NIST Special Publication 1217

# A workshop report on “Electron Microscopy Frontiers: Challenges and Opportunities”

John Bonevich
Ann Chiaramonti Debay
Andrew Herzing
Bob Keller
June Lau

This publication is available free of charge from:
https://doi.org/10.6028/NIST.SP.1217

NIST
National Institute of
Standards and Technology
U.S. Department of Commerce

**NIST Special Publication 1217**

# A workshop report on "Electron Microscopy Frontiers: Challenges and Opportunities"

John Bonevich
Ann Chiaramonti Debay
Andrew Herzing
Bob Keller
June Lau
*Material Measurement Laboratory*

This publication is available free of charge from:
https://doi.org/10.6028/NIST.SP.1217

December 2017

U.S. Department of Commerce
*Wilbur L. Ross, Jr., Secretary*

National Institute of Standards and Technology
*Walter Copan, NIST Director and Undersecretary of Commerce for Standards and Technology*

Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.

**National Institute of Standards and Technology Special Publication 1217**
**Natl. Inst. Stand. Technol. Spec. Publ. 1217, 41 pages (December 2017)**
**CODEN: NSPUE2**

**This publication is available free of charge from:**
**https://doi.org/10.6028/NIST.SP.1217**

MARCH 8 – 9, 2017
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
GAITHERSBURG, MD 20899

## Meeting Organizers

John Bonevich
Materials Science and Engineering Division
john.bonevich@nist.gov
(301) 975-5428

Ann Chiaramonti Debay
Applied Chemicals and Materials Division
chiaramonti@nist.gov
(303) 497-5701

Andrew Herzing
Materials Measurement Science Division
andrew.herzing@nist.gov
(301) 975-2860

Bob Keller
Applied Chemicals and Materials Division
bob.keller@nist.gov
(303) 497-7651

June Lau
Materials Science and Engineering Division
june.lau@nist.gov
(301) 975-5711

About the image: The metaphor of inferring the state of the forest through the study of a few selected trees has often been invoked to describe electron microscopy sciences. For this strategic planning, the organizers find this image of a trail through the varying flora and terrain appealing. This photograph of the Resurrection Pass was belonging to one of the organizers may be freely distributed in its unaltered state.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

## EXECUTIVE SUMMARY

1. Workshop Purpose:
    a. Identify strategic areas of investment in electron microscopy (EM) to maximize returns for our stakeholders.
    b. A self-assessment of Material Measurement Laboratory (MML)'s present EM footprint as a preamble for a microscopy strategic plan.

2. Approach:
    a. Chose speakers from three different industry sectors, well-aligned with the MML strategic plan to help us identify where we need to be.
    b. Conducted an inventory of MML's EM capability to see where we are today.
    c. Brainstormed ways to help us get closer to where we want to go.

3. Conclusions
    a. Reoccurring themes: *in situ*, *in operando*, dynamics, high-throughput, temperature, 3D, and data
    b. Problems tend to be interdisciplinary and require significant coordination
    c. MML and her organically-grown EM portfolio
    d. Optimization needed, but how?

4. Recommendations:
    a. EM portfolio assessment
    b. Recalibrating our internal compass
    c. Improving the EM community experience
    d. Innovative ideas within reach

5. Impact aspirations: By 2025, we would like NIST MML microscopy to be at the consciousness forefront of US industries as the go-to place for EM consultation and new measurement partnerships. We want to be the EM idea clearinghouse for US industries.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217


**Abstract**

For two days beginning on March 8, 2017, a planning workshop entitled "Electron Microscopy Frontiers: Opportunities and Challenges" was sponsored by the Material Measurement Laboratory (MML) of the National Institute of Standards and Technology (NIST). Electron microscopists across three MML divisions (642, 643, and 647) and two campuses (Boulder and Gaithersburg) co-organized this grass-root event. The organizers (in alphabetical order), John Bonevich, Ann Chiaramonti Debay, Andrew Herzing, Robert Keller, and June Lau, contributed equally to this workshop as well as this final report.
The information contained herein is based in part on the results of the workshop, which was attended by stakeholders working in the field of electron microscopy (EM), as well as non-microscopy experts in selected fields. This report presents the expert perspectives of the participants, but is not intended to be taken as prevailing opinion across the represented field. Ideas from a series of conversations among the organizers prior to, and following the workshop, are also stated in this writing.



**Key words**

Electron microscopy; metrology needs; planning.

# TABLE OF CONTENTS

**1 Report Summary ........ 1**
1.1. Workshop Purpose ........ 2
1.2. What does EM in MML look like? ........ 2
1.3. Who came to speak and why? ........ 5
1.4. What we heard ........ 5
1.5. Is our 'today' consistent with where we need to be tomorrow? ........ 6
1.6. What needs to happen? – Our recommendations ........ 8
1.6.1. EM portfolio assessment ........ 8
1.6.2. Recalibrating our internal compass ........ 9
1.6.3. Improving the EM community experience ........ 10
1.6.4. Innovative ideas within reach ........ 11
1.7. Impact aspirations ........ 12
**2 Meeting agenda ........ 13**
**3 Findings from the keynote session ........ 16**
3.1. Who came to speak? ........ 17
3.2. Speakers Charge ........ 17
3.3. When tools are available, science discovery is accelerated ........ 18
3.4. NIST role ........ 18
3.5. Summary ........ 19
**4 Findings from the high-performance computing session ........ 20**
4.1. Why we chose Nanoelectronics and High-Performance Computing as a topic.. 21
4.2. Present NIST footprint ........ 21
4.3. Who came to speak? ........ 22
4.4. Where some consensus emerged: ........ 22
4.5. Where can MML microscopists make an impact? ........ 23
4.6. Summary ........ 24
**5 Findings from the biological and soft matter validation session ........ 25**
5.1. Why we chose Biological and Soft Matter Validation as a topic? ........ 26
5.2. Who came to speak? ........ 26
5.3. Issues highlighted during the Biological and Soft Matter Validation Session .... 27

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

**6 Findings from the metal additive manufacturing session ........ 28**
6.1. Why we chose metal additive manufacturing as a topic? ........ 29
6.2. Present NIST footprint ........ 29
6.3. Who came to speak? ........ 30
6.4. Where everyone agreed: melt pool, melt pool, melt pool ........ 30
6.5. Where can MML microscopists make an impact? ........ 31
6.6. Summary ........ 32
**Acknowledgements ........ 33**
**References ........ 33**

## LIST OF TABLES

Table 1. MML-EM asset, access model, usage, and annual operation budget ........ 4
Table 2. MML investments in EM measurement science since FY 2012 ........ 5
Table 3. Nanoelectronics/Computing Alignment with MML Capabilities ........ 24
Table 4. Nanoelectronics/Computing Needs: Scientific vs. Direct Tech Transfer ........ 24

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

# 1 REPORT SUMMARY

## 1.1. Workshop Purpose

Electron microscopy is a core enabling capability that is foundational for materials science research because of its unsurpassed ability to image and characterize matter at sub-nanometer length scales. EM sees wide application across diverse fields, including electronics and semiconductors, metallurgical alloys, polymeric and biological systems. Advances in chemical, compositional, and structural analysis using electron micro- and nano-probes have decoded many complex relationships between structure and property, enabling continued progress in the development of improved, advanced materials for present and future industries. However, emerging technologies associated with the rapid development of advanced materials continue to place increased demand on the ability of electron microscopy to provide solutions.

The current MML EM environment developed over many years through the gradual growth of several concurrent and independent Division-level sub-groups. This has sufficed for the purposes of competent EM support for numerous materials science and engineering and chemistry problems prioritized by those Divisions, as well as occasional forays into new EM technique development. However, this operating model is becoming limited in its ability to effectively address the rapidly-growing demands resulting from accelerated materials discovery in recent years.

This workshop enabled the organizers to identify specific areas of opportunity where MML-led EM measurement advances have the potential to result in widespread industrial impacts. Identifying such opportunities also required us to candidly self-assess the suitability of MML's present EM inventory (instruments, expertise, staffing/teaming, resource allocation) and operating environment from the perspective of whether we are now well-positioned to generate the required innovations.

## 1.2. What does EM in MML look like?

Looking outwards, MML has historically been very responsive to the needs of industry. We have solicited and continue to solicit input from industry, and have made large investments in microscopes and technique development as a response. MML continues to enjoy partnerships with other federal agencies, (examples: National Institute of Health (NIH), Food and Drug Administration, Consumer Product Safety Commission, National Aeronautics and Space Administration, Department of Energy (DoE), Department of the Interior, Department of Transportation, and the Department of Defense) where our mission spaces intersect.

Internally, MML consists of six science divisions across two primary campuses – roughly two each with expertise in biological, chemical, and materials science disciplines. Three of these six divisions own and operate electron microscopes. Table 1 lists the microscopes that MML owns or operates, their approximate cost, and the inaugural year of service. Within the last 20 years alone, MML has procured scanning and transmission electron microscopes (SEM and TEM respectively), and associated

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

instrumentation, valued at over $15M, which does not include service contracts, a-la-carte repairs, peripheral equipment refresh and upkeep, or consumables. Table 1 is also color-coded to show the access model and the annual operational budget for each instrument, where data is available. The access models are either shared-use based (Nexus, PIF), or Division-centric. From Table 1, instruments in blue are part of the Microscopy Nexus, which consists of microscopes owned by two divisions and is administered by division 642. Instruments in green are part of the Precision Imaging Facility (PIF). These instruments are owned by the Physical Measurement Laboratory but are co-operated by div. 647 personnel. All other microscopes listed in Table 1 have restricted access based on divisions, projects, or PIs.

In addition to equipment and operational investments, each division also own a number of expensive (>$50k) pieces of peripheral equipment such as plasma cleaners and ion-mills. Furthermore, MML (since its inception) has made several investments towards the development of new capabilities in EM. Table 2 presents a summary.

Furthermore, NIST is currently in a strategic partnership with the NIH's National Cancer Institute and the University of Maryland (UMD)'s Institute for Bioscience and Biotechnology Research to advance biological structure determination at high spatial resolution through cryo-EM. The partnership will build a research program in cryo-EM that aligns well with the NIST mission and is expected to have a wide impact on the academic and industrial research communities. NIST has dedicated over $1.5 million to the procurement of new cryo-EM instrumentation for a 'shared use' facility (location not yet finalized), and managed by the NIST-NIH-UMD partnership. Active areas of research include Cryo-EM Structure Validation as well as modeling of Cryo-EM structures relevant for biopharmaceuticals. For structure validation, NIST contributes to developing a framework to standardize the statistical reporting and validation of conformationally heterogeneous structures determined by single-molecule cryo-EM. NIST also will develop model systems that advance the application of cryo-EM to problems in pharmaceutical research (e.g., mAb structure, aggregate structure, mAb-target structure). These efforts are highly complementary to existing NMR and neutron scattering activities.

Considered in whole, MML has devoted a substantial amount (approx. $24.3M) of its resources towards maintenance, upgrade, and innovations in EM. How might we manage such a portfolio in a landscape of changing national priorities and decreasing Congressional appropriations?

**Table 1.** MML-EM asset, access model (blue – Nexus, green - PIF), usage, and annual operation budget

| Equipment | Division | Inaugural year | Purchase price (k$) | Users | Annual budget (k$) |
|---|---|---|---|---|---|
| Hitachi S4700 FE-SEM | 642 | 2001 | ---- | 45 | 375 (PM contracts), 40 (operations) |
| JEOL JSM7100 TFE-SEM | 642 | 2013 | ---- | | |
| Philips EM 400 TEM | 642 | 1999 | ----❖ | | |
| JEOL JEM 3010 TEM | 642 | 1995 | ---- | | |
| FEI Titan TEM/STEM | 642 | 2009 | ---- | | |
| Philips CM 30 TEM | 642 | 1991 | ---- | | |
| FEI Quanta 200 E-SEM | 643 | 2007 | ---- | | |
| ASPEX Automated SEM | 643 | 2005 | ---- | 4 | NA |
| FEI Apreo E-SEM | 643 | 2017 | ---- | 5 | |
| FEI Nova NanoLab 600 FIB | 643 | 2006 | ---- | 14 | 72 |
| FEI Helios 660 FIB | 643 | 2011 | ---- | 5 | 80 |
| JEOL JXA 8500F microprobe | 643 | 2006 | ---- | 6 | 53 |
| FEI Titan TEM/STEM Probe-corrected | 643 | 2007 | ---- | 6 | 205 (PM) |
| TESCAN FEG-SEM | 643 | 2012 | ---- | 5 | 40 |
| Zeiss Gemini 300 VP-TFE-SEM | 647 | 2017 | ---- | 4 | NA |
| FEI Quanta 400 E-SEM | 647 | 2016 | ----❖ | 5 | NA |
| LEO 1525 TFE-SEM | 647 | 2000 | ---- | 5 | 26 (PM), 7 (Ops) |
| JEOL 2000FX TEM | 647 | 2003 | ---- | 5 | 37 (PM), 5 (Ops) |
| JEOL ARM200F TEM/STEM Probe-corrected | Non-MML | 2012 | ---- | 4 | MML share: 35 (PM), 5 (Ops) |
| Zeiss Auriga FIB | Non-MML | 2012 | ---- | 15+ | MML share: 35 (PM), 10(Ops) |
| Zeiss Orion Plus HIM | Non-MML | 2012 | ---- | 3 | MML share: 5 (Ops) |
| **TOTAL MML Assets/Ops** | | | ***$19,095,000*** | ***131+*** | ***>$580,000/yr*** |

**PIF instruments were acquired with ARRA funds, not directly provided by MML. However, MML does pay for maintenance and operations costs. If we add in PIF instrument costs, then the total reaches $25,179,000.*

❖ Cost associated with used-equipment transfer agreement

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

**Table 2.** MML investments in EM measurement science since FY 2012

| Project | Division | FY funded | Budget (k$) | FTE | NRC post-docs |
|---|---|---|---|---|---|
| GHz stroboscopic TEM | 642 | 2016 - 2017 | 650 (one-time) | 0.5 | 1 |
| Pixelated Detection System for 4D Scanning (S)TEM | 643 | 2016 | 175 (one-time) | 2.0 | |
| Transmission EBSD | 647 | 2012-2014 | 200/yr | 2.0 | 1 |
| Transmission SEM Imaging | 647 | 2014-2017 | 225/yr | 1.5 | 2 |
| AFM in SEM | 647 | 2016 | 125/yr | 0.5 | 1 |
| Cryo EM (NIST-NIH-UMD) | MML | 2017 | 1,500(one-time) | | |
| **TOTAL MML Project Investments** | | | **$4,600,000** | **6.5/yr** | **5** |

## 1.3. Who came to speak and why?

The organizers chose three areas of focus for this workshop: Nanoelectronics and High-Performance Computing, Biological and Soft Matter Validation, and Metal Additive Manufacturing. These are wide ranging industry sectors well-aligned with the MML strategic plan, chosen with anticipation that field-independent trends and needs will emerge by the end of the workshop. To gain a fuller appreciation for the connections between EM advancement and materials innovations, we kicked off the meeting with a pair of keynote speakers whose credentials include deep foundational EM knowledge and broad ties to materials science. The names of the speakers and their presentation titles can be found in section 2 of this document.

## 1.4. What we heard

We heard that EM and materials science have been coevolving for more than half a century. Materials science and engineering often drives electron microscopy. Materials discovery is occurring at a breakneck pace, driven in large part by advances in sophisticated computational capabilities. However, the ability to experimentally verify and validate remains a critical bottleneck. This can result in an insufficient, overly conservative, and semi-empirical macroscale approach to structure and component design, when electron microscopy is not properly involved. Materials science and engineering can therefore help to define what new types of electron microscopy measurements must be developed, to address specific problems of verification and validation.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

Conversely, and concurrently, electron microscopy sometimes drives materials science and engineering; this however, the organizers realize comes with a caveat. The measurement science of electron microscopy can lead to new approaches for interrogating the nature of engineered materials or naturally-occurring substances. When such approaches are applied to materials, then new insights can be gleaned about their functionality, driving innovative uses and prompting advances in material development. While microscopy can drive materials science and engineering, microscopists must be mindful to not simply perform electron microscopy experiments "because they can," especially in times where financial, infrastructural, and time resources are few and precious.

Recurring EM themes common to the technical sessions were: standards, data quality standards, and integration, *in situ, in operando*, dynamic, high-throughput, temperature, and 3D. Conspicuously absent was any mention of the need for higher spatial resolution, which is often a hallmark theme articulated by those driven by non-industry sector interests. This means that contrary to some opinions, participants in this meeting did not feel limited in their problem-solving by the inability to resolve certain atomic lattice spacings in materials.

It is the organizer's opinion that the scale of the efforts required to address some of the critical needs highlighted in the workshop extends well beyond MML's present operational capacity, as the research problems represent measurement science solutions of considerable scope. For example, designing custom *in operando* correlative systems to provide multiple forms of physical property measurements during application of an external force, coupled with material characteristics. We believe this is not simply the case for more microscopes, instead, we need to use them better.

### 1.5. Is our 'today' consistent with where we need to be tomorrow?

Table 1 shows a diverse arsenal of electron microscope and related measurement equipment that is available in MML, including 10 SEMs, 7 TEM/STEMs, and 5 ion-based instruments. These tools are spread relatively uniformly across MML, including the Boulder Division, providing considerable measurement capability that is suitable for supporting many materials- and biology-centric efforts. Even with this impressive inventory of instruments, the organizers felt that the MML's EM operational model, while sufficient for many years, is not optimized for handling the large-scale, interdisciplinary approaches required to solve problems articulated at this meeting. To produce a significant impact on some of the problem scope we heard, we envision that some level of strategic planning activity for our EM resources will be required. Part of this strategic planning could point towards investments in microscopes optimized for *in situ*, high throughput, and 3-D (to name a few ideas expressed in the meeting). Another part of the strategic planning will depend on MML microscopists coming together to form a tight, collaborative community.

EM instrumentation, technique development, and advancements are premium enterprises. We recognize that the present MML portfolio in EM instrument

investments was a direct result of both annual increases in NIST STRS for the last 10 years, and a historically favorable overhead structure for capital equipment and service contracts. It also reflects the longevity of independently operated project spaces mentioned earlier. Compounding the recent overhead structure change with an environment of flat or declining research budget, we believe that creative means to simultaneously sustain our core instrumentation and generate effective innovation must be considered.

Even as Table 1 shows an impressive scale of capital investment over the last two decades, the organizers found that it conceals many barriers towards smooth, large-scale coordination. The effective use of electron microscopes requires, as a rule, extensive training and hands-on experience. With the diverse backgrounds of MML scientific and technical staff, spanning numerous forms of physical science and engineering, only those with significant materials-related experience will likely have had any meaningful exposure to these types of instruments; and those levels of exposure will vary greatly from staff member to staff member. As a result, we find that out of several hundred highly-qualified scientific and technical staff members in MML, only a small fraction of them have an in-depth understanding of the microscopes, and can generate high-quality data. Therefore effectively, most MML people with that experience are serving dual roles of performing measurement research and supporting others' projects. That model, if not carefully managed, can conflict with our ability to perform microscopy-centric measurement science that could provide innovative solutions to key materials problems.

We found that coordination of the critical EM-centric measurement science role with the equally-critical support role is a problem at the most basic level. Consider the following example of EM workflow in MML, involving *in situ* measurements, which was one of the most frequently cited capability and access needs during the workshop. The following example, which describes work requiring an electrical-biasing holder, illustrates a few of the practical hurdles that must be surmounted while pursuing microscopy innovation. First the researcher acquires a lead on who "owns" the electrical biasing holder and proceeds to contact the tool owner. Next, the researcher is told that in order to use this holder, the specimen must be prepared using a FIB followed by a wire-bonding step. The researcher inquires with the tool owners for tool access. The researcher also identifies the microscope owner for the microscope that he/she wishes to use for the experiment. The path from idea inception to data collection goes through many tool owners and training steps, often outside of one's division and operational unit (OU), before any measurements are even made. There are similar parallels that can be drawn for other experiments involving, for example, liquid cell and microtome/cryo measurements. Furthermore, since tool owners usually serve first as project leaders, often with other demands on their attention, they may not be in a good position to provide technical expertise to the researcher seeking assistance. Many projects operate without a resident microscopist. These projects depend on collaboration with microscopists, or, the project leader may even determine that the benefits are not worth the hassle.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

Both the MML strategic plan and the participants in this workshop have identified a common set of strategic measurement needs that could or should involve a significant EM component. These strategic directions involve measurements that are highly-complex, highly-choreographed affairs that are becoming increasingly difficult to fulfill within a single division or by a couple of microscopists. It is the organizers' collective view that our EM resources can be better managed to streamline the operational process for providing support for our non-EM scientific staff, while concurrently providing continued high-level support as well as high-value EM measurement science. Below, we outline several ideas that might improve how EM is performed within MML, largely meant to be cost neutral by leveraging existing MML capabilities.

## 1.6. What needs to happen? – Our recommendations[☼]

### 1.6.1. *EM portfolio assessment*

While the instruments residing at each division are, for the most part, meeting the host division's needs, the discussion from the previous section leads us to conclude that these resources are not globally optimized from the standpoint of maximizing staff usage, project participation, and instrument lifetime ownership cost. Within MML, two small facilities (Nexus, PIF) are in operation, supporting a substantial user base. Maintenance and improvements to the structure and operations to these facilities occur on an *ad hoc* basis and are also far from optimized. Even as our historically program or project-level, organically-derived EM portfolio might seem *ad hoc*, it also means there are many opportunities to leverage this diversity to solve problems.

Therefore, we recommend that the management and the microscopists of MML conduct an analysis of our microscope and sample preparation equipment portfolio to evaluate (1) barriers to access for MML researchers (with or without EM background) in terms of equipment availability and the availability of staff assistance, (2) whether we have the correct balance of shared versus limited-access instruments, and (3) whether some instruments should be retired or repurposed. Removing barriers to access will increase instrument use, rebalancing the number of shared use and limited-use instruments may improve the overall operation efficiency, and sun-setting instruments will reduce service contract liabilities. Implicit to this recommendation is that any portfolio rebalancing is accompanied by a cost-benefit evaluation on the division level and across MML. This initial step can potentially maximize the use of existing resources with little additional cost.

Division 642 had undergone a mini-version of this exercise two years ago for the Microscopy Nexus. There, the small ratio between knowledgeable microscopy staff and non-EM division researchers was a substantial access barrier. Also, instruments slated for retirements are repurposed for use as idea incubation instruments. Proposals to

[☼] Unless otherwise specified, the views of the "Recommendations" section are those of the meeting organizers.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

develop incubation instruments are accepted from all of NIST. The concept of an incubation space is already familiar to both Boulder and Gaithersburg EM groups.
To be upfront, we are not arguing for total consolidation of the MML EM portfolio for the sake of efficiency. A User Center approach to EM (like Department of Energy's EM facilities) is on one end of the solution spectrum; single-PI instruments are situated on the other end. We are respectful of both solution concepts, particularly the nimble and truly unique instruments with single-PI stewards. However, some blend of solution must work better than others for lowering the access and dialog barriers in this community.
Portfolio reallocation alone is not going to help us solve great problems – particularly when staff views maintaining their own projects and supporting the needs of their colleagues as a conflict. Additional prescriptions are needed.

### 1.6.2. *Recalibrating our internal compass*

As is the case in many organizations, NIST and MML can at times gravitate towards important and pressing problems *du jour.* While this is necessary on occasion, it can lead to particularly poor outcomes for EM because these instruments are an order of magnitude costlier than the typical bench instruments, and because often high-quality EM solutions will require significant experimental design, considerable operation time, and extensive data analysis. For this reason alone, this discipline has significant inertia that requires greater organizational commitment, in terms of both project lifecycle and staffing. Serious EM-based measurement solutions do not usually emerge in just 2 to 3 years with 1 to 2 full-time-employee effort, but require sustained commitment from both the EM scientists and their supporting management. Another part of the solution requires action by EM scientists and their management together, to strategically identify the most important/potentially impactful problem types that may be left unaddressed by more "basic" research efforts. Problem types may include:

- standards work: SRMs, best practices, possibly SRD (we need to determine the appropriate balance with more development-facing efforts, given the relatively small number of MML EM-experienced staff)
- technique development targeting near-term, customer-articulated problem spaces *e.g.*, solutions to specific, but somewhat limited-scope problems articulated by industry, such as those in this workshop
- technique development targeting medium- and long-term customer needs, via informed speculation, *e.g.*, National Strategic Computing Initiative (NSCI) and related forecasts where specific problems are not yet identified, but broader-scoped classes of problems may be easily seen
- EM measurement methodology, measurement hardware/software
- broader technique development as appropriate, *e.g.*, improvements to widely-known, ever-evolving measurement needs such as spatial and spectral resolution, contrast improvement, signal improvement, etc.

#### 1.6.3. *Improving the EM community experience*

A hub, whether it is an online community, or a physical location like the Ideas Lab, or periodic gathering of colleagues would be a tremendous resource for the MML microscopy community. Some proto-hubs already exist within MML. In the two Gaithersburg materials divisions, microscopists talk about, for example, safety, data needs, equipment wishes, and measurement challenges, both in person and online. These hub activities are a significant resource for self-help and support for our community. Beyond these small efforts, the MML EM community is not set up to foster conversations in standardization of protocols, methods, uncertainty, and data (data and metadata formats, data management), all of which are critical needs identified by the workshop. Strengthening basic community engagement and interaction can potentially enable these advances.

In the hub, we can think about lowering the barrier to equipment access by staffing shared resources with motivated people committed to helping MML scientists excel. The PIF and the Nexus, for example, are staffed by research scientists using a co-op-like model. The collaborative atmosphere is excellent in these cases, but the operational model is not scalable as-is. The Center for Nanoscale Science and Technology (CNST) nanofab fee-for-service model, by contrast is highly scalable; but it does little to promote inter-PI cohesion. With some tweaking, the NIST Center for Neutron Research instrument scientist model is closest in spirit to what might best serve the MML EM needs.

Related to strengthening community engagement, we lay out how MML microscopists go about initiating innovation today. Identifying grand challenges and knowing what can be done about them is perhaps the one characteristic that binds the workshop organizers; each of us has led innovative and high-risk microscopy projects. However, we have one recurring frustration: often, we have too many good ideas amongst ourselves and we end up inadvertently competing within NIST for small funding odds. Competition is healthy, but annual competition that usurps 4 to 9 months of a calendar year is disabling and exhausts all of us. During this workshop, several "grand challenges" have emerged. We could, as a community, decide that *in situ* temperature measurement (as an example), is the one goal most worthy of us to collectively pursue in the coming year.

Beyond MML, mechanisms to encourage other OU contributions must be explored. For example, CNST has an ongoing effort focused on building specialized specimen holders for EM applications. CNST should be invited to bring this piece to the table in exchange for access to our hub. Finally, half of our best minds are located at the other campus. All options for encouraging closer ties between the two sites must be considered. One idea is a biannual group travel: Boulder can host a Spring visit from Gaithersburg colleagues, some will give seminars, others might bring samples to be examined by microscopes unique to Boulder. Then Gaithersburg can reciprocate in the Fall. Another idea going forward could be the purposeful differentiation of EM capabilities and expertise in the two campuses. As an example, Boulder(Gaithersburg) could be the innovation center for SEM(TEM)-related techniques. One can also imagine a division of labor along other

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

paradigms like imaging/spectroscopy and *in situ*, *in operando*/high-speed, high-throughput. A well-considered differentiation of expertise should ideally encourage community cohesion and support cross-pollination of ideas across campuses. However, this line of thinking must be extensively debated to avoid unforeseen problems.

Once the notion of a broadly shared EM hub takes root, we can then sensibly think about new instrument purchases as a community decision. As such, we recommend that MML provide the mechanisms to enable MML-level equipment procurement for microscopes, distinct from division equipment funds. With this funding model, we can simultaneously support a larger pool of researchers with higher-end microscopes. Expected savings from scaling up the EM economy is also expected, as instrument service contracts, utility services, safety planning, software licenses, and peripheral hardware may be budgeted and negotiated together.

#### 1.6.4. *Innovative ideas within reach*

During the workshop, we heard repeatedly from the speakers about the need for *in situ*, *in operando*, dynamic, high-throughput, temperature, and 3D. We can be more deliberate about fulfilling these needs by developing a community-based prioritized purchase plan for specialized specimen holder platforms such as heating, cooling, fluid flow, electromagnetic and optical stimuli. We can inventory the specialized holders we already have in order to make them more accessible to the EM community (Nexus has recently done this). Or, we can formalize a partnership with CNST to develop unique specimen holders. Within NIST, there is one environmental TEM and a number of environmental SEMs. There is still plenty of room on the existing instruments for *in situ* and *in operando* research. Particularly with our e-SEMs, we can modify one to do something highly unconventional, like direct metal laser sintering.

Another identified area is coping with the ever-increasing amount of data being generated by modern EM instrumentation, and the need for those data sets to bridge vast length and timescales. The recent emergence of terabyte-scale data sets is driven by the simultaneous maturity of several source technologies: high-throughput measurements (through improved stitching hardware and software, multi-beam instruments), tomography, hyperspectral and multimodal imaging, diffraction, and spectroscopies, *in situ* and *in operando* environments which then drive detection schemes capable of parsing time. Here, we see a path forward for data maintenance and integration. First, for non-proprietary and non-clearance-required research, MML must bring the microscopy data infrastructure to a common standard capable of integrating data ecosystems (*i.e.*, across campuses, divisions, and PIs). Ways to do this may include a common microscopy data curation platform and file servers. Modernizing the data framework for EM is an important first step to enabling new technology paradigms such as machine-learning and deep-learning for materials science.

Two important advantages for MML EM stood out: First, was our willingness to modify expensive and complicated instruments. This differentiates us from most other EM institutions, and it allows us to produce truly innovative measurement solutions. The other important advantage is the magnitude of the MML EM portfolio, the diversity of

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

materials examined, and in-house expertise. With proper coordination, unique collaboration modes can be enabled that would be difficult to support in conventional academic environments. Improving the data ecosystem is one such example.

## 1.7. Impact aspirations[☼]

By 2025, we aspire to be *the* go-to organization for US industry for developing application-driven electron microscopy solutions. "Industry" shall include developers of materials, users of materials, developers of instruments that measure structure and chemistry of materials. Not only could they come to us, but they SHOULD come to us for electron microscopy solutions to specific materials problems, and we want those industry representatives to know this.

NIST has always stood as the guardian of scientific integrity. While we have real institutional limitations that affects our competitiveness, there is also much institutional strength that we can better leverage. We have the capacity to be our industry's EM idea clearinghouse, and the destination for launching careers in application-driven electron microscopy.

[☼] Unless otherwise specified, the views of the "Recommendations" section are those of the meeting organizers.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

# 2 MEETING AGENDA

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

Wednesday March 8, 2017

| | Keynote Session |
|---|---|
| 09:00 | **NIST and MML Metrology Overview**<br><br>Mike Fasolka<br>Material Measurement Laboratory, Acting Director<br>National Institute of Standards and Technology |
| 09:35 | **Confessions of a former microscopist: What *really* drives progress in science and technology**<br><br>Alexander H. King<br>Critical Materials Institute, Director<br>The Ames Laboratory |
| 10:25 | **Coffee break** |
| 10:40 | **Atomic-Scale Analytical Tomography: Taking the Best from (S)TEM and Atom Probe**<br><br>Tom Kelly<br>Vice President for Innovation and New Technologies<br>CAMECA |
| 11:30 | **Moderated discussion** |
| 12:00 | **Lunch** |
| | Session I: Nanoelectronics and High-Performance Computing |
| 13:00 | **Transmission Electron Microscopy for Nanoelectronics**<br><br>Alain Diebold<br>Interim Dean - College of Nanoscale Sciences<br>SUNY Polytechnic Institute |
| 13:40 | **Transmission Electron Microscopy for Microelectronics Reliability**<br><br>Brendan Foran<br>Senior Scientist<br>The Aerospace Corporation |
| 14:20 | **Coffee break + Posters** |
| 14:50 | **Improving the Resolution, Acquisition Time and Quality of Scanning Microscopy Images Through Computational Rather Than Hardware Means**<br><br>Eric Lifshin<br>Professor of Nanoscience<br>SUNY Polytechnic Institute |
| 15:30 | **Moderated discussion** |
| 16:30 | **Day 1 adjourn** |
| 17:30 | **Happy hour** |

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

Thursday - March 9, 2017

| | |
|---|---|
| | **Session II: Soft Matter Validation** |
| 09:00 | **Validation of CryoEM Atomic Resolution Structure**<br><br>Wah Chiu<br>Distinguished Service Professor, Department of Biochemistry and Molecular Biology<br>Baylor College of Medicine |
| 09:40 | **Individual-Particle Electron Tomography (IPET): an approach to study flexible soft-/bio-molecular structure and dynamics**<br><br>Gary Ren<br>Molecular Foundry, Staff Scientist<br>Lawrence Berkley National Laboratory |
| 10:20 | **Coffee break** |
| 10:35 | **Morphology Characterization and Directing Self-Assembly in Nanostructured Soft Materials**<br><br>Chinedum Osuji<br>Professor of Chemical & Environmental Engineering<br>Yale University |
| 11:15 | **Moderated discussion** |
| 12:15 | **Lunch** |
| | **Session III: Additive Manufacturing** |
| 13:30 | **Electron-microscopy studies of additively manufactured 17-4PH stainless steel**<br><br>Rainer Hebert<br>Additive Manufacturing Innovation Center, Director<br>University of Connecticut |
| 14:10 | **The role of microscopy on ICME Methods development during the DARPA OM Program**<br><br>Alonso Peralta<br>M&PE Life Prediction Group, Staff Engineer<br>Honeywell Aerospace |
| 14:50 | **Coffee break + Posters** |
| 15:20 | **Powder-Bed Additive Manufacturing Modeling and Measurement Challenges**<br><br>Ade Makinde<br>Manufacturing & Materials Technologies, Principal Engineer<br>GE Global Research |
| 16:00 | **Moderated discussion and workshop conclusion** |
| 17:00 | **Meeting Adjourn** |

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

# 3 FINDINGS FROM THE KEYNOTE SESSION

### 3.1. Who came to speak?

For the Keynote session, we chose two eminent scientists with a strong background in electron microscopy. These scientists were chosen because they have a long history of performing EM-focused research, but have both since expanded outside of the field and so can offer a first-hand perspective on the *context and value* of EM research within a larger materials science and engineering portfolio. Together they have very broad and varied experience (university, industry, and national laboratory), and could speak to past, present, and future trends both within EM and in related fields.

The first speaker was Dr. Alexander King, Director of the Critical Materials Institute at Ames Laboratory -- one of DoE's four Energy Innovation Hubs. He concurrently holds the Bergdahl Professorship of Materials Science & Engineering at Iowa State University, and is also the former Director of the Ames Laboratory. Dr. King holds degrees from Sheffield University and Oxford University, worked as a post-doc at the Massachusetts Institute of Technology, and has held leadership positions at Stony Brook and Purdue Universities. He is a Fellow of the Institute of Materials Minerals and Mining; ASM International; and the Materials Research Society. Dr. King has also been the President of the Materials Research Society; Chair of the University Materials Council; and Chair of the American Physical Society's Group on Energy Research and Applications.

The second Keynote speaker was Dr. Thomas Kelly, Vice President for Innovation and New Technologies at CAMECA Scientific Instruments. Thomas F. Kelly received his Ph.D. in Materials Science in December 1981 from MIT. He was on the faculty at the University of Wisconsin-Madison from January 1983 until September 2001. Tom was also Director of the Materials Science Center from 1992 to 1999. Tom founded Imago Scientific Instruments in 1999 to commercialize his invention, the Local Electrode Atom Probe, or LEAP. He has published over 250 papers and 16 patents in these fields in that time. Tom was President of the International Field Emission Society from 2006 to 2008 and was President of the Microanalysis Society from August 2014 to August 2016. He is a fellow of the Microscopy Society of America and the International Field Emission Society.

### 3.2. Speakers Charge

The keynote session was intended for foundational leaders in the field of electron microscopy and materials science to provide their perspective on recent technological trends and drivers, based on the body of their considerable lifetime contributions to the field. We charged the Keynote speakers to comment on the broad context of how advances in electron microscopy have been made historically versus how they believe they are being made now and will be in the future. We asked them to comment on how advances in EM enable advances in materials science and engineering and other related fields. Finally, we asked them to comment specifically the strengths of NIST, and where they thought NIST would be able to make the greatest impact going forward in areas of critical national importance.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

### 3.3. When tools are available, science discovery is accelerated

A central theme that emerged in both Keynote addresses was the nature of the causal relationship in materials science discovery. One example given was the critical role of EM in the ability to make materials stronger on a fundamental level through the understanding of the role of dislocations. The story is that the existence of dislocations was theorized for many years but only after they were convincingly imaged for the first time using EM was the entirety of dislocation theory validated. This lead directly to dislocation theory becoming a central theme in all of materials science and engineering. This abundance of research into dislocations drove further EM instrumentation and technique development. This fully developed dislocation theory and the ability to image them lead directly to the understanding of how to make materials stronger.

"When the tools are available, the science is accelerated." A second example along these lines, although not related directly to EM, was given to emphasize the adage that "Technology doesn't need better microscopes, science does. But the gap is shrinking, and one necessarily follows the other." When the ENIAC computer was being built by the U.S. Army, some people questioned the necessity of such a machine, saying essentially that there were no problems complex enough to require such computing power! The lesson learned is that (1) you have no idea what may be coming down the road. (2) If you build it, there will be a need.

Another comment was given on the popularity of graphene. One of the speakers posited that the interest in graphene probably wouldn't be so high if we were not able to use EM to image it with atomic resolution. And this ability to image with atomic resolution was driven by a completely different technological need.

In summary, both speakers emphasized the critical importance of developing advanced instruments for the acceleration of game changing discovery materials science and engineering research. "When we get better microscopes, we tend to discover things that we did not know we were looking for."

### 3.4. NIST role

In the opinion of the Keynote speakers, NIST cannot respond fast (for example due to long procurement cycles). Our key strengths are in helping to set standards, develop standard data formats, standard experimental protocols, helping to get statistically relevant data sets and/or conclusions from the data, and provide a better understanding of the need for statistics and uncertainty.
That said, both speakers acknowledged that there are several critical areas that need significant research investment to fill the "gaps" and give EM the ability to radically alter materials science and engineering research. They felt that due to the strong history of technique development, NIST can play a critical role. These include:

- Developing *in situ* techniques
- Developing low-cost microscopes

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

- Developing high-throughput microscopes

- The ability to accurately measure temperature at the actual sample location
- The ability to detect and map light elements in 3D

- Big data
- Providing better length scale integration
- Providing surface and interface information

### 3.5. Summary

In summary, the Keynote addresses emphasized the critical need for and enormous potential reward that comes from investing in new EM technologies, techniques, and hardware, not necessarily to address a *current* technological or industry need, but instead to be positioned and ready when the next as yet unforeseen need arises. It used to be 20 years from material discovery to production and now it can be as rapid as less than one year, as for the example of the aluminum alloy developed for a highly-anticipated smart phone. Electron microscopes are a truly unique tool for materials discovery, particularly for the present-day economy because so much of the advances depends on structures on the nanoscale. With this pace of materials innovation, to NOT invest in EM would be akin to not getting in line.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

# 4 FINDINGS FROM THE HIGH-PERFORMANCE COMPUTING SESSION

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

### 4.1. Why we chose Nanoelectronics and High-Performance Computing as a topic

NIST/MML have a long history of positive impacts supporting the semiconductor industry, touching virtually all measurement aspects of integrated circuit, computer processor, and memory chip manufacturing. Recent high-level planning activities have addressed the strategic importance of positioning NIST/MML for success in supporting future computing technologies that encompass both today's state-of-the-art as well as proposed computing paradigms targeting the 15+ year timeframe.

In terms of worldwide sales, the semiconductor industry showed revenues of $338B in 2016, with anticipated revenues approaching $387B in 2018 [1]. The historically rapid pace of growth in this industry, described empirically by Moore's Law, has seen a coupling of technology advances with increases in economic efficiency, both in large part due to improvements in the ability to manufacture and integrate materials.

Computing developments targeting the near-term (< 5 years) continue the hardware paradigm of electron-based power and information transport – *i.e.*, nanoelectronics. The basic components and devices forming the foundation of the industry require the integration of numerous disparate materials, each with characteristic processing requirements and resulting properties. A key driver for industry success is continued dimensional scaling, presently at the level of 5 nm feature sizes, which introduces further-yet demands on materials. At such scales, even minor changes in individual atom positions can dramatically affect performance and reliability. Methods such as electron microscopy, which can measure these aspects of materials, already play a critical role in virtually all phases of device and component manufacture. Any future emerging new paradigm for high-performance computing will either mimic aspects of electronics-based computing, or require the integration of completely new combinations of materials – in either case, electron microscopy will need to keep up to provide the high spatial resolution material measurements that tell us the "what and where" of the atoms making up future devices.

### 4.2. Present NIST footprint

NIST activities in the broad area of electronics peaked in the mid-2000s, and underwent a downturn since the dissolution of the NIST Office of Microelectronics Programs shortly after 2011. The advent of discussions in 2015 associated with a pending National Strategic Computing Initiative has revived more active planning in this realm. In terms of active projects in nanoelectronics and high-performance computing, there are too many to provide a comprehensive list. In terms of electron microscopy development as applied to nanoelectronics and high-performance computing, the following represent non-MML projects, as identified by searches on [http://www.nist.gov](http://www.nist.gov) for "electronics" and for "electron microscopy," with investigator names in parentheses. For the latter, we read the descriptions, and made the

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

determination whether the effort addressed electronics. Results are limited to active projects within NIST, and exclude publication records older than 10 years.

- Nanomagnetic Imaging (~ SEM with polarization analysis, or SEMPA), CNST (John Unguris)
- Three-Dimensional Nanometer Metrology, PML (Andras Vladar)

Following are MML projects with measurement development ties to nanoelectronics and high-performance computing:

- Microscopy Methods (John Bonevich)
- Analytical Transmission Scanning Electron Microscopy (Jason Holm)
- Strain Mapping and Simulation (Mark Vaudin)

### 4.3. Who came to speak?

For this session, we invited three speakers representing both microscopy technique experts as well as industry leaders who use electron microscopy methods in nanoelectronics and high-performance computing. The first speaker was Alain Diebold, who is Dean of the College of Nanoscale Sciences at SUNY Polytechnic Institute. He initiated and chaired the International Technology Roadmap for Semiconductors for many years. The second speaker was Brendan Foran, who is a Senior Scientist at The Aerospace Corporation, and led the electron microscopy team in process characterization for SEMATECH. The final speaker was Eric Lifshin, who is a Professor at SUNY Polytechnic, and managed the Characterization and Environmental Technology Lab at General Electric (GE). Eric co-authored the major textbook in the field of Scanning Electron Microscopy and X-ray Microanalysis.

### 4.4. Where some consensus emerged:

The use of electron microscopy in nanoelectronics and high-performance computing targets ultimately the manufacture of highly-complex nanoscale devices that exhibit high performance and low cost. Electron microscopy is used for both quality control and for failure analysis. Primary drivers for EM use include measurements of dimensions, composition, stress-strain response, interface structure, and reliability, to improve processes and device designs.

The requirements and challenges identified can be classified into three categories: instrument operation, instrument capabilities, and standards/methodology. The following common themes emerged in terms of more specific technological needs for this industry:

- <u>high throughput</u> – the ability to measure many samples in a short time, preferably in an automated fashion in the quality control context; targets include 15,000 images/month/tool and 5-minute elemental maps by energy dispersive spectroscopy;

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

- fast, low-cost, easy microscope operation – rapid solutions are of prime importance to this industry, to minimize downtime for fabrication lines;
- controlling environmental influence – measurements can be adversely affected by factors such as specimen/stage drift, acoustic/radio-frequency/ electromagnetic noise, all of which must be controlled;
- 3-D measurements – the ability to measure composition, electronic structure, and device dimensions/geometries over multiple length scales, perhaps reached via a hybrid tool, multi-beam tool, or some form of correlative tomography;
- *in operando* measurements – ensuring device functionality is the goal, and observing dynamic processes associated with degradation and failure is key, and involves effects of temperature, voltage, and radiation;
- standards/methodology – a wide variety of standards are desired:
    - definitions of resolutions (spatial, chemical/compositional), their limits, and RMs to measure them;
    - beam-sample interactions that may perturb measurements – interaction volumes, ion/knock-on damage, thermal diffusion, ionization damage;
    - optimization schemes for proper EM measurement method/detection mode – different imaging modes give different information about a single event or feature;
    - sample preparation effects – amorphization, implantation, stress relaxation, altering metastable materials, contamination.

These considerations are expected to continue to exist, no matter what dominant paradigms emerge for future computing, since all present and any future computing approaches necessarily require integrated multi-material systems with multi-faceted material demands that extend beyond the design of pure computing functionality.

### 4.5. Where can MML microscopists make an impact?

MML is scientifically and technologically capable of impacting all the needs indicated above to varying extents. We first bin the above needs into categories that describe how well-aligned they are with existing MML capabilities. We must also consider our targeted balance between peer-reviewed scientific research and direct user-technology transfer (which may or may not be appropriate for publication in the literature).

Table 3 depicts alignment with existing MML capabilities, items in blue representing possible IMS (Innovation in Measurement Science) funding opportunities:

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

**Table 3: Nanoelectronics/Computing Alignment with MML Capabilities**

| Well-Aligned (important and requiring minor re-tooling) | Not Well-Aligned (important but may need major re-tooling) | Not Appropriate |
|---|---|---|
| - *In operando* measurements<br>- Beam-sample interactions/ perturbations<br>- Resolution definitions, limits, and RMs | - Correlative approaches<br>- Managing sample preparation artifacts<br>- 3-D imaging, composition, electronic structure/ tomography<br>- Higher throughput/ faster operation | Optimization algorithms for EM approaches/ detection |

The following table presents the need in the context of efforts that would be more scientific-oriented versus direct user-tech transfer oriented.

**Table 4: Nanoelectronics/Computing Needs: Scientific vs. Direct Tech Transfer**

| Scientific (results in peer-reviewed publications) | Direct Tech Transfer (less likely to result in peer-reviewed publication) |
|---|---|
| - *In operando* measurements<br>- Beam-sample interactions/perturbations<br>- Resolution definitions, limits, and reference materials<br>- Correlative approaches<br>- 3-D imaging, composition, electronic structure/tomography | - Managing sample preparation artifacts<br>- Higher throughput/ faster operation |

## 4.6. Summary

Nanoelectronics/high-performance computing, like the other session topics, presents a broad set of measurement challenges. Industry experts have articulated detailed needs, many of which can be identified as spanning all technical sessions. Before committing to specific research plans, we must decide on a desired portfolio balance, covering both scientific (peer-review quality) efforts and more direct technology transfer efforts that can immediately benefit the user communities.

# 5 FINDINGS FROM THE BIOLOGICAL AND SOFT MATTER VALIDATION SESSION

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

## 5.1. Why we chose Biological and Soft Matter Validation as a topic?

The characterization of biological and soft matter using electron microscopy techniques presents unique problems for the analyst. Most importantly, the electron energies typically employed to simultaneously achieve high spatial resolution and electron transmission can cause alteration of the atomic structure being probed or even total specimen destruction. In addition, the sample preparation protocols typically employed for these materials differ markedly from those developed for solid-state materials. Because of these factors, EM analysis of biological and soft materials has virtually existed as an independent field. Recently, technological advances enabling automated sample preparation and data collection and the advent of new detectors has revitalized the field. This has been most widely highlighted for biological characterization, where cryogenic EM was recently named the Method of the Year by Nature (doi:10.1038/nmeth.3730). The number of biological structures solved each year is dominated by those determined via X-ray crystallography, but cryo-EM is rapidly advancing and allows for structural determination in systems not accessible via X-ray or neutron-based methods. While less widely discussed, this resurgence has also extended to non-biological soft materials, where novel hardware and methods are poised to enable new understandings of the interplay between structure, property, and processing.

## 5.2. Who came to speak?

For this session, we invited three leading academic researchers working in biological and soft matter characterization to discuss their work and the way in which advancing EM technology could aid them in overcoming measurement challenges. The first was Prof. Wah Chu, who is the Alvin Romansky Professor of Biochemistry and the Distinguished Service Professor at Baylor College of Medicine in Houston, Texas. He is a pioneer in methodology development for electron cryo-microscopy whose work has made multiple transformational contributions in developing single particle electron cryo-microscopy as a tool for the structural determination of molecules at atomic resolution. The second speaker was Dr. Gang (Gary) Ren, a career staff scientist and group leader at Molecular Foundry, Lawrence Berkeley National Laboratory. His research group at LBNL is focused on the development of electron tomography techniques to study soft-materials and protein structure and dynamics. Finally, the final speaker of the session was Prof. Chinedum Osuji Associate Professor in the Department of Chemical and Environmental Engineering at Yale University. He leads an experimental research group focused on structure and dynamics of soft matter and complex fluids with specific interest in structure-property relationships in ordered soft materials, directed self-assembly of block copolymers, and rheology and slow dynamics of disordered systems.

## 5.3. Issues highlighted during the Biological and Soft Matter Validation Session

There was broad agreement in this session that NIST must get involved in the rapidly advancing field of CryoEM. While the field has advanced at an impressive rate in recent years, the techniques employed are entirely devoid of standardization. All aspects of the work, including sample preparation, data collection and processing, and structural modeling vary not only from lab to lab, but from analyst to analyst. NIST is ideally suited to addressing these challenges since they fall well within its mission space and the speakers stated it was difficult to obtain funding for such efforts with the field more-focused on achieving greater resolution.

In addition, the process of sample preparation was singled out as the rate limiting step in the cryo-EM work flow. In order to increase throughput, and thereby statistical rigor in the measurements, developing optimized, repeatable sample preparation protocols would be a great improvement.

For the non-biological soft matter applications discussed in this session, we heard that the tools required for advanced measurements already exist. However, significant opportunities were identified as to how to best use the existing technologies. In other words, while the required measurements would not be pushing the envelope of existing technologies in terms of spatial or temporal resolution, standard methods for measuring various phenomena and features with quantifiable uncertainties were very much in need. The case for *in situ* measurements of phase development and crystallization were specifically highlighted in this context.

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

# 6 FINDINGS FROM THE METAL ADDITIVE MANUFACTURING SESSION

## 6.1. Why we chose metal additive manufacturing as a topic?

Electron microscopes have had a long history of development optimized for non-beam sensitive materials like metals. Therefore, for this workshop, we limited our scope to metal additive manufacturing (AM) where we have a greater chance in developing effective solutions. If successful, we can then apply the lessons learned with metals to polymer AM materials that can be tougher to image.

In metal AM, geometrically complex 3D objects may be produced layer-by-layer, using a computer to guide a rapid laser-melting and solidification trajectory of alloy feedstock materials. Once regarded only as a prototyping technique, geometrically complex parts, such as the fuel nozzles for GE's LEAP engine, are now being produced additively. A NIST economic analysis report [2] indicated that revenues for AM equipment and services was worth $4B in 2014, and is estimated to be between $5B and $6B, globally, by 2016 [3]. However, AM is far from a mature manufacturing method; many of the fundamental aspects of the process are still not understood.

"*Developing the material measurement infrastructure that will enable additive approaches to production to prosper and revitalize manufacturing in the U.S.*" is a specific strategy named in the most recent MML strategic plan (https://mmlstrategy.nist.gov/) for the area of materials science [4]. Indeed, given the substantial size of the footprint and early investment by U.S. firms (GE, Honeywell, United Technologies), AM's potential for rapid growth, and the subsequent implications for the U.S. economy, we had a compelling reason to have a closer look at the metrology issues involving this technology.

## 6.2. Present NIST footprint

The NIST portfolio on metal AM is distributed primarily across two different OUs: EL and MML. Partnership across MML and EL on this topic has been prolific.
There are several active metal AM projects within EL. PI names are in parenthesis:

- Characterization of Additive Manufacturing Materials (Alkan Donmez)
- Qualification of Additive Manufacturing Materials, Processes, & Parts (Shawn Moylan)
- Real-Time Monitoring and Control of Additive Manufacturing Processes (Brandon Lane)
- Systems Integration for Additive Manufacturing (Paul Witherell)

Within MML, two projects span multiple divisions:

- Additive Manufacturing of Metals (Lyle Levine)
- Fatigue and Fracture of Additive Processed Metals (Nik Hrabe)

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

### 6.3. Who came to speak?

For this session, we invited three leaders of metal AM to speak about measurement challenges that they face. The first was Dr. Rainer Hebert, who is the Director of the Additive Manufacturing Innovation Center (in partnership with Pratt & Whitney) at the University of Connecticut. There, he leads projects that focus on basic material science aspects of metal AM. The second speaker was Dr. Alonso Peralta, a staff engineer at Honeywell Aerospace. For the last 5 years, he led several Honeywell AM programs, as well as the DARPA Open Manufacturing program. Finally, we heard Dr. Ade Makinde from GE Global Research speak about the challenges he faced in his AM program. Dr. Makinde is the principal engineer for manufacturing process modeling at GE with over 19 years of experience in nonlinear mechanics.

During one workshop organization session, the organizers came to recognize that while we are clever at inventing new functionalities and measurement techniques centered around electron microscopy, we could all benefit by changing how we view external problems and proposing ways in which EM can help. None of this session's speakers self-identified as an electron microscopist. In our view, this provided a valuable change in perception.

### 6.4. Where everyone agreed: melt pool, melt pool, melt pool

The modern metallurgical framework which is foundational in predicting performance cannot be fully utilized for AM components without a thorough understanding of the melt pool physics and dynamics. The one central message that we heard from all three speakers is that **our limited understanding of the liquid metal pool – produced during laser melting, is the primary handicap which limits the growth of this industry.** Specifically, there is urgent need for time-resolved *in situ* characterization of the melt pool to measure:

- How the feed powder and the surrounding areas melt
- The melt pool shape
- Phases that form during solidification and the order of their appearance
- Residual stresses
- Temperature
- How the pool solidifies, which includes questions like:
    - Viscosity of the melt (as a function of temperature)
    - Thermal conductivity
    - Surface tension
    - Heat transfer mechanism and their order of importance (Convection? Conduction? Sublimation? Radiation?)
- Debris products: Where do they go and what do they do?

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

In addition, if some of the above *in situ* measurements can be executed in 3D, critically important information such melt pool geometry, and re-melt and re-nucleation events can be revealed. However, a violent plume of ejected metal particles, plasma and gases is always generated at the melt site. Measuring anything in the presence of the plume is exceedingly difficult; this a challenge that is commonly recognized.

Unknowns regarding the composition and size distribution of the virgin and re-solidified powder feedstock, along with temporal and spatial profiles of the laser, are also recognized as significant challenges to standardizing operation conditions. This lack of standards for laser and feedstock material is a part of a broader problem; while sophisticated computational toolkits exist for AM, what is lacking is NIST-traceable standard reference data as input for these models. Finally, this industry needs better ways to seamlessly integrate measured data across vast length scales; e.g., determining mechanical properties of a single phase (nm) that may have evolved from non-equilibrium conditions in the nanoscale, to how these properties change across phase boundaries (μm), and how this translates into macroscopically measured properties for the built object (m). Yet smooth and meaningful data integration among different measurements across these different length scales remains a challenge.

### 6.5. Where can MML microscopists make an impact?

For metal AM, this committee did not identify any investment bargains with a short-term payoff. There were several challenges such as stock powder uniformity and laser characteristics that should be addressed by different means. However, with modest re-tooling, we saw several opportunities for electron microscopists to make ground-breaking contributions.

We heard again and again, that there is an *in situ* and *in operando* measurement gap for a large variety of non-equilibrium phenomena (conductivity, temperature, stresses, etc.) using probes that are at or below the typical melt pool dimensions. Additionally, these measurements, which we cannot yet do, must ideally be done in 3D. The opportunity here is to provide the metal AM industry with NIST-quality experimental data as the foundation for its computational models. The lack of experimental data is the limiting factor in material property prediction and process optimization, which is a major bottleneck for this industry.

Microscopists within MML have a strong record of performing both 3D and *in situ* experiments. We have a significant inventory of heating, cryo, electrical, environment cell, and tomographic specimen holders. We have also begun exploring and building capability for dynamic high-speed measurements, for example, the in-TEM-column ultrahigh-speed AFM and the GHz stroboscopic TEM. Management invested in these capabilities because MML had also identified "Dynamic Measurements for Materials Manufacturing" as a key strategy in its strategic plan. It is therefore not a coincidence

that the other topics at this workshop have also identified the same needs; 3D, *in situ* and *in operando* is a part of the global nanoscience trend.

The biggest obstacle towards true *in situ* and *in operando* measurements during the build process is the concurrent eruption of a violent plume consisting of gases, liquid metal, and plasma caused by melting with a high-power laser. Instead of treating this plume as a kind of nuisance, it should be regarded as a treasure trove in that the material's composition, melt pool geometry and temperature, and laser characteristics are all encoded within. How can a mono-energetic beam of focused electrons be used to decode the plume? Answering this question alone can potentially produce new innovations in the measurement sciences. While the list of measurement challenges is extensive, electron microscopy is uniquely suited to addressing some of these challenges.

Finally, for standard data, some of our colleagues are already working on experiments that provide key reference data that can be applied to computational models with a high degree of confidence. As just one example, in FY17, DOE began funding NIST to provide reference and validation measurement data in support of the world's most ambitious AM simulation effort, the joint LLNL/LANL/ORNL/NIST project, "Transforming Additive Manufacturing through Exascale Simulation (ExaAM)." In another example, the NIST Thermodynamics Research Center (Boulder) recently launched a data product compiling curated thermo-physical data for metals and binary alloys for the modeling community.

For data integration, this industry needs better ways to seamlessly integrate measured data across vast length scales: e.g., from determining mechanical properties of a single phase that may have evolved from non-equilibrium conditions in the nanoscale, to how these properties change across phase boundaries in the mesoscale, and how this translates into macroscopically measured properties for the built object. With the nascent efforts within MML to redesigning microscopy data management to align with the FAIR data principles [5], we are already one step closer to multiscale data integration. Once a robust microscopy data management platform is in place, collaboration and co-investment with the machine learning community within NIST could be a logical next step. With the right series of investments, the author imagines that a Google Earth-like solution for the data length scale problem is one day possible.

### 6.6. Summary

Metal AM has a rich set of problems that the NIST electron microscopy community should find both intellectually stimulating and a great fit for the NIST mission. Many elements necessary for making a meaningful impact are already in house. An intentional coalescence of these efforts could go a long way in helping this important industry. All the organizers of this workshop have an established record of innovative thinking, which will be critical for tackling the "plume problem".

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

This publication is available free of charge from: https://doi.org/10.6028/NIST.SP.1217

## Acknowledgements

We conclude by thanking the individuals and sponsors who have made this workshop possible. We are thankful for the detailed notes captured by our scribes, Aaron Johnston-Peck, Michael Katz, and Andrea Szakal. We gratefully acknowledge the funding provided by divisions 642, 643, 647, and the MML office for this activity. We are also appreciative of the arrangements provided by the generous support from ThermoFisher, Direct Electron, and Zeiss. These provisions have substantially enhanced the quality of interaction and engagement of this workshop. Finally, we thank the speakers and participants for their insights and discussions that have made this engaging workshop possible.

## References

[1] Semiconductor Industry Association Industry Statistics, (accessed 6/13/17). https://www.semiconductors.org/industry_statistics/global_sales_report/

[2] Scott TJ, Beaulieu TJ, Rothrock GD, O'Connor, AC (2016) *Economic Analysis of Technology Infrastructure Needs for Advanced Manufacturing: Additive Manufacturing*, RTI International, Research Triangle Park, NC 27709, RTI Project Number 0214105

[3] Makinde A (2017) Opening statement to the presentation titled "Powder-Bed Additive Manufacturing"

[4] Materials Measurement Laboratory strategic plan. https://mmlstrategy.nist.gov/#materials-science

[5] The FAIR data principles. https://www.force11.org/group/fairgroup/fairprinciples