement approach (infrared emissivity) for attacking this problem. The magnitude of the effort may be too small to make a major impact. If successful, when the hydrogen economy becomes a reality, this project could assist in spawning new industries, thus improving U.S. competitiveness.

## POLYMERS DIVISION

The Bioimaging Program and efforts in optical coherence microscopy, broadband coherent anti-Stokes Raman scattering (CARS) microcopy, and the associated technology efforts fit the mission of ACI. The Optical Coherence Microscopy Program is on its pathway to commercialization with an SBIR Phase III program. CARS technology is unique because of its ability to image cells with three-dimensional spatial resolution, non-invasively and without using labels. Using contrast in vibrational spectra, CARS microscopy can provide physical and chemical information from the cells to characterize their response to different materials as well as determine differences in spatial heterogeneities between normal and diseased cells.

The Bioimaging Program built the first broadband CARS instrument in 2004, has more recently developed time-resolved detection methods to reduce nonresonant background, and has developed algorithms for faster and flexible spectral recovery.

# Overall Conclusions

The Materials Science and Engineering Laboratory is meeting its stated objectives of providing state-of-the-art metrology services to both traditional industry and emerging industries. The partnerships with other federal agencies, industry, and universities to extend the MSEL's expertise into the biology and medical fields are noteworthy. The laboratory's facilities are very good, given the budget constraints, and a number of unique and state-of-the-art experimental capabilities exist in the MSEL. The MSEL programs could benefit from a modest increase in theory and modeling to complement the strong experimental studies. The laboratory's project evaluation process to ensure focus on the needs of NIST's customers has had a beneficial effect. Progress on the recently initiated ACI-related programs is excellent.