EERE Quality Control Workshop Final Report

Proceedings from the EERE Quality Control Workshop, in support of the DOE Clean Energy Manufacturing Initiative
Golden, Colorado
December 9–10, 2013

NREL is a national laboratory of the U.S. Department of Energy
Office of Energy Efficiency & Renewable Energy
Operated by the Alliance for Sustainable Energy, LLC

Workshop Proceedings
NREL/BK-5900-61889
May 2014

Contract No. DE-AC36-08GO28308

EERE Quality Control Workshop Final Report

Proceedings from the EERE Quality Control Workshop, in support of the DOE Clean Energy Manufacturing Initiative
Golden, Colorado
December 9–10, 2013

NREL Contact: Michael Ulsh

Prepared under Task No. BE4C.SC01

National Renewable Energy Laboratory
15013 Denver West Parkway
Golden, CO 80401
303-275-3000 • www.nrel.gov

Workshop Proceedings
NREL/BK-5900-61889
May 2014

Contract No. DE-AC36-08GO28308

NOTICE

This report was prepared as an account of work sponsored by an agency of the United States government. Neither the United States government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States government or any agency thereof.

This report is available at no cost from the National Renewable Energy Laboratory (NREL) at www.nrel.gov/publications.

Available electronically at http://www.osti.gov/scitech

Available for a processing fee to U.S. Department of Energy and its contractors, in paper, from:

U.S. Department of Energy
Office of Scientific and Technical Information
P.O. Box 62
Oak Ridge, TN 37831-0062
phone: 865.576.8401
fax: 865.576.5728
email: mailto:reports@adonis.osti.gov

Available for sale to the public, in paper, from:

U.S. Department of Commerce
National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
phone: 800.553.6847
fax: 703.605.6900
email: orders@ntis.fedworld.gov
online ordering: http://www.ntis.gov/help/ordermethods.aspx

Cover Photos: (left to right) photo by Pat Corkery, NREL 16416, photo from SunEdison, NREL 17423, photo by Pat Corkery, NREL 16560, photo by Dennis Schroeder, NREL 17613, photo by Dean Armstrong, NREL 17436, photo by Pat Corkery, NREL 17721.

Printed on paper containing at least 50% wastepaper, including 10% post consumer waste.

EERE Quality Control Workshop

Workshop held December 9–10, 2013
National Renewable Energy Laboratory, Energy Systems Integration Facility
15013 Denver West Parkway
Golden, CO 80401

Co-Sponsored By:
U.S. Department of Energy—Fuel Cell Technologies Office (FCTO), Solar Energy Technologies Office (SETO), Vehicle Technologies Office (VTO), Building Technologies Office (BTO), and Advanced Manufacturing Office (AMO)

Organized and Hosted By:
National Renewable Energy Laboratory (NREL)

Lead Organizers:
Nancy Garland, FCTO
Michael Ulsh, NREL

Organizing Committee:
Nancy Garland, FCTO
Cassidy Houchins, SRA
Devanand Shenoy, AMO
Joe Cresko, AMO
David Hardy, AMO
Peter Faguy, VTO
Antonio Bouza, BTO
Shubhra Bansal, SETO
Michael Bolen, SETO
Jesse Adams, FCTO
Michael Ulsh, NREL

Breakout Group Facilitators:
Michael Bolen, SETO
Nancy Garland, FCTO
Peter Faguy, VTO
Antonio Bouza, BTO
Michael Ulsh, NREL
Claus Daniel, Oak Ridge National Laboratory
Jesse Adams, FCTO

Breakout Group Scribes:
Kim Cierpik, CAS-Navarro Joint Venture, LLC
Cassidy Houchins, SRA
Nathan Moore, CAS-Navarro Joint Venture, LLC
Stephanie Hodge, CAS-Navarro Joint Venture, LLC
Shaun Onorato, CAS-Navarro Joint Venture, LLC

Executive Summary

The U.S. manufacturing sector accounts for about 12% of the nation's gross domestic product, 70% of domestic industrial research and development spending, and 86% of all U.S. export goods. It employs 60% of industry's scientists and engineers. Each of these jobs, on average, supports 2.5 jobs in other sectors and, for high-tech manufacturing jobs, up to 16 other jobs are supported. Similarly, every dollar of manufactured goods sales supports $1.35 in output from other sectors—the highest "multiplier" of any sector of the economy.

In response to this importance, and in the face of ever increasing global competition for U.S. manufacturers, the U.S. government has initiated a whole-of-government Advanced Manufacturing Initiative to enable manufacturing innovation, secure America's talent pipeline, and improve the business climate for manufacturing. Among the cross-cutting technologies seen as critical to enable manufacturing innovation is a family of disciplines including advanced sensing and measurement, process control and feedback, and information technology for manufacturing. The need for sensing, data gathering and analysis, and integration of manufacturing data for high quality, efficiency, and resource optimization is especially great for clean energy technologies, many of which are characterized by low market volumes and are being developed by small and medium sized enterprises often not having the corporate competencies to adequately address process scale-up and the associated quality inspection and control issues.

Within this backdrop, the U.S. Department of Energy (DOE) Office of Energy Efficiency and Renewable Energy (EERE) has recognized the cross-cutting, pre-competitive, and enabling nature of quality control (QC) for a wide range of clean energy technologies. As such, the Fuel Cell Technologies Office, Solar Energy Technologies Office, Vehicle Technologies Office, and Building Technologies Office, as well EERE's Advanced Manufacturing Office and Clean Energy Manufacturing Initiative, decided to explore needs and potential cross-office synergies in this area by holding a workshop. The purpose of the workshop was to convene government, industry, and other stakeholders to discuss the current status of quality control and metrology in manufacturing processes relevant to the EERE offices, note gaps in which current techniques are inadequate or missing altogether, discuss similarities in materials inspection and metrology needs across technologies, and identify opportunities for collaboration across EERE offices to address shared challenges.

The scope of the workshop was determined to be quality control at the part and/or sub-assembly level during manufacturing, specifically for materials in the form of sheets or characterized by two-dimensional surfaces. This topic was selected in part because many of the existing activities supported by these offices to address quality control relate to these kinds of materials. Because these materials are often produced using continuous processes (e.g., roll-to-roll or belt-fed processes), "in-line" inspection—or techniques that can be used directly on the manufacturing line while the material is being produced, and thus are typically rapid, non-contact, and non-destructive in nature—was of highest interest. The workshop was held at the National Renewable Energy Laboratory's Energy Systems Integration Facility in Golden, Colorado, on December 9 and 10, 2013.

The structure of the workshop was designed to (a) establish a common baseline, in terms of processes, materials, and quality control techniques and (b) facilitate open and collaborative discussion to identify potential synergies, remaining needs, and key recommendations. These two requirements were important to enable easy and fruitful interaction between the participants, who represented a broad range of technologies. The key information-gathering element of the workshop was a series of three breakout sessions. The first session was a review of quality control challenges across the four technology offices. The second was a discussion of potential synergies across offices and quality control techniques. And the third was a discussion of key quality control needs that are not currently being addressed, and final recommendations.

Forty participants from industry, labs, and DOE, representing a wide range of clean energy technologies, provided their thoughts and experiences about QC challenges and contributed their ideas and suggestions about potential synergistic activities and how to move forward collaboratively in addressing these challenges. The outputs of the workshop logically lent themselves to categorization into conclusions and recommendations, the former taking the form of specific technical topics to be addressed and the latter as proposed activities for EERE and industry to pursue.

Three broad technical topics were identified for further action.

1. Cross-cutting QC development needs, including:
    a. Thickness measurement
    b. Inspection for mechanical defects such as pinholes and cracks
    c. Measurement of electrical properties
    d. Measurement of surface texture, structure, and morphology
    e. Inspection for inter-layer delamination and voids
    f. Improving the sensitivity of sensors
    g. Advancing tools and methods for QC data collection, analysis, storage, and use.
2. Correlation of defects to cell or device performance and lifetime, including:
    a. Methods for differentiating between fatal defects and process drift
    b. Process control for defect reduction
    c. Defect marking, removal, and/or correction.
3. Development of analyses to define and communicate the costs and benefits of in-line QC as a function of material requirements and manufacturing processes, yields, volumes, and costs.

In addition, two key programmatic areas were identified to assist industry, labs, and DOE in moving forward with cross-office, collaborative efforts to address these technical areas.

1. Methods to improve communications and interactions with and technology transfer to industry, including:
    a. Holding webinars and posting case studies on the Web

    b. Connecting with other U.S. government entities that may have established best practices for interaction with industry on manufacturing topics
    c. Supporting continued inclusion of QC-related topics in funding opportunities and Small Business Innovation Research (SBIR) calls
    d. Developing and making available a catalog of QC techniques, their capabilities, applications, and suppliers/developers.
2. Facilitation of more detailed technical exchange between researchers, developers, and vendors across the different technology areas to better identify synergies, including:
    a. Convening technical meetings to enable detailed understanding of techniques, capabilities, equipment, and methods, possibly co-located with relevant conferences or DOE program reviews
    b. Developing improved methods to enable, fund, and reward cross-office collaborations between lab researchers within the Annual Operating Plan process.

Table of Contents

List of Figures

List of Tables

Introduction

Background and Motivation

American manufacturing, and the competitiveness of this sector of the U.S. economy, has been a topic of extensive and growing discussion in economic, technical, and political spheres over the past few years. Indeed, while the U.S. manufacturing sector has shed millions of jobs since the late 1970s [1], it remains a critical driver of the American economy. Manufacturing provides about 12% of the nation's gross domestic product. It accounts for 70% of domestic industrial research and development (R&D) spending and 86% of all U.S. export goods, and it employs 60% of industry's scientists and engineers [2, 3]. Furthermore, manufacturing jobs and economic output have a strong impact on the rest of the economy. On average, each manufacturing job supports 2.5 jobs in other sectors; for high-tech manufacturing jobs, up to 16 other jobs are supported. Similarly, every dollar of manufactured goods sales supports $1.35 in output from other sectors—the highest "multiplier" of any sector of the economy [1]. The strength of the American manufacturing sector is also critical to national security. According to the 2010 Quadrennial Defense Review [4], "In the mid to long term, it is imperative that we have a robust industrial base with sufficient manufacturing capability and capacity to preserve our technological edge and provide for the reset and recapitalization of our force."

With this importance in mind, several studies by the Executive Office of the President [1, 3, 5] have called for a national strategy for *advanced manufacturing* and the creation of a "whole-of-government" Advanced Manufacturing Initiative (AMI). The whole-of-government effort officially began on June 24, 2011, with the announcement by President Obama of the Advanced Manufacturing Partnership [6], a collaboration between industry and several government agencies including the U.S. Department of Energy (DOE), with a stated mission to "Identify opportunities for investments in R&D, pre-competitive collaboration, and shared facilities and infrastructure that have the potential to transform advanced manufacturing in the United States, and recommend collaborative approaches that will realize these opportunities."

President's Council of Advisors on Science and Technology definition of advanced manufacturing [3]:

"a family of activities that (a) depend on the use and coordination of information, automation, computation, software, sensing, and networking, and/or (b) make use of cutting edge materials and emerging capabilities enabled by the physical and biological sciences, for example nanotechnology, chemistry and biology. This involves both new ways to manufacture existing products, and especially the manufacture of new products emerging from new advanced technologies."

The AMI is to be framed by the three pillars of: (i) enabling manufacturing innovation, (ii) securing America's talent pipeline, and (iii) improving the business climate for manufacturing [1]. Within the first pillar, a strong emphasis was placed on cross-cutting technologies that are vital to advanced manufacturing. The need for cross-cutting investments was echoed by the National Science and Technology Council, which described efforts that are "beyond the purview of any one agency or private-sector entity, but are collectively viewed as critical to advancing key national interests" and "should be used to strengthen the industrial commons in a way that would benefit all the participating agencies (and their stakeholders)" [5].

The President's Council of Advisors on Science and Technology further defines a list of "Top Cross-Cutting Technologies" that spans a spectrum from process technologies to enabling technologies to sustainable business practices [1]. Several of the categories, including "Advanced Sensing, Measurement, and Process Control," "Visualization, Informatics, and Digital Manufacturing Technologies," "Sustainable Manufacturing," and "Advanced Manufacturing and Testing Equipment," highlight the criticality of capturing and utilizing data during the manufacture of advanced products. The group describes as a key national need "technologies and systems that enable optimal raw material, energy, and resource utilization," and states that "megatrends of energy and resource efficiency, better safety, and higher quality also depend highly on advances in sensing and automatic process control." In a similarly-focused study, the Massachusetts Institute of Technology (MIT) assessed the importance of production in our innovation economy [7, 8] by surveying large universities on the importance of a set of manufacturing-based technologies or technology needs. Highly rated categories included "Advanced Sensing," "Information Tech for Manufacturing," "Continuous Process Control," and "Advanced Metrology".

The need for sensing, data gathering and analysis, and integration of manufacturing data for high quality, efficiency, and resource optimization is especially great for clean energy technologies. As stated by DOE's Advanced Manufacturing Office (AMO) in the introduction to their Membrane Technology Workshop Report [9], "New membrane materials, developed at laboratory scale, offer significant benefits over currently available commercial membranes. However, manufacturing defects and high module costs at full production scale are preventing widespread adoption in applications where membranes hold the greatest promise to reduce energy consumption and manufacturing costs."

This assessment is indicative of many clean energy technologies and has motivated several DOE Office of Energy Efficiency and Renewable Energy (EERE) technology offices, including the Fuel Cell Technologies Office (FCTO), Solar Energy Technologies Office (SETO), and Building Technologies Office (BTO), to hold similar workshops wherein quality control (QC) and inspection needs are discussed concurrently with material and process development needs [10, 11, 12]. In the case of FCTO, these learnings for hydrogen and fuel cell technologies have been included in updates to multi-year program plans in the form of specific tasks and milestones [13]. Similarly, in their review of critical manufacturing R&D needs in three key technology areas—hydrogen technologies, nanomanufacturing, and intelligent and integrated manufacturing—the National Science and Technology Council points out the need for "ensuring near-zero defect standards in manufacturing." In specific examples, the group lists the need to develop in-line quality control methods concurrent with development of advanced processes for fuel cell membrane fabrication and catalyst layer deposition and instrumentation and metrology for nanomanufacturing processes, where thin-film and nanoscale production—relevant to many clean energy technologies such as photovoltaics (PV)—will present particular challenges [14]. Another reason this area of quality inspection is important for clean energy technologies, as discussed generally in MIT's report on production and innovation [8], is that many of the companies working to develop these technologies are small and medium sized enterprises that do not have the corporate bandwidth and competencies to adequately address process scale-up and the associated quality inspection and control issues.

Workshop Description

Within this backdrop, several EERE technology offices, as well as AMO and EERE's Clean Energy Manufacturing Initiative (CEMI), have recognized the cross-cutting, pre-competitive, and enabling nature of quality control for a wide range of clean energy technologies.

Furthermore, there was a realization that many components and assemblies of interest to these offices are of similar format and that inspection and metrology solutions available or in development for one product may be applied, perhaps with minor modification, to other products of similar format. As a result, these offices and activities decided to explore QC needs and potential cross-office synergies by holding a workshop. The purpose of the workshop was to convene government, industry, and other stakeholders to discuss the current status of quality control/quality assurance and metrology in manufacturing processes relevant to the EERE offices; note gaps in which current diagnostic techniques are inadequate or missing altogether; discuss similarities in materials inspection and metrology needs across technologies; and identify opportunities for collaboration across EERE offices to address shared challenges. The results of the workshop are expected to inform EERE of opportunities to address synergistic and cross-cutting measurement and inspection needs for EERE technologies, leading to accelerated collaboration with and transfer of these technologies to industry.

Clean Energy Manufacturing Initiative

The Clean Energy Manufacturing Initiative is an integration of manufacturing efforts across all offices of EERE to advance (a) the production of competitive clean energy technologies and (b) activities and technologies that strengthen competitiveness across multiple manufacturing industries through increased energy productivity [15].

Photo from the Clean Energy Manufacturing Initiative launch on March 26, 2013 at the opening of the Carbon Fiber Technology Facility, part of the DOE Manufacturing Demonstration Facility at ORNL

A key initial task for the workshop planning team, which comprised members from FCTO, SETO, the Vehicle Technologies Office (VTO), BTO, AMO, and the National Renewable Energy Laboratory (NREL), was to establish the scope of the workshop within the technically broad topic of quality control. Generically, the team characterized quality needs across the spectrum of the manufacturing enterprise as shown in Figure 1. Important quality requirements exist across several categorizations of manufacturing activities, from quality assurance of incoming lots of raw materials to final testing of the operational performance of a system or assembly in the factory and at the point of use. However, the focus for this workshop was quality control at the part and/or sub-assembly level during manufacturing. At a lower level, the team identified three categorizations of products or materials of interest to these EERE offices that could require very different inspection and metrology capabilities: materials in the form of sheets or characterized by two-dimensional (2D) surfaces, piece parts or three-dimensional (3D) structures, and liquids. The team selected the first of these categories as the topic for the workshop—in part because many of the existing activities supported by these offices to address quality control relate to these kinds of materials—although a strong desire for follow-on workshops to address the other two areas was clearly noted. Because this first category of materials is often produced using continuous processes (e.g., roll-to-roll or belt-fed processes), "in-line" inspection—or techniques that can be used directly on the manufacturing line while the material is being produced, and thus are typically rapid, non-contact, and non-destructive in nature—is often of highest interest.

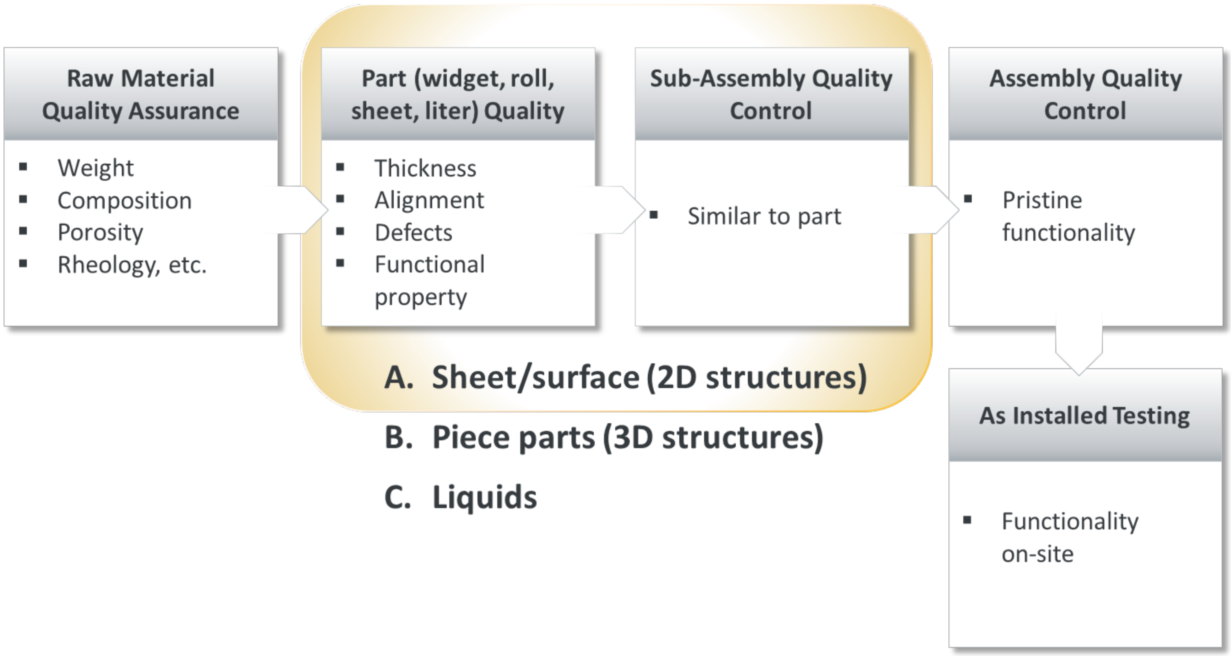


Figure 1. Workshop scope

The workshop was held at NREL’s Energy Systems Integration Facility in Golden, Colorado, on December 9 and 10, 2013. The structure of the workshop was designed to (a) establish a common baseline, in terms of processes, materials, and quality control techniques and (b) facilitate open and collaborative discussion to identify potential synergies, remaining needs, and key recommendations. These two requirements were important to enable easy and fruitful interaction between the participants, who represented a broad range of technologies. The following format was used:

- Welcome statements from NREL, FCTO, and AMO
- Overview presentations on relevant materials, processes, and quality control techniques
- An industry panel discussion on manufacturing quality control
- A summary presentation on current activities and in-line quality control needs and challenges previously identified by the participating offices
- Three breakout sessions
    - Breakout 1: A review of quality control challenges (four sessions were held, one for each of the technology offices)
    - Breakout 2: A discussion of potential synergies across offices and quality control techniques (three identical sessions were held, with mixed groupings of participants from across all technologies)
    - Breakout 3: A discussion of key quality control needs that are not currently available or being addressed, and final recommendations (again, three identical sessions were held, with mixed groupings of participants from across all technologies)
- A summary and wrap-up.

Tours of several NREL laboratories with ongoing research in quality control technique development were also held.

The final workshop agenda is given in Appendix A. Forty participants from DOE, industry, labs, and academia were present, as listed in Appendix B. Finally, detailed notes from each of the breakout sessions are given in Appendix C.

Current Status / State-of-the-Art for In-Line QC

As noted previously, similarities in the format and structure of materials across the different EERE offices were the main motivation for this workshop. Silicon wafers, battery electrodes, fuel cell membranes, and electrochromic window films are just a few examples of materials that are characterized by a two-dimensional functional surface, often with one or more coated or deposited layers. Not surprisingly, these materials are often made using similar processes—namely continuous roll-to-roll, belt-fed, or conveyor-based processes that enable successive steps to build a final construction at high throughput. While these processes are typically desirable from a cost-per-unit-area point of view, they make quality control difficult because measurements must be made while the material is moving and potentially under process line conditions (e.g., temperature, humidity, pressure, and non-air gas environments) that may be challenging for certain measurement techniques.

Example battery electrode (left) and thin film (right) materials. *Photos from David Wood, Oak Ridge National Laboratory (L) and Warren Gretz, NREL 03541 (R)*

Overview presentations were given to facilitate a common basis of understanding of the processes and quality control techniques in use and potentially of interest for this workshop. To further orient the participants, a summary of recent and ongoing EERE-sponsored quality measurement and inspection development activities relevant to these materials and processes was given. In addition, in recognition of the very important role that industry participant input would play in the success of the workshop, a panel discussion was held with panel members whose experience spanned several of the technologies of interest to the participating EERE offices. Summaries of these presentations and discussions are given in the following sections.

Overview of Relevant Processes

This overview by Michael Ulsh of NREL was broken down into two sections: a review of types of continuous production machinery, or “lines”, that these materials are typically fabricated on, and a review of the general process types that are employed on these lines to create the two-dimensional, often multi-layer surfaces of interest.

Continuous sheet-based lines take several forms. Roll-to-roll lines are used when a flexible continuous sheet, or “web”, can be conveyed on the line as exemplified in Figure 2. In addition to the web speed, the tension of the web is typically controlled to ensure that the motion of the web across and around a multiplicity of rollers is done in a way that does not cause stretching or wrinkling of the web. Belt-fed lines are similar and are used when support of the web during processing is required, for example during high-temperature process steps, or when sheet materials are not flexible enough to feed around rollers. Float lines are similar in concept and allow long sheets of material such as glass to be processed while moving and supported on a liquid surface. Finally, conveyors are used for cases such as silicon photovoltaic wafers wherein discrete parts are processed in a continuous fashion.

Figure 2. Roll-to-roll line. *Photo by Michael Ulsh, NREL*

Many different permutations of processes are used on these continuous lines—too many to review in detail. Instead, broad categories of processes were highlighted. Most of the materials of interest for the workshop involve some kind of coating or deposition—often several in series—to create functional layers and surfaces. These additive processes are categorized by the pressure at which the coating is applied: either at atmospheric (room) pressure or in a vacuum. Atmospheric coatings take several generic forms. Roll coating is characterized by two or more rollers, in a wide variety of configurations, being used to “pick up” a thin layer of liquid from a bath and apply it to a surface of a web. Knife coating is similar to roll coating, wherein a stationary bar or rod—the “knife”—is set to a certain stand-off distance from the web and is used to control the amount of liquid deposited onto the web from a reservoir. Various masks or other limits to the location or position of the coated liquid can be employed, as in screen printing. Two industry examples of knife-based techniques are shown in Figure 3. Die coating is the generic term for a wide variety of techniques characterized by a sheet of coating being dropped or laid onto the web. The die comprises two or more typically metal plates with machined flowfields between to enable the creation of a highly uniform sheet of coating. And finally, for the atmospheric coatings, spray methods are often employed using a spray head, or an array of spray heads, to coat the web from side to side. Low-temperature systems are used most often, including a variety of jet methods as well as systems where the head is ultrasonically actuated to break up droplets and particles into a very fine spray. In cases where the substrate or base material can withstand the thermal load, high-temperature sprays can be used, including electrical arc and plasma-based methods. In almost all cases of liquid coatings applied under atmospheric pressure, some kind of drying and/or curing of the coating is required. Drying is used to drive off solvents that are used to make a coat-able mixture but are not desired in the final layer, and it is typically accomplished using heated gas or infrared heat sources. Curing is a post-treatment process to finalize the chemical or morphological nature of the coating by irradiation with an energy source such as infrared or ultraviolet lamps, or an electron beam.

Figure 3. (Left) screen printing and (right) tape casting. *Photos from M. Richards, Versa Power Systems*

Vacuum coating techniques also come in a wide variety of types, including sputtering, many different kinds of vapor deposition processes, and evaporative coating. These processes are typically used for very thin coatings—usually less than a micrometer in thickness. Importantly, when vacuum processes are used in continuous production, complicated and expensive line equipment must be employed to allow movement of the web while still maintaining very low pressure. An example of this highly specialized type of equipment is shown in Figure 4. Especially in the case of silicon photovoltaics, several mechanical processing steps are also used, including cutting or sawing processes, texturing of the surface, and creation of electrical junctions.

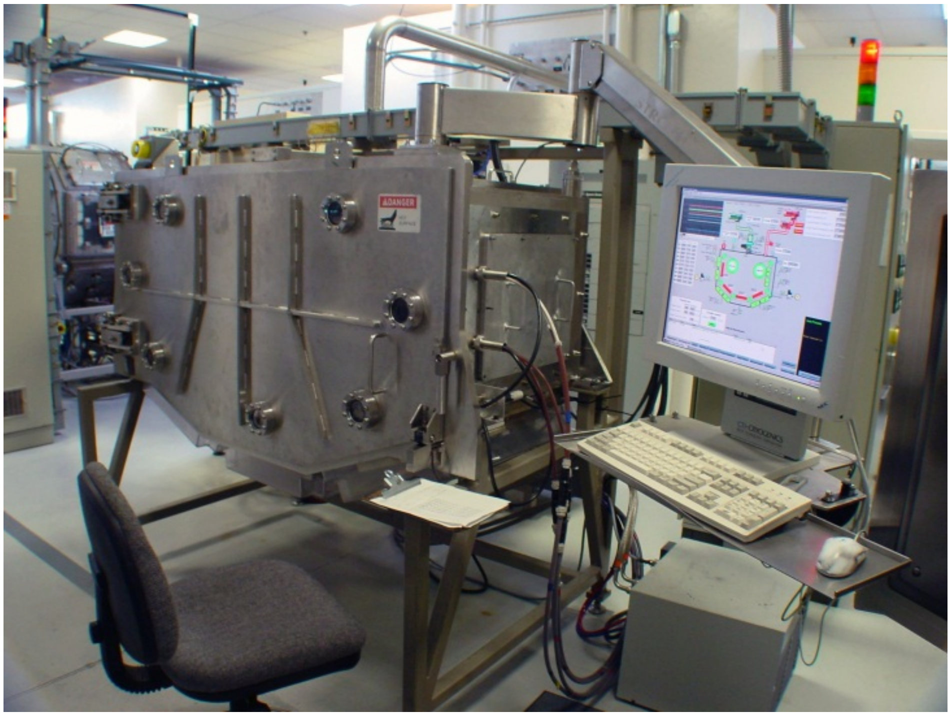


Figure 4. Reel-to-reel vacuum deposition line. *Photo from Global Solar Energy, NREL 13414*

Overview of Lithium Ion Electrode QC Development Activities at Oak Ridge

As a specific example of DOE-supported work on the development and validation of QC techniques for clean energy technologies, David Wood of Oak Ridge National Laboratory gave a presentation describing the state-of-the-art of production quality control for lithium ion battery electrodes. For in-line measurements, beta transmission gauges are commonly used to measure thickness and/or areal weight of the coating, and optical charge-coupled device (CCD) cameras are used for visible defects. The greatest needs for lithium ion electrode QC were given as pass/fail criteria for electrodes, correlation of defects with cell performance and capacity fade, and implementation of feedback loops from coating to deposition. X-ray fluorescence (XRF) for compositional analysis and optical microscopy for surface structure were described as techniques currently being employed off-line. A focus of the work at Oak Ridge National Laboratory (ORNL), which is sponsored by VTO, is to identify and develop techniques that cost less and are more easily implementable on production lines. To this end, laser-based thickness measurement and infrared (IR) thermography-based detection of defects is being demonstrated. Other activities being pursued included acoustic agglomerate-size measurement of electrode dispersions, IR-based thermal diffusivity measurement of electrode coatings to determine porosity, XRF techniques with improved accuracy and speed of measurement, and spectrophotometric analysis for thickness and areal weight uniformity. Highlights of these developments were given, including laser-based thickness measurement, as shown in Figure 5 for a wet lithium-manganese-rich cathode, where Δd/d represents a normalized thickness value.

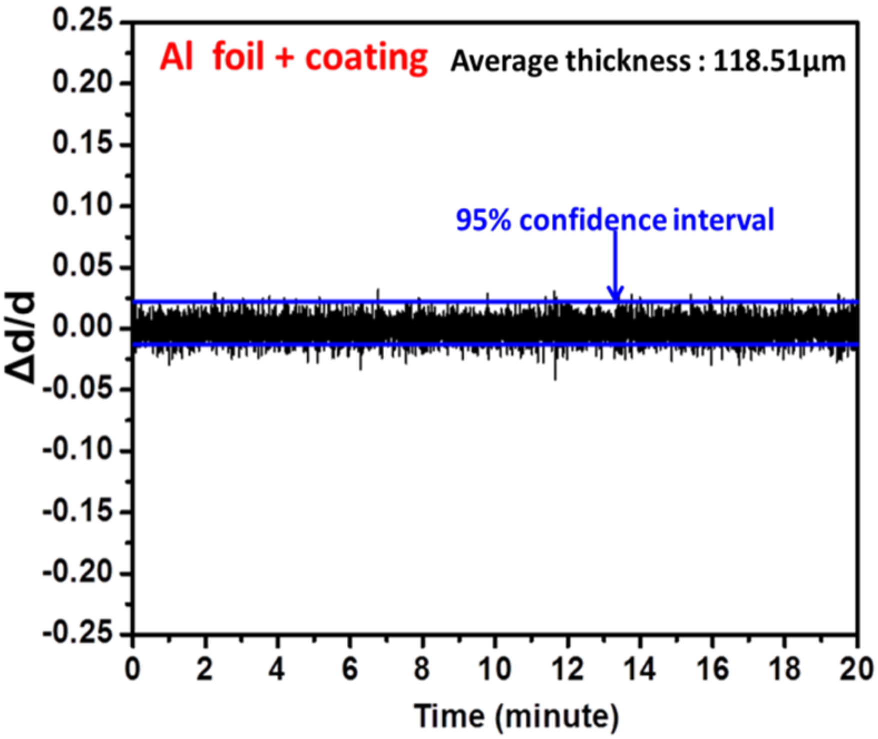


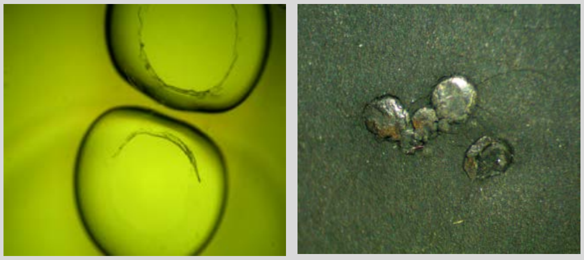
Figure 5. Thickness measurement of lithium manganese cathode using a Keyence dual laser system

Overview of Quality Control Techniques

As previously discussed, the materials of interest for this workshop are characterized by a functional surface created by one or more thin layers. Given that functionality is usually related to quantity, the thickness of the layers is usually very important. Beyond this, however, a wide range of measurements could be of interest, depending on the composition and functionality of the material. These measurements can be made with either single-point or multiple-point detectors. As a complicating factor, non-uniformity of these layers—resulting from a myriad of possible sources—often is not detected by single-point measurements. In a third presentation, Michael Ulsh of NREL reviewed schemes for enabling the required measurements given the above considerations, different types of measurements of interest, and examples of point, line, and areal detectors and techniques currently used or of interest.

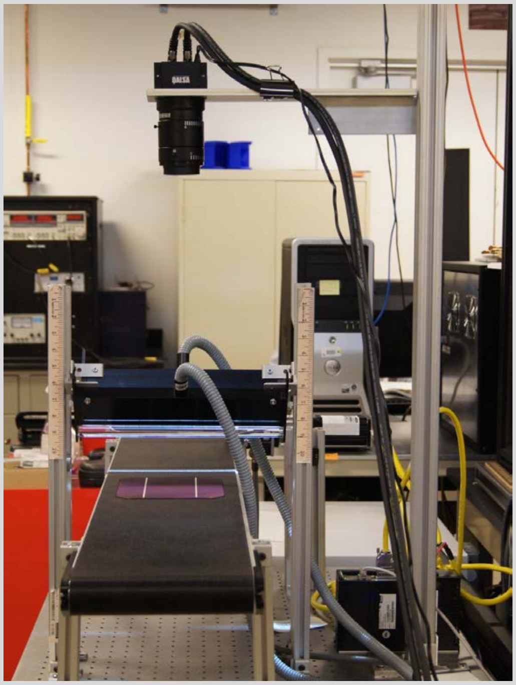
Beyond thickness, various dimensional measurements may be of interest. Measurement of the coated width of the material is often of interest for controlling scrap and ensuring materials are made to specification. Alternately, if the coating does not cover the entire surface of the substrate material, the distance from the edge of the coating to the edge of the substrate is often of interest. In cases of multiple layers, especially when patterned or patch coatings are employed—using screen printing or other masking methods—the registration or alignment of one layer on top of the previous layer is often very important. For example, misalignment of a fuel cell electrode on top of an ion-conducting membrane can reduce performance and lead to accelerated degradation.

In addition to dimensional measurements, functional measurements—or measurements of material properties—are often of interest. Chemical composition and mechanical, electrical, and optical properties may all be important to understand in-line, both from the perspective of process control as well as for proper functionality of the material. Catalyst content of electrodes, wafer grain boundaries, hydrophobic additive content in fuel cell gas diffusion media, in-plane and through-plane conductance, and the reflectance of a textured wafer surface all are examples of functional or property measurements.



Two examples of non-uniformities in materials
Photos by Michael Ulsh, NREL

Finally, identifying non-uniformities is almost always extremely critical for proper performance of these materials. Non-uniformities can come in many forms and result from many sources. Some non-uniformities can be surface effects—for example due to improper drying conditions—that cover all or large sections of a surface. They can be linear in the direction of motion of the web, for example lines of reduced thickness caused by a particle caught in a die. Or, they can be randomly dispersed discrete or point defects, such as pinholes, voids, or agglomerates.



Reflectance-based wafer monitoring system
Photo from Michael Ulsh, NREL

Many types of devices are commercially available or in development to meet these measurement needs, most of which are for point measurements. Laser-based measurements are often used for thickness, edge location, and alignment. Lasers typically probe only a single point (e.g., “triangulation” type gauges), but more recently “sheet” lasers of up to two inches width are becoming available to measure a local height profile or edge. A wide variety of other optical systems exist, especially for film and multi-layer thickness measurements. These devices are often based on interferometric physics and in some cases have the ability for spectroscopic interrogation. Devices based on various nuclear and X-ray point sources are widely available to measure coating thickness or weight and elemental composition. Beta and gamma gauges as well as XRF devices are well-known examples.

As mentioned above, the criticality of a particular measurement will determine how the device or devices are configured. In many cases, a single point measurement of the web is considered sufficient. In other cases, several point measurements are made at static locations across the web. In a final configuration of point measurements, which is often utilized with nuclear or X-ray sources, the source and detector are mounted on a linear motion device and rastered back and forth across the web. This final configuration is particularly useful for identifying die lines or other systemic variations that are continuous in the direction of motion. However, when discrete types of variability can cause significant performance or lifetime effects, point measurements may not be sufficient. In this case, areal or imaging-based measurements are required. “Machine vision systems” are widely known and used; they consist simply of a video camera coupled with front or back lighting and complex image processing algorithms. For defects characterized by visual contrast from their background, these systems are often sufficient and highly valuable. However, in many cases, more complex measurement techniques using CCD or pixel-array detectors are used or in development. In some cases, these systems use light as a source and can operate using reflective, transmissive, or absorptive modes. Detectors are, in some cases, also available for ultraviolet, IR, and X-ray based measurements. Figure 6 shows an example of a reflectance-based optical measurement showing grain boundaries and orientation on a silicon wafer surface. In addition, line detectors (i.e., a linear array of pixels) increasingly are available at various wavelengths that in some cases provide a higher pixel density than two-dimensional array detectors, and these can be used if a line measurement is sufficient.

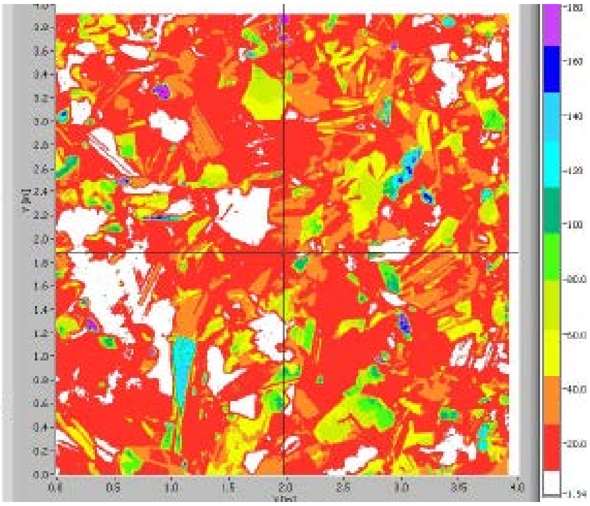


Figure 6. Optical reflectance measurement of silicon wafer grain boundaries

In most cases when line or array detectors are used to make measurements other than visual wavelength images or videos, an excitation source is used to excite some measureable response from the surface or layer of interest. Various wavelengths of electromagnetic radiation can be used, as discussed previously. An electronic excitation can be used either to make a functional measurement of an electrical property or to excite a thermal response via Joule heating that can be detected using an infrared CCD detector. Figure 7 shows an example of this latter case for a fuel cell electrode with a void. Similarly, a thermal heat source can be used to heat the material directly and, in specific cases where it is relevant, a reactive excitation can be used to generate a thermal signature.

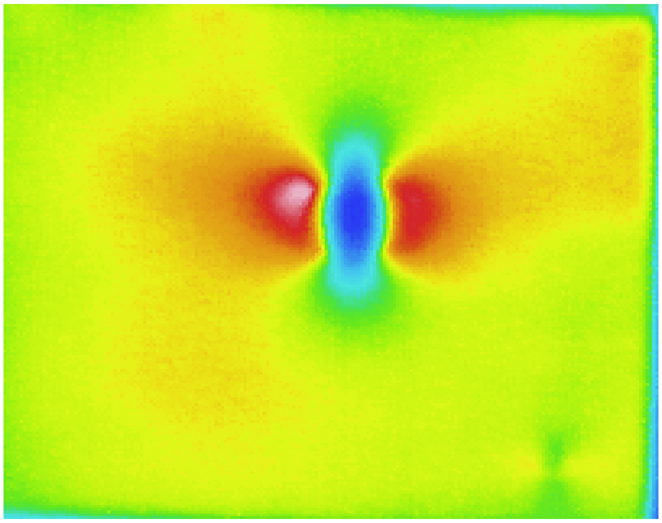


Figure 7. Thermal signature of fuel cell electrode with a void using electrical excitation and IR CCD detector

Status of EERE Activities

Workshop planning team members from each of the five EERE offices were queried to determine the status of previous and current activities in their offices related to the workshop scope. The offices were asked to provided information about current projects, past activities to determine needs and barriers, and whether specific needs and barriers have been addressed in program-wide plans as targets, tasks, goals, or milestones. This detailed information is given in Table 1 and was presented in summary form to the workshop participants to further inform them of the status of EERE activities across the different offices.

Table 1. Summary of EERE Activities

Question	Office	Response
Ongoing QC projects	AMO	Current projects: • Penn State University—Additive Manufacturing (NAMII, now America Makes) expands the use of thermal imaging for process monitoring and control • ORNL Manufacturing Demonstration Facility Projects Recent QC efforts funded by the Industrial Technologies Program “Grand Challenge” portfolio: • Next-Generation Wireless Instrumentation Integrated with Mathematical Modeling for Use in Aluminum Production (Wireless Industrial Technologies, Inc.) • Advanced Optical Sensors to Minimize Energy Consumption in Polymer Extrusion Processes (Guided Wave, Inc.)
	BTO	• The windows and building envelope program within BTO does not have any active projects specifically related to QC. • Process-related activities in BTO: ○ NREL—VI Window Film ○ ITN Energy Systems—Low-Cost, Highly Transparent Flexible low-e Coating Film to Enable Electrochromic Windows with Increased Energy Savings ○ Industrial Science & Technology Network, Inc.—A New

		Generation of Building Insulation by Foaming Polymer Blend Materials with CO_2 ○ Pacific Northwest National Laboratory (PNNL)—Dynamically Responsive IR Window Coatings
	FCTO	• NREL—Fuel Cell Membrane Electrode Assembly (MEA) Manufacturing R&D • LBNL—Manufacturing R&D for MEAs (sub to NREL)
	SETO	• Spire Corp—Photoluminescence for Solar Cell Crack Detection • Janoch Engineering, Inc.—In-Line Light Beam Induced Current • Sinton Instruments—Device-Physics-Accurate Cost-Effective and Module Test Instruments • MicroXact, Inc.—Real Time PV Manufacturing Diagnostic System • Tau Sciences Corporation—Apparatus for Optimizing PV Solar Manufacturing Efficiency through Real-Time Process Feedback and Spectral Binning • Plant PV—Three Dimensional Minority Carrier Lifetime Mapping of Thin Film Semiconductors for Solar Cells Application
	VTO	• ORNL—Lithium Ion Electrode Production Non-Destructive Evaluation and Quality Control Development
QC needs and barriers identified? (With industry participation or validation?)	AMO	• Road-mapping process conducted by NAMII and a topic added to the current open project call • The Industrial Technologies Program (AMO's predecessor) had a dedicated program activity entitled "Sensors & Automation" that funded R&D activities and also engaged industrial stakeholders
	BTO	Yes, this was identified as a need/barrier at: • Windows and Building Envelope Roadmap Workshop (April 2013) • CEMI Midwest Regional Summit (June 2013)
	FCTO	Yes, Hydrogen and Fuel Cell Manufacturing R&D Workshop (August 2011)
	SETO	SunShot Metrology Workshop (February 2012)
	VTO	Non-destructive techniques for the evaluation of the integrity of joints need further development (Multi-Year Program Plan, December 2010)
Documented office-wide specific targets, tasks, goals or milestones relative to QC?	AMO	No specific targets, tasks, goals, or milestones related to QC are currently included in AMO's planning documents
	BTO	No
	FCTO	Yes. Manufacturing R&D chapter in FCTO Multi-Year RD&D Plan including goals, targets, tasks, and milestones. http://energy.gov/eere/fuelcells/fuel-cell-technologies-office-multi-year-research-development-and-demonstration-plan
	SETO	SunShot vision study outlines $/W or levelized cost of electricity targets for solar. QC has an impact on yield and hence manufacturing cost. http://www1.eere.energy.gov/solar/pdfs/47927.pdf
	VTO	No
Additional comments concerning the relevance or importance of QC.	AMO	Suggest workshop discussion on QC needs be primarily focused on truly cross-cutting areas where multiple offices would benefit. QC efforts should also consider broader discussion beyond EERE, such as inclusion of the National Institute of Standards and Technology (NIST), which conducts a variety of activities in the manufacturing metrology space (e.g., http://www.nist.gov/manufacturing-metrology-portal.cfm).
	BTO	Future non-vapor compression systems will bring a lot of new joining challenges to HVAC, water heating, and appliances. Better joining

		techniques and testing these new joints will require new QA/QC testing procedures. A workshop that includes joining will allow manufacturers to communicate challenges to enable moving toward higher-performing (more efficient) designs and identifying reliable and cost effective techniques.
	FCTO	None
	SETO	QC methods to improve efficiency and yield and lower cost are very relevant to achieving SunShot goals. Our program currently funds projects under multiple funding opportunity announcements (FOAs) on this cross-cutting topic.
	VTO	The R&D community needs to produce other innovative in-line non-destructive evaluation techniques such as XRF, particle size measurements, FTIR, Raman.

The responses span a range of previous activities, likely related to the deemed criticality of manufacturing issues in the development and advancement of the different technologies. The impact of the breadth of materials and systems relevant to the different technology offices is also seen. For example, sheet materials such as window films are only a small fraction of the vast set of materials, systems, and technologies that are supported by the BTO for buildings.

Preliminary Industry Inputs

As an additional preliminary data gathering step, the workshop planning team decided to develop a short questionnaire for a small set of stakeholders. The intent of the questionnaire was to gain additional understanding of the status and implementation of in-line QC techniques for these or related technologies to aid the team in setting the structure and most useful discussion threads for the workshop breakout sessions. A summary of the responses to the preliminary questionnaire is given in Table 2.

Table 2. Industry Responses to Questionnaire

Question	Response
What materials are being produced in-line in a roll-to-roll process, what processes are being used to manufacture the materials, and what are the critical measurements of interest?	• Materials: o Bipolar Plates o Electrodes o Electrochromic films o Thin film flexible solid state batteries o Flexible thin-film copper indium gallium selenide (CIGS) based PV o Transparent conducting oxide and moisture barrier films o Non-woven fabrics textiles and films • Measurements of interest: o Micro-cracks o Catalyst homogeneity, pinholes o Band gap o Open circuit voltage o Conversion efficiency o Surface sheet resistance o Optical transmission measurements o Areas of roping (congealing)
What techniques are used in industry to identify and quantify defects in materials?	• Vision detection systems for cracks • Fluorescence of functional coatings applied to

	textiles - Non-contact eddy current measurements for surface sheet resistance - Non-contact optical measurements for band gap and relative thickness of coatings - Non-contact XRF for composition and also thickness of coatings - Photo-imaging for physical defects
What issues exist with the current quality assurance/quality control techniques?	- Lack of standards. A few companies sell cameras and algorithms, but not necessarily tuned to the application - Hardware exists. Main gap is software relevant to specific application - Need to be able to scan for the composition of coatings (for multi-material coatings) and physical defects across full width and length of web while web is in motion
What measurements are needed for in-line quality control that current techniques do not address?	- Physical defect density and/or pinhole density - Band gap measurements - Surface sheet resistance of coatings - Optical transmission - Relative thickness of coatings across and along the length of the web - Material composition measurements - Networking-cloud data transmission

Industry Panel Session

This section provides a summary of the industry panel session. Panel members were selected across multiple technologies and with backgrounds representing manufacturing, QC device development, and research. Questions for the panel were predetermined and are given below in Table 3 along with the names of the moderator and panel members. Panel responses to these questions are given in Table 4. After the moderated Q&A session, the session was opened to the workshop participants for additional Q&A, and these questions and responses are summarized in Table 5.

Table 3. Industry Panel Discussion Session

Industry Panel
Moderator: Arrelaine Dameron, NREL **Panelists:** - Everett Anderson, Proton OnSite, maker of PEM electrolyzers for hydrogen production - Bogdan Lita, Consultant, formerly of GE Primestar, maker of thin film PV - David Gotthold, PNNL, formerly in semiconductor industry - Ron Sinton, President of Sinton Instruments, maker of measurement devices for PV manufacturing
Panel Questions 1. What types of QC have you found critical and which are useful in-line? 2. Are there specific technical aspects of the manufacturing processes where current QC methods don’t work well (i.e., line speed, temperature, etc.)? 3. What are the economic considerations in choosing to implement in-line QC?

Table 4. Summary of Key Points from Moderated Panel Session

Panel Question	Panel Comments
1. What types of QC have you found critical and which are useful in-line?	• Existing thickness measurement techniques are not accurate enough for thin films (e.g., 10s of nanometers for organic light emitting diode coatings) • Large area thickness measurement is important for thin films • Need to measure platinum group metal catalyst loading in electrode coatings • Need to understand how to measure the quality of advanced material structures (e.g., binder morphology) • The move toward higher efficiency silicon technologies will necessitate additional/new QC techniques • There is very little understanding of the correlation between the defects detected on-line and the effect on the expected 20 year lifetime (of PV) • Many materials still use batch processes and no in-line QC • Processes are still changing for many of these materials—there will continue to be new or different measurement needs • Precision and repeatability of measurements are more important than accuracy • The use of statistical process control, in particular analysis of process capability, does or can determine needed frequency of measurement • For thickness or composition, a point works well to look for process drift • The two techniques (point and areal measurements) are complementary for silicon: point gets you a number, areal gets you an image (uniformity) • Identifying defects in black surfaces on black substrates is difficult for optical methods • Methods to locate an edge or alignment of edge are needed • There has been a push to use in-line imaging on PV lines, but it hasn’t happened yet to any great extent
2. Are there specific technical aspects of the manufacturing processes where current QC methods don't work well (i.e., line speed, temperature, etc.)?	• Fabrication tools generate heat as part of process: need measurements that can be independent and tolerant of process temperature • Process condition considerations: high vs. low temperature, wet vs. dry, vacuum vs. atmospheric pressure • (For polymer electrolyte membrane [PEM] electrolyzer MEAs) Electrodes are fabricated while the materials are wet: QC becomes difficult • Being able to make measurements at process speeds is important: but measurements are always “not fast enough”, regardless of line speed • Speed not as critical for early market technologies (e.g., hydrogen production equipment)

3. What are the economic considerations in choosing to implement in-line QC?	• QC must be applied when it can affect yield • We don’t want to make scrap, especially with very expensive raw materials • Perception or observation that in industry, in-line data is ignored because they don’t want to shut down the equipment just because the measurement is showing something out of spec—“the measurement must be wrong” • Using in-line data in a feedback-loop to control equipment is ideal, but not really happening • Perception or observation that industry uses metrology to help optimize the process, but once the line is running, they turn the measurements off • Use of in-line QC to some extent depends on the commodity level of the material: lower price = measure less

Table 5. Summary of Audience Q&A Session with Panelists

Audience Question	Panel Answer
Are there specific QC/metrology needs for clean energy products?	QC techniques may have to be aligned to specific technologies, but generically they must all be non-invasive and cannot slow down the line.
Scaling up processes means transitioning from human to software-based decisions—are software control systems ready?	It’s happening but still at the R&D level. Needs to be cheap as well as fast and accurate.
We need to discuss process control and feedback. If we’re not measuring or providing feedback, the process isn’t controlled. How realistic is in-line process control with these complex systems?	(a) There are off-line measurements for voltage, open cell voltage, thickness, and resistivity at each part made to make sure the process is in control and to measure for batch-to-batch variation/impact. (b) Determination of “in control” doesn’t need to be in-line—it’s easier to do off-line, but we should hope to move more in-line. (c) If finished product shows something wrong, where did the process go bad? Would like in-line measurements to identify where in the process or at which process step things go bad.
These clean energy industries are immature compared with others (e.g., steel making, auto industries). Six-sigma has not been built in to all of the process steps. Are there lessons learned?	Don’t know where to start with lessons learned for the steel industry. PV is immature—there have been large changes made in a short amount of time. It is not yet understood how to predict how these changes will affect the final product.

Summary and Key Learnings from the Breakout Sessions

Three breakout sessions were held to engage the workshop participants in creative and critical discussions on QC needs and opportunities for the materials of interest for the workshop. Breakout 1 was intended as a technology-specific review of QC challenges. As such, four separate sessions were held, attended by the industry, lab, and DOE participants associated with each of the four technology offices. Breakout 2 was a discussion of similarities among measurement and inspection needs across the different technologies and of whether synergies may exist where QC technologies or techniques in development for one technology may be useful for another technology. To enable cross-cutting perspectives, the participants representing the technologies of each office were mixed together in three identical groups. Finally, breakout 3 was an assessment of quality control needs that are not being addressed by current development activities and a discussion of ways that industry together with EERE can move ahead in identifying and supporting cross-cutting QC development activities. This session was attended by the same three mixed groups as for breakout 2. Each session was led by a facilitator and assisted by a scribe. In the sections below, the discussion questions, summary outputs, and key learnings from each breakout are presented and discussed. Detailed notes from each of the breakout sessions are given in Appendix C.

Breakout 1: QC Challenges

Table 6 shows the objective, questions, and discussion threads posed to the participants in each of the breakout 1 groups. The purpose of this breakout was to provide each technology group an opportunity to review previous activities supported by their office to identify QC needs and barriers, to discuss the overviews presented earlier in the workshop, and to propose any needs that were not previously identified.

Table 6. Objective and Discussion Questions for Breakout Session 1: QC Challenges

Breakout 1 Objective: ***Produce a prioritized list of QC challenges for your technology area***
Questions and discussion threads: • Many of the offices have supported manufacturing workshops or other data gathering activities in the past few years—review these outcomes, especially if QC, measurements, and metrology were discussed. • Current office activities in this area were summarized earlier in the workshop—are there any comments, questions, or discussion about those activities? • Overviews of current processes and QC techniques were given earlier in the workshop—are there any comments, questions, or discussion about what was presented in those overviews? • Do any of you have any additional thoughts or inputs in regard to what QC challenges exist in our technology area that may not have been captured in the overviews or the previous DOE information-gathering activities?

Table 7 shows the summarized responses from the breakout by technology group. Given the differences in status and maturity of each of the technologies, the specific materials and processes of interest to each group, and the activities that each office has chosen to pursue in the past relative to manufacturing and quality issues, it is not surprising that the outputs of the different groups varied significantly. However, several key topics emerged that were similar across the groups and could be taken as cross-cutting learnings.

- Most of the groups identified "understanding the effects that specific types of defects will have on cell/device performance and lifetime" as a critical need. Associated with this need was an improved understanding of the role that accelerated testing can and/or should play and the need for development of methods to mark defects for later removal.
- While expressed in different ways by different groups, the participants identified a general need for better ways to quantify the return on investment associated with developing and implementing in-line QC, especially given the currently small production volumes of many of the technologies of interest. An exemplary anecdote was given of a company that decided to leave a cheap gate valve in a system because it was less expensive to replace the few that failed than to include a more expensive valve in each system.
- There was a general assessment that many of the critical QC needs were related to functional rather than dimensional measurements, with the exception of layer or coating thickness.

Table 7. Summary Responses from Breakout Session 1 by Technology Group

Technology Group	Responses
SETO *(this group voted on most important items in discussion, as indicated in parentheses)*	• Understanding of defects with respect to product performance (Everyone) • Cost of new QC technology implementation and return on investment (Everyone) • How do defects affect the expected reliability over decades of expected lifetime (4) • Speed of QC data acquisition (4) • Resolution of measurement techniques/field of view (3) • Challenges to justify cost of QC due to size of market • Observation thermography for detection of solar cell shunts, shorts • Optical defects can be significant: deviation in color, optical flatness, bubbles, voids • Need improved sensitivity of sensors, along with algorithms for the sensors, to detect defects • How many and what type of defects matter? • Can a Pareto chart with main types of defects be developed? • Algorithms must be able to evaluate between number, type, and criticality of defects • "X" defect will do "Y" to the system is not always known • Provide service instead of tools? Prepay for consulting services to help instruct the users on the proper use of the equipment? • Return on investment—customers don't see the cost benefit of having correct QC/metrology • Return on investment—lump in with cost of new technology?
FCTO	• Difficult to measure electrochemical properties of solid oxide fuel cells (SOFC) in-line (need to bring up to operating temperature) • Dimensional metrology is well understood; thickness, height, dimensions can be measured

	- Membranes: limited pinhole detection; mainly rely on supplier
	  - Pinhole length scale is not well understood; isn't clear what size results in failure
	- Bipolar plate:
	  - Detecting micro-cracks
	  - Measuring nitride (anti-corrosion) layer thickness
	  - Degree of planarity/flatness
	  - Leak checking
	- Electrodes: thickness and catalyst content
	- Gaskets and seals: need better understanding of what properties lead to better performing seals
	- Need to correlate defects to performance loss or degradation
	- Need further development of accelerated testing methods for all components

VTO	- Key defects/criticality:
	  - Metals from mixing/slitting debris causing short circuit, overheating. Effects can be grave, but seldom happens. Possible metrology: optical, thermography
	  - Coating defects (pinhole, blister, porosity) from mixing, slurry process affect life, medium gravity/frequency
	  - Material agglomerates from slurry affect safety, medium gravity/frequency
	- May need to mark scrap material—a lot of material scrapped due to false positives
	- Performance or quality of battery: has this been mapped back to defects?
	- How much can we learn from other technologies?

BTO	- Accelerated aging tests - Embedded sensors to provide real-time data - "Buildings" is a very large topic with many components; treat building like a system - Environmental and public pressures on manufacturers to change a material and/or product make developing QC for specific materials difficult

Breakout 2: Synergies

Table 8 shows the objective, questions, and discussion threads posed to the participants in each of the breakout 2 groups. The purpose of this breakout was to determine if, and in what ways, synergies existed between the QC needs and/or capabilities of one technology and the needs and/or capabilities of the other technologies. For example, in a collaboration exemplified during the overview talks, NREL has previously taken QC techniques developed for in-line inspection of PV wafers and, with modifications appropriate to the differences in the materials, developed and demonstrated techniques useful for fuel cell membranes and electrodes. These kinds of opportunities, across offices, manufacturers, QC device makers, and labs, are being sought by breakout 2.

Table 8. Objective and Discussion Questions for Breakout Session 2: Synergies

Breakout 2 Objective: ***Produce a prioritized listing of possible synergies***

Questions and discussion threads:

- We just heard summaries of the most critical challenges and needs from each of the technology areas—are there any initial thoughts or comments?
- Discuss what we mean by synergies
- Are there any initial thoughts about synergies when you look at the challenges across the different technologies?
- List any ideas you have of where a technique that is available or being developed for one material or technology may be applicable to a different material, keeping in mind the key challenges we heard about earlier
- Again, given what we've heard about key challenges, we'd like to get your input on prioritizing these ideas
- What ideas do you have about facilitating different companies or researchers from different areas getting together to address these potential synergies?

Table 9 shows the summarized responses from the breakout by group. While each group pursued a slightly different path in its discussion, many similar themes emerged across the three groups. Key synergistic opportunities, based on the types of measurements needed for the different materials, were identified as follows:

- Measurement of thickness
- Inspection for "mechanical" defects such as pinholes and cracks
- Optical measurements of texture, morphology, and surface structure
- Measurement of electrical properties.

In general it was recognized that all techniques need to be rapid, non-contact, and non-destructive. Associated with these similarities, however, the groups identified several key factors that must be taken into consideration when assessing and pursuing potential synergies.

- Relative to the critical measurement of thickness, the groups pointed out that the difference in length scale between the thicknesses of, for example, thin film PV layers and coatings for battery or fuel cell electrodes may span three or more orders of magnitude, from 10s of nanometers to 10s or 100s of micrometers.
- Relative to electrical measurements, the groups pointed out that in the case of PV cells, actual voltage measurements are desired based on the junctions deposited on the wafer. On the other hand, for battery and fuel cell electrodes, no *ex situ* voltage exists. Rather, measurements of electrical properties such as in-plane and through-plane resistance are needed.
- Differences in the criticality of similar types of defects or non-uniformities across the different applications were identified. For example, one group pointed out that a pinhole in a layer of a PV device may have a small performance effect, but the same-size pinhole in a battery separator may cause failure.

Additional synergistic considerations were identified, including restatement of some of the themes from breakout 1.

- The cross-cutting needs to better understand the effects of defects on performance and to have tools and methods to quantify the value of QC in low-volume markets were repeated.
- The linkage of QC measurements to process control, and more broadly the acquisition, analysis, and use of large quantities of data in a continuous manufacturing environment, was seen as a need across all of the technologies.
- The need for improved sensitivity of devices was also identified as cross-cutting—not only sensitivity to smaller length scales, but sensitivity to lower levels of contrast. One group discussed the impact of developing cameras and software that can "see and evaluate" differences like the human eye and brain can.

Finally, there were several administrative ideas about how to more efficiently identify and initiate synergistic activities between vendors and researchers who supply and/or develop QC techniques, but who rarely get to interact because they support different technologies or different offices. In general the suggestion was for DOE to support follow-on activities to the workshop of a much more technical nature to facilitate relationship building and a more detailed level of understanding of what capabilities exist, what is under development, and what similarities may be exploited.

Table 9. Summary Responses from Breakout Session 2 by Group

Group	Responses
Group 2A	• Topography—similar for batteries and fuel cells? • Thickness measurements of different scales—nanometer vs. micrometer • Measurements related to texture, scattering (PV, fuel cells, batteries) • Intellectual property issues are ever-present for universities or labs trying to work with industry • Pre-commercialization is a good time for labs and universities to develop process control systems—before a company scales up • Who is responsible for making QC checks (the device maker or the material supplier)? • In-line X-ray measurement • For PV, electrical measurements made of actual device voltage once connections are made on cell—for fuel cell and battery, would need *in situ* test for voltage, so want to make measurements of electrical properties, not actual voltage measurement • Some defects accelerate failure • Exchange of technical methodologies needed between researchers • Smaller technical meetings (or webinars) to discuss methodology and how one new device/process could be adopted by others either in their field or across technologies • National lab summit • Not high enough volumes yet to readily delve into looking at these issues • Electrical or electronic measurements important • How and when in the process should these measurements be taken? • Carve out a session at this year's Hydrogen/Vehicles Programs' Annual Merit Review? What about doing a national lab webinar series? • Each office needs to better understand the impact of defects on performance and durability to correlate those defects to not only reduced scrap/higher yield, but also to some kind of cost benefit
Group 2B	• Method needs to be non-invasive, non-contact, fast • Correlation of defects to performance is not clear • Common problems are difficult to define due to different applications

	- Coatings have common mechanical problems: pinholes, cracks
	- Does defect affect performance later or result in immediate problem?
	- Cost of defect detection has to be lower than cost of defect—perceived return on investment
	- 10 nm scale thickness needed for thin films
	- Commonality between applications
	  ○ Pinholes, micro-cracks
	  ○ Critical defects for different applications might differ in size
	  ○ Compositional homogeneity
	  ○ Reliability
	  ○ Accuracy, false positive avoidance
	  ○ Statistical quality control is required to perform metrology effectively

Group 2C	- Although there are synergies across the technologies, they are application specific. For instance a type of defect might be catastrophic to one technology, but not to another ○ Pinhole is limited in how it will affect solar technologies; for battery it could kill the entire battery - Quality of deposition, registration of layers - Techniques for leakage and pinholes - Not enough understanding of long term aging effects - Inspection systems that approximate the acuity of human vision system would probably solve problems in the industry across all technologies

Breakout 3: Remaining R&D Needs and Recommendations

Table 10 shows the objective, questions, and discussion threads posed to the participants in each of the breakout 3 groups. The purpose of this breakout was to identify critical quality control needs that are not being addressed by currently-in-development activities and to suggest directions and actions that industry together with EERE can take to identify and support cross-cutting QC development and demonstration activities.

Table 10. **Objective and Discussion Questions for Breakout Session 3: Remaining R&D Needs and Recommendations**

Breakout 3 Objective: ***Produce a prioritized listing of R&D needs for EERE to address, identifying those that are cross-cutting***
Questions and discussion threads: - We've just heard summaries from the first two breakouts of the workshop—are there any comments or discussion? - Breakout 1 discussed QC challenges and we've heard about existing or developmental QC techniques that do or may address these challenges—are there key measurement and inspection needs that aren't being met by commercially available techniques or EERE-supported technology development efforts? - Could any of the techniques that have been discussed be applied to these needs? - Are there techniques that haven't been discussed that may be applicable? - Are any of these needs cross-cutting?

Table 11 shows the summarized responses from the breakout by group. In terms of missing capabilities, while many needs undoubtedly still exist, the main discussions centered around three main areas:

- Inspection for delamination, inter-layer voids, and bond strength, noting that almost all of the materials of interest to the workshop are multi-layer in construction (either fabricated in successive layers or laminated)
- Machine vision systems that can replicate the human eye—and going beyond, further development of inspection techniques that are not limited by optical wavelength
- Improved tools and methods to collect, analyze, store, and use the large amounts of data generated by in-line QC, with additional discussion of "post-production" data mining to identify the sources of product failure or poor performance in the field.

Table 11. Summary Responses from Breakout Session 3 by Group

Group	Responses
Group 3A	• Pinholes/defects that do not seem to be an issue at the time the device is manufactured but rather as the device works • Delamination of multi-layer structures; layer-to-layer bonding/adhesion • Bond strength may be difficult to measure in-line while void detection may be easier to observe in-line • Acoustic tests: each sample has its own resonance curve, adhesion defects will show in a modified resonance • What will DOE do with these ideas? Support for cross-office activities • Find lessons-learned from Europe, where large collaborative scopes are more common • Make teaming necessary for FOAs? • Need buy-in from manufacturers and tech transfer to industry, enabled by Small Business Innovation Research (SBIR) funding? • Improve collaboration mechanisms between industry, vendors, and researchers • Cost benefit of QC ○ Use manufacturing cost analysis, if QC is included in models? ○ What is the cost of *not* having QC? Real processes never have 100% yield, but the manufacturers never give out the exact yield—business sensitive ○ Example of fuel cell stack yield vs. increase in stack costs; if QC measures are not taken, the percentage of bad product significantly increases costs • Set up more fabrication labs to investigate the use of QC/metrology in manufacturing setting • Intellectual property issues undermine collaborations and effective teaming • A major challenge/barrier to working on synergistic research topics is how the Annual Operating Plan (for DOE labs) process works ○ Encourages principal investigators to work inside their silo ○ Need some sort of shareable Annual Operating Plan ○ Also need better support for working with industry partners
Group 3B	• Process control development is still a need • Example of a Defense Advanced Research Projects Agency (DARPA)-supported consortium with nine companies to look at vapor phase manufacturing of CIGS and how to control four element deposition correctly • Offline techniques might be available—how to bring them online? • BTO has more of a focus on equipment design to improve the energy efficiency of a

	piece of equipment or appliance than on QC or metrology as a subject, though QC might be used or needed for a product • Catalogues needed of available QC techniques • Understanding lacking on the laboratory side about industry/manufacturing needs • Manufacturer does not want metrology on their line: only accepted if there is an impact on their productivity and bottom line; if they have a problem and it hurts • Most QC techniques signal a problem but do not give guidance for correction • Must have collaboration with manufacturer • Need software enabling use of in-line data for process control

Group 3C	• Vision system that replicates the processing ability of human brain/eye
	• Detect things smaller than the diffraction limit of what the human eye can detect
	• Look at physical features in intermediate scales (looking at small things over large areas—one defect in a vast area)
	• How to store measured data now to be analyzed later?
	• Use big data mining techniques to accumulate a lot of data
	• Oversample and then try to look back at the data
	• Go back in time and see what is statistically significant—large data set may show trends, but we need the data set first
	• Getting process equipment manufacturers to work more closely with QC vendors
	• Define a real vs. a perceived defect. What’s a variation vs. an actual defect?
	• We throw out a lot of material from being too conservative with perceived defects
	• Generate a table that summarizes QC techniques and their capabilities (defect size, line speeds, etc.)
	  ○ Make a table for renewable energy technologies for defect definition, irregularities, how to detect them and different synergies
	  ○ A table showing QC techniques used and how they are applied and then have equipment vendors and others come in and fill gaps for areas of QC that aren’t well defined
	• Metrology webinar series under the AMO umbrella
	• Taking technology from the laboratories and demonstrating it at real companies for a brief period
	• Could make companies hesitant because of QC issues that get sampled—manufacturers don’t necessarily want their defects or manufacturing issues to be known by the outside

Looking forward, the groups discussed numerous issues for EERE and industry to collectively overcome to more effectively pursue impactful cross-cutting QC RD&D. One broad category of ideas centered on improving collaboration with industry. Participants noted that manufacturers often are hesitant to collaborate on QC issues because they do not want their manufacturing issues to be known. This hesitance is compounded by ever-present intellectual property concerns, especially when working with universities or labs. Better methods to assess and present the cost-of-poor-quality/cost-benefit of QC was seen as a potential way to improve manufacturer buy-in and ultimately lead to increased technology transfer. Similarly, the groups saw finding methods to foster increased interaction and integration between fabrication equipment manufacturers and QC vendors as a valuable direction. Finally, topics discussed in previous breakouts were echoed, including increased efforts in correlating defects to performance to mitigate what was stated to be widespread scrapping of material due to “perceived defects”, and integration of QC into process control.

Key recommendations for EERE to facilitate better cross-technology and cross-office interactions were the following:

- Evaluate and implement better ways to promote teaming across labs, industries, and activities—in particular, better ways for labs to collaborate within the very office-specific Annual Operating Plan process
- Consider setting up focused industry/lab collaborations on specific issues, with the DARPA-sponsored CIGS roadmap activity as an example
- Continue and expand SBIR topics in this area to promote industry/lab collaboration and technology transfer
- Catalog for clean energy technologies the types of defects and non-uniformities and the available QC technologies for addressing them
- Hold a metrology webinar series and/or other means of informative outreach to improve manufacturers' exposure to and understanding of available QC techniques, especially the small and medium enterprises that typically do not have the internal resources to pursue QC.

Conclusions and Recommendations

The EERE QC Workshop was structured to create a common understanding of needs and capabilities across the technologies represented by the five offices that co-sponsored the activity and to facilitate creative and critical discussion regarding opportunities for EERE and industry to work together in addressing cross-cutting needs for in-line quality control, inspection, and metrology. From the overview presentations to the industry panel and series of breakout sessions, participants were engaged and encouraged to identify synergistic opportunities for collaboration and ways that industry and EERE could promote improved communication, collaboration, and technology transfer between labs, equipment suppliers, clean energy technology manufacturers, and QC device vendors. The outputs of the workshop logically lend themselves to categorization into conclusions and recommendations, the former taking the form of specific technical topics to be addressed and the latter as proposed activities for EERE and industry to pursue.

Conclusions

Cross-cutting QC needs: The participants clearly identified the following QC measurement needs that cross-cut the different technology areas and that are potential opportunities for cross-office collaboration:

- Thickness measurement
- Inspection for mechanical defects such as pinholes and cracks
- Measurement of electrical properties such as resistance
- Measurement of surface texture, structure, and morphology
- Inspection for inter-layer delamination and voids.

Participants also identified several counter-balances to these synergies—considerations that must be made when establishing cross-office collaborations—including:

- Differences in scale—for example, measuring nanometer-scale layers in thin-film PV as opposed to micrometer-scale coatings in fuel cell and battery materials
- Differences in criticality of defects or non-uniformities between applications—for example, pinholes in PV layers versus in a battery separator
- Whether or not actual performance can be made *ex situ*—for example, voltage measurement in a PV cell with junctions versus through-place resistance of a fuel cell membrane electrode assembly.

In addition to these specific measurement needs and considerations, the participants identified two higher-level technical goals:

- Improving the sensitivity of detectors to smaller size scales (i.e., from micrometers to 10s of nanometers) as well as to smaller levels of optical contrast

- Advancing tools and methods for the collection, analysis, storage, and use (either in real time or for later data mining) of high volumes of in-line QC data and for the integration of these data into process control and feedback systems.

Correlation of defects to performance: Throughout every part of the workshop, participants communicated the need for an improved understanding of the effects that defects in each of these materials have on device performance and lifetime. The complexity of the need was clearly stated, as these effects could and do depend on, for example, the shape, size, depth, extent (single vs. multiple defects or defects that cover large areas of surfaces), aspect ratio, and location on device of the defect. The fact that the criticality of similar defects in different applications could be very different was also clearly stated. This need implies detailed *ex situ* and *in situ* testing of materials as well as correlated model development efforts to increase the capabilities of measurement systems to not only indicate a defect but also suggest causes and steps for mitigation.

Cost-benefit analysis: The early market, low volume status of many of these clean energy technologies hinders collaboration with manufacturers in the development and demonstration of new and useful QC techniques, either because the companies are small and lack the internal expertise to address this scale-up issue or because there is a lack of funds to address an issue that is perceived to be important or valuable only in full-scale production. Accordingly, the development of tools and methods to enable a manufacturing-cost-based analysis of the cost benefit of in-line QC implementation was seen as a strong need. A preliminary assessment of the cost-of-poor-quality as a function of manufacturing volume for automotive fuel cell stacks was shown as an example, and it was noted that detailed manufacturing cost models have been developed for several of the technologies and could perhaps be utilized as a basis for these analyses.

Recommendations

Improve communications and interactions with industry: As noted above, a variety of factors hinder interaction with manufacturers on this topic, including real or perceived lack of value as well as very real and complex issues such as confidentiality of manufacturing activities and information and associated intellectual property issues. The following pathways were suggested to increase communication, better understand best practices, and address confidentiality issues:

- Develop and make available—via a Web-based searchable database, for example—a catalog of QC techniques, their capabilities, applications, and suppliers/developers, with inputs from QC vendors and lab developers
- Support continued inclusion of QC-related topics in funding opportunities and SBIR calls and consider focused DOE-supported industry/lab collaborations to address specific technical issues
- Hold webinars and post case studies on appropriate websites on QC topics and techniques to enhance industry's understanding of the potential capabilities and value of QC implementation and to facilitate connections between the material manufacturers, QC vendors, and lab researchers

- Connect with other U.S. government entities who may have established best practices for interaction with industry on manufacturing topics, including the Advanced Research Projects Agency-Energy (ARPA-E), DARPA, the U.S. Department of Defense Manufacturing Technology Program (DoD ManTech), and NIST
- Develop QC-specific tasks within the Partner Program, a potential new effort under CEMI, where national lab staff can work directly with industry to develop appropriate tools and capabilities that can benefit the broader industry without compromising an individual company's intellectual property.

Facilitate technical exchange between researchers, developers, and vendors: As noted in the breakout summary section, interaction and relationship development between researchers, developers, and suppliers is hindered because they most often work in or support only one specific technology or office and, for example, typically do not attend the same technical conferences or program reviews. Thus, technical exchange at the level of detail perhaps required to facilitate a deeper understanding of QC development activities, methods, and techniques—and to actively seek and identify synergistic activities—rarely occurs. Participants recommended the organization of events, potentially co-located with technical conferences or DOE reviews that at least some of the researchers and developers would normally attend, where scientists could present details of their own work, capabilities, equipment, and methods and could have group and one-on-one interactions and discussions with other researchers.

Participants also identified the difficulties that lab researchers, who normally support a single EERE office through an Annual Operating Plan that is very focused on the goals and milestones of that office, encounter when trying to collaborate with researchers in other technology areas to support cross-cutting needs. Specifically, there is a perceived lack of support, facilitation, and recognition/reward for such interactions by the EERE offices. Participants recommended further discussion on improved methods and mechanisms at the EERE level to enable effective cross-office interactions between lab researchers. A near-term opportunity of this type is the developing collaboration between NREL and ORNL for in-line QC. The group aims to leverage the considerable capabilities at both labs in QC and process development, with an initial focus on addressing battery electrode QC needs. This effort brings together projects and researchers currently supported by FCTO, VTO, and SETO and links them with strong AMO-supported activities for manufacturing development at ORNL, potentially providing a pilot for broad, cross-office EERE collaboration in this important enabling topic area of advanced manufacturing.

References

1. “Report to the President on Capturing Domestic Competitive Advantage in Advanced Manufacturing,” President’s Council of Advisors on Science and Technology, July 2012.

2. “National Network for Manufacturing Innovation: A Preliminary Design,” National Science and Technology Council and Advanced Manufacturing National Program Office, January 2013.

3. “Report to the President on Ensuring American Leadership in Advanced Manufacturing,” President’s Council of Advisors on Science and Technology, June 2011.

4. Department of Defense, *Quadrennial Defense Review Report*, February 2010, p.103.

5. “A National Strategic Plan for Advanced Manufacturing,” National Science and Technology Council, February 2012.

6. “Manufacturing Demonstration Facilities Workshop,” DOE Advanced Manufacturing Office, March 12, 2012, Chicago, IL.

7. “Production in the Innovation Economy,” MIT Club of Washington DC—Annual Seminar Series, November 13, 2012.

8. “Report of the MIT Taskforce on Innovation and Production,” February 2013.

9. “Membrane Technology Workshop Summary Report,” DOE Advanced Manufacturing Office, November 2012.

10. “2011 NREL/DOE Hydrogen and Fuel Cell Manufacturing R&D Workshop Report,” DOE Fuel Cell Technologies Office, February 2012.

11. “Metrology Workshop Report,” DOE SunShot Program, February 7, 2012, Crystal City, VA.

12. “Solid-State Lighting Research and Development: Manufacturing Roadmap,” DOE Building Technologies Office, August 2012.

13. Fuel Cell Technologies Office Multi-Year Research, Development and Demonstration Plan, Chapter 3.5: Manufacturing R&D, 2012.

14. “Manufacturing the Future-Federal Priorities for Manufacturing R&D,” National Science and Technology Council, March 2008.

Appendix A. Workshop Agenda

EERE QC Workshop Agenda
December 9–10, 2013
NREL, Energy Systems Integration Facility

Monday, December 9

8:00	Sign-in and continental breakfast
8:30	NREL welcome: Keith Wipke, Lab Program Manager, NREL
8:50	EERE welcome: Nancy Garland, FCTO; Joe Cresko, AMO
9:15	Intro to the structure and objectives of the workshop: Cassidy Houchins, SRA
9:30	Overview of current processes: Michael Ulsh, NREL
9:50	Review of Oak Ridge process and QC activities: David Wood, ORNL
10:05	Morning break and networking
10:20	Overview of current in-line QC: Michael Ulsh, NREL
10:45	Summary of current QC activities in EERE: Cassidy Houchins, SRA
11:05	Industry Panel: Quality Control for Clean Energy Manufacturing
	Moderator: Arrelaine Dameron, NREL
	Panelist: Everett Anderson, Proton OnSite
	Panelist: Bogdan Lita, Consultant, formerly of GE Primestar
	Panelist: David Gotthold, PNNL
	Panelist: Ron Sinton, Sinton Instruments
12:00	Lunch
1:00	Breakout 1: QC challenges by technology area
1:50	Break/pause for breakout 1 summaries
2:00	Report-out on breakout 1 (3 or so minutes for each breakout)
2:15	Breakout 2: Synergies across technologies
3:15	Break/pause for breakout 2 summaries
3:25	Report-out on breakout 2 (5 or so minutes for each breakout)
3:50	NREL tours
5:00	Adjourn

Tuesday, December 10

8:00	Continental breakfast
8:30	Summary from breakouts 1 and 2: workshop planning team
9:00	Breakout 3: R&D needs for QC techniques
10:15	Break/pause for breakout 3 summaries
10:25	Report-out on breakout 3 (5 or so minutes for each breakout)
10:50	Closing remarks: workshop planning team
11:15	Adjourn

Appendix B. List of Attendees

Name	Organization
Jesse Adams	Fuel Cell Technologies Office, DOE
Everett Anderson	Proton OnSite
Joe Berry	National Renewable Energy Laboratory
Jim Bogert	Fischer Technology
Michael Bolen	Solar Energy Technologies Office, DOE
Antonio Bouza	Building Technologies Office, DOE
Karen Buechler	ALD NanoSolutions, Inc.
Kim Cierpik	CAS-Navarro Joint Venture
Joe Cresko	Advanced Manufacturing Office, DOE
Arrelaine Dameron	National Renewable Energy Laboratory
Claus Daniel	Oak Ridge National Laboratory
Ralph Dinwiddie	Oak Ridge National Laboratory
Peter Faguy	Vehicle Technologies Office, DOE
Halden Field	PV Measurements, Inc.
Nancy Garland	Fuel Cell Technologies Office, DOE
Dave Gotthold	Pacific Northwest National Laboratory
David Hardy	Advanced Manufacturing Office, DOE
Stephanie Hodge	CAS-Navarro Joint Venture
Cassidy Houchins	SRA
Carlos Jorquera	Boulder Imaging
Dennis Kosisko	Dr. Schenk
Dean Levi	National Renewable Energy Laboratory
Bogdan Lita	Consultant
Daryl Ludlow	Rensselaer Polytechnic Institute
Wil McCarthy	Ravenbrick
Ashutosh Misra	ITN Energy Systems
Nathan Moore	CAS-Navarro Joint Venture
Shaun Onorato	CAS-Navarro Joint Venture
Sergei Ostapenko	Ultrasonic Technologies
Ahmad Pesaran	National Renewable Energy Laboratory
Yarom Polsky	Oak Ridge National Laboratory
Mark Richards	FuelCell Energy, Versa Power Systems
Shriram Santhanagopalan	National Renewable Energy Laboratory
Ron Sinton	Sinton Instruments
Bhushan Sopori	National Renewable Energy Laboratory
Robert Tenent	National Renewable Energy Laboratory
Michael Ulsh	National Renewable Energy Laboratory
Maikel vanHest	National Renewable Energy Laboratory
Ed Vineyard	Oak Ridge National Laboratory
David Wood	Oak Ridge National Laboratory

Appendix C.
Detailed Notes From Breakout Sessions

Breakout 1: QC Challenges

Table C-1. Notes from Breakout 1A—SETO

QC Challenges

Summary:
• Algorithm/automation development (3)
• Sensitivity of sensors/measurement (0)
• Understanding of defects with respect to product performance (Everyone)
• Cost of new technology implementation and return on investment (Everyone)
  ○ Custom tools/degree of customization
  ○ Challenges to justify cost of QC due to size of market
• Education of customer and qualification of new tools (1)
• How do defects affect the expected reliability over decades of expected lifetime (4)
• Resolution of measurement techniques / field of view (3)
• Speed of QC data acquisition (4)
• Moisture, shunts, shorts, electric activity, modeling cause and effect (1)
• Accuracy of actual techniques / PV defect mapping, location, type (3)
 	
Detailed notes:
• Observation thermography for detection of solar cell shunts, shorts
• Building-integrated PV:
  ○ Clear photovoltaics on windows—optical defects significant
  ○ Deviation in color, optical flatness, bubbles, voids
  ○ Inspection visually = hard to take large panel and look; circle defects/characterize types of defects hard work
• Way to automate? Current sensors don't have same sensitivity to optical deviation as human eyes
• Divergence of parallel lines—can be detected by human eye
• Photography fails when trying to detect "warble" defects, whereas to the eye it is very apparent
  ○ Can create artificial situation to detect optical distortion—escape quantification
• Need improved sensitivity of sensors, along with algorithms for the sensors, to detect defects
• How much development/understanding to failure modes is needed? "How good is good enough?"
• Quantify size and type and extent—what defects are "relevant"?
  ○ "Fish eyes" vs. "warble"
    ▪ "Fish eyes" are easy to detect and quantify
    ▪ Off-axis color—coatings or film (not looking to directly but to the side or against different lights)
  ○ How many <u>and</u> what type? What about below vs. above tolerance?
• How hard is it to develop Pareto chart with main types of defects?
  ○ Can it be done in software?
  ○ Big difference between number, type, annoyance -> algorithm (assuming defects can be detected by sensors)
• Ignore defects that have no relevance to the outcome?
  ○ Different technologies have different requirement with different final product purpose
  ○ "X" defect will do "Y" to the system is not always known for each application
• Cosmetic defects vs. major defects that could kill the system
  ○ Incoming inspection is a high request (for end user)

        - Coatings
        - Roll-to-roll Al metal from companies who don’t do a lot of control
        - Breakage in material (edge could propagate)
        - Glass—TV and computer displays with defects will crack if put in a frame
- In-line approaches? 100% inspection system and edges with glass
- How hard is it go to in-line tools PV systems for QC/metrology?
    - Turnkey solution like incoming glass inspections
    - Glass industry already has technology
    - Specific: develop in-house or customized
    - Generic: build on existing industries
    - Specific generally harder to implement and develop
    - Yield improvement and breakage—important for commercial scale
- Difficulties in new R&D metrology into brand new process/material set/industry?
        - Metrology from lab to industry: must be fast, non-contact, low-temp (?)
- Challenges that the customer sees?
    - They don’t understand the in-line metrology equipment or how it works
    - What happens if we don’t use QC?
- Provide service instead of tools? Prepay for consulting services to help instruct the users on the proper use of the in-line metrology equipment?
    - Customers think it is expensive to have engineers on-site; but can they afford not to do QC checks?
- Return on investment—consultant to keep QC, but customers don’t see the cost benefit of having correct QC/metrology

Table C-2. Notes from Breakout 1B—FCTO

QC Challenges

What are the current QC techniques being used?

- Optical (human vision, machine vision)
- X-ray to identify voids in ceramics (SOFC)
- High frequency resistance measurements for catalyst layer
- Machine vision for process control (e.g., registration), but human for final part QC
- X-ray diffraction of powders to screen for size
- Physical dimension measurements of supplied parts
- Thickness uniformity (measured by weight) of anode supported cast tapes

What are current QC challenges?

- Difficult to measure electrochemical properties of SOFC in line (need to bring up to operating temperature)
- Membrane: limited pinhole detection; rely on supplier
- Bipolar plate: detecting micro-cracks, measuring nitride (anti-corrosion) layer thickness, degree of planarity/flatness
    - Leak checking bipolar plate—compress plates, put in water, and pressurize to find crack/leak manually
- Electrodes: thickness and catalyst content
- Gaskets and seals: need better understanding of what properties lead to better performing seals

Comments:

- Dimensional metrology is well understood; thickness, height, dimensions can be measured
- Pinhole length scale is not well understood; isn’t clear what size results in failure
- Need to correlate defects to performance loss or degradation
- Need further development of accelerated testing methods for all components

Table C-3. Notes from Breakout 1C—VTO

QC Challenges

Defect types and criticality for battery electrodes:

Defect	**Source**	**Result**	**Gravity/ Frequency**	**Non-Metrology Remedy**	**Metrology**
Metal	Mixing debris, slitting debris	Short circuit, overheating	Grave/seldom		Optical, thermography
Coating (pinhole, blister, porosity)	Mixing, slurry process	Life	Medium		
Coating thickness	Pump, roll position	Capacity, safety	Low		
Material agglomerates	Slurry	Safety	Medium		
Moisture	Air	Safety	Low/expensive?		
Powder homogeneity	Milling				

Challenges:

- Measurement challenge on one sided electrodes. Are we measuring something that matters??
- Scrap material: we may need to be marking scrap material; at this point no one is doing this yet. We scrap a lot of material due to false positives
- Defects (see summary table above)
- Homogeneity: particle size, distribution, porosity, thickness
- Performance or quality of battery: has this been mapped back to defects?
- Slow formation cycles
- Battery lifetime
- Safety
- Clean room environments

Questions for thought:

- How much can we learn from other technologies?
    - Are there similarities with the AMO side? Are we using the same techniques?
- Should/can we replace beta gauge with something cheaper?
- Is optical a better way to go as this is a more controlled environment?
- How do you represent quality?
- Do we have a tool that can measure porosity or large area?

Table C-4. Notes from Breakout 1D—BTO

QC Challenges

- “Buildings” is a very large topic, with many components
- Material selection in windows is an important part of QC
    - Good at sale of product vs. X years of use (e.g., window gas)
    - What QC is needed with new materials?
- Material choices are often made by manufacturers by spot cost of material
- Flammable refrigerants = smaller heat exchangers in HVAC equipment = different risks
- Compressors: oil-less have tighter tolerances, new testing methods and thresholds are needed
- Everything fails, eventually
- How can materials be tested without destroying them?
- What accelerated aging tests are appropriate?
- Embedded sensors provide real-time data
    - Treat building like a system
- Quality of certifications matter
- Environmental and public pressures on manufacturers to change a material and/or product
- Risk mitigation for manufacturers is very important
- Construction is often a low-tech exercise at erection of structure
- New products require new regulations

Breakout 2: Synergies

Table C-5. Notes from Breakout 2A

Synergies

- Battery electrodes:
    - Electrode deposition is similar to other coating applications
    - In-line X-ray measurement
        - X-ray fluorescence (to measure areal loading) is utilized by a couple of fuel cell companies
        - Not yet established as an in-line measurement for batteries; should be translatable
    - Off-line X-ray
        - Primary Li-ion batteries X-ray to measure electrodes—make adjustment to the foil on the spot?
        - Secondary to the Li-ion people, are not doing that yet
        - Li and cobalt or cobalt-oxides → measured CoO areal weight
        - Transition metal areal weight by XRF
- PV measures electrical characteristic of a cell
    - Are there similarities in electronic or electrochemical measurements for fuel cell or battery materials?
    - For PV, electrical measurements are made of actual device voltage once connections are made on cell
        - P-n junction is deposited during coating
    - For fuel cell and battery, no *ex situ* voltage exists, so want to make measurements of electrical properties, not actual voltage
    - Look at electronic characteristics (and properties) of silicon wafers by photoluminescence (or some other method)
        - Thin films need junction to measure voltage
        - Measure conductivity of substrate; lateral sheet resistance
        - Radio wave? Electronic impedance? Capacitance? Eddy current? Excitation for

battery or fuel cell

- Crystalline materials—determined by grain properties
- Non-contact measurements—some have constraints on thickness
    - Spot fusing
    - Electronic properties/permeability
    - Expensive transducers in the past
    - Microwave deflection? Look at absorption and transmission then back out the dielectric current?
- Some defects accelerate failure (i.e., in fuel cells)
- Maybe a better format for identifying synergies would be to have an exchange of technical methodology of metrology examples. Maybe focus on smaller technical meetings (or webinars) to discuss methodology and how one new device/process could be adopted by others either in their field or across technologies
- Cross-cutting FOAs? Who can justify the need for DOE to engage in these cross-cutting activities?
- Theme of national lab summit or showcase
    - For wider participation try webinars or national conference
    - Have a QC side-event at Annual Merit Reviews?
    - Solar office incubators/start-ups provide a 1-minute elevator pitch at a webinar with venture capitalists as well. Have a list of attendees for cheap, quick networking
    - Virtual showcase
    - Learn high-level what other programs have done, mistakes made
- Low-cost IR camera techniques
    - Other uses?
    - Resolution?
    - Similar spatial resolution or more expensive
    - No built-in screens could be a detraction of the cheaper cameras. Depends on the particular needs
- Overlap measure of texture? Scattering? Could this PV technique be developed with fuel cells, batteries?
    - What would the process control outputs look like?
- Desired outcomes:
    - Collaboration
    - Transfer technologies to industry
- Why aren’t manufacturers using in-line QC techniques? QC techniques are becoming useful but manufacturers say volumes are not high enough yet to justify the time or cost of testing and implementing them
- Manufacturer specifications versus in-line checks. Who is responsible for making QC checks? Some rely on manufacturers to provide good materials
- Good area for national labs to collaborate and work on these issues. They can address issues faced by manufacturers before manufacturers need to put processes in place
    - Some of these technologies have not been scaled up yet. Manufacturers have not been able to look at process development, and this is where labs can step in
- Manufacturing customization—make same tool to work with fuel cells, PV, etc.?
    - Thickness, surface inspection
    - But, adds cost
- University challenge of staying relevant. Struggle to work with industry due to intellectual property constraints
- Topography—important texturing step in PV
    - Morphology: measure for fuel cells and PV; look into battery electrodes?
- Thickness and porosity
    - Similar thickness needs across technology platforms
    - But, PV and fuel cells/batteries have different thicknesses (nanometer versus micrometer)
- Relationship between dielectric properties and porosity?

    - Manufacturer specs can provide some information
    - Measure transport limits
    - Not a real study of pore size distribution
    - Coating for battery not the same as for glass
- How and when in the process should these measurements be taken?
- Conductive layers/in-plane resistance (but not a specific layer to measure)
- Pinholes in batteries and fuel cells
    - In the coating and separator
    - Tier 1—pinholes in the battery electrode is of interest
    - Tier 2—battery separator manufacturer should take care of their pinholes
    - Fuel cells with pinholes in membranes—shorts can occur
- For batteries, transition metal areal weights are measurable but total metal areal weights are difficult to measure
- For PV, conductivity is a critical as soon as p-n junction is formed
- Idea for DOE to provide a forum for researchers to see others' monthly reports
- Maybe we can carve out a session at this year's AMR? What about doing a national lab webinar series?
- Each office needs to better understand the impact of defects on performance and durability to correlate those defects to not only reduced scrap/higher yield, but also to some cost benefit

Table C-6. Notes from Breakout 2B

Synergies

- QC methods need to be non-invasive, non-contact, fast
- Performance correlation is not clear
    - Does defect affect performance later or result in immediate problem?
- Common problems are difficult to define due to different applications
- Thin films, coatings—common mechanical problems
- Software—analysis of results, interface, and algorithms for classification
- Cost of defect detection has to be lower than cost of defect—perceived return on investment
- In some cases, techniques are available but do not know how to apply it to the different applications

Defects which cannot be measured at this time:

- Physical vapor deposition of glass onto plastics—10^{-5} per day moisture permeation needed—defects on the scale of 10 nm cannot be measured
    - Application maybe of 5 micro-inch cast to such problems? Scale?
- Cost might inhibit existing technique to be used and applied

Commonality between applications:

- Pinholes, micro cracks
    - Might have different size depending on application
- Compositional homogeneity
- Reliability
- Accuracy, false positive avoidance
- Software
    - Maturity at which software gets implemented varies a lot
- Process control to reduce need for metrology—understand impact
    - Statistical quality control is required to perform metrology effectively
- Where should we focus our thought? 0-5 years of horizon for bringing technology to market or changing application for technology

Table C-7. Notes from Breakout 2C

Synergies

- Although there are synergies across the technologies, they are application specific. For instance a type of defect might be catastrophic to one technology, but not to another
- Defect examples: pinholes, thickness, X-ray fluorescence, metal fragments, sheet resistance
- Summary of synergies:
    - Materials need to work together for long term life
    - Quality of deposit, registration
    - Techniques for leakage and pinholes
    - Thickness
    - Not enough understanding of long term aging effects at this time for products
    - Current technologies need to be better to be able to use across technologies
    - Defects that affect lifespan are different than defects that affect whether it is usable at all
- Defect at point of manufacturing important, but defect shortly thereafter is more important for environmental impact
    - Testing currently is to put into field and see what happens
- Defects are a matter of scale among the technologies
    - A pinhole may be bad, but may not be a killer defect. For example, a pinhole is limited in how it will affect solar technologies; for battery it could kill the entire battery
- Some defects can be detected by the eye, but not a camera
    - Examples would be optical flatness and off axis color
    - Inspection systems that approximate the acuity of human vision system would probably solve problems in the industry across all technologies
- How do you measure the quality of white light, i.e. how well a light source used for PV testing mimics the sun in terms of spectrum, spatial uniformity at the device-under-test, and reproducibility?
- Need for DOE support of metrology development

Breakout 3: Remaining R&D Needs and Recommendations

Table C-8. Notes from Breakout 3A

Remaining R&D Needs and Recommendations

R&D needs for QC:

- Are there any solutions to address the issue with pinholes/defects that do not seem to be an issue at the time the device is manufactured but appear during operation?
- Idea that a lot, if not all, structures are multi-layer structures (thin films, membranes, coatings): is there a need to evaluate layer-to-layer binding/adhesion?
    - Questions about delamination and discontinuations in the bond
    - Interface states affected by the bonding used
    - Interfacial bonding is very important for many technologies
    - Delamination of contacts is an issue
    - In fuel cells, electrode-to-electrolyte adhesion is very important
- Delamination—sometimes a manufacturing issue, but often happens during operation
- Batteries (Li-ion) and fuel cells
    - Adhesion is important when putting down electrodes
    - Ceramic to metal joints can be unstable under load in SOFC
    - Binder (polymer) to metal substrate not easy to accomplish; high contact resistance and ohmic losses
    - Polymer electrolyte membrane (PEM) fuel cells have same polymer in membrane and catalyst layer so adhesion during fabrication is not as much of an issue, although

    delamination can occur as a result of operation

- Currently no good way to do in-line testing for delamination
    - Destructive tests or energies of adhesion/contact angle analyses
- Search for delaminations by acoustic tests? (not roll-to-roll)
    - Thickness limitations with ultrasonics?
    - 80 microns for silicon wafers
    - Must use the appropriate frequency: higher frequency for thinner materials
- Electronic technique (mm wave energies)—quality of the interface
    - Tune frequency to thickness?
    - Each sample has its own resonance curve; adhesion defects will show modified resonance
- Interface—electro-migration happens during operation then have to dissect part to look for defects
- Voids are easier to detect than bond strength, but would like to have *both* voids and strength
    - Should do cost benefit analysis of doing both
- Polycrystalline coatings
    - Adhesion of crystals
    - Coating grain size needs to be consistent when using multiple coatings
    - Grain size mismatch could lead to void/defects and granularity
- Previous development of in-process measurement that uses high frequency impedance for lamination quality of PEM cell

Questions on next steps:

- What will DOE do with these ideas?
    - Support for cross-office activities
    - Hard to fund in an AOP-funded manner; brainstorm ways to work together
    - Cross-cutting FOAs? Make teaming necessary?
    - What if $25 million cross-cut FOA in future? Require part two of workshops with other planning steps to add more technical principal investigator presentations
    - Europe labs get funding with large collaborative scopes. Find lessons-learned from Europe that could be applied in the United States
- SBIR program focuses on some of these issues.
    - SBIR ensures buy-in from manufacturers and tech transfer to industry
- How to evaluate cost benefit of QC?
    - Use manufacturing cost analysis? Get QC into models
    - Yield numbers not provided by companies
    - Use models such as the ones produced by DTI (for fuel cells)?
    - Argonne collects data on Li-ion batteries (VTO research funds)
    - Can use competitiveness analyses such as recent NREL work for solar PV?
    - What is the cost of *not* having QC?
        - Real processes never have 100% yield, but the manufacturers never give out the exact yield—business sensitive
        - Example of fuel cell stack yield effect on stack cost: if QC measures are not taken, the % of bad product increases costs
- Example of thermography/IR cameras resulting in record dollar savings in power plants
    - Costs to show return on investment of having thermography inspection
    - Leads to using same cameras for other applications that help reduce the cost of the manufacturing plant
    - Thermography now required for electric plants (save lives and money)
- Nanotech center in New York has a pilot fabrication (FAB) lab for data collection. Set up more of these fab labs to investigate the use of QC/metrology in manufacturing setting
- Intellectual property issues inhibit collaborations and effective teaming

Table C-9. Notes from Breakout 3B

Remaining R&D Needs and Recommendations

R&D needs for QC:

- Intelligent process control needs
    - Example: CIGS—DARPA, consortium with nine companies, vapor phase manufacturing, how to control four element deposition correctly
    - Run machines dynamically for 30 or 40 hours or more
    - CIGS roadmap from Sematech
- Offline techniques might be available—how to bring them online
- Understand what needs to be measured
    - Solar might have an understanding; batteries are lagging
    - Difference in needs between surface and bulk
    - Classification for focusing effort needed
- Six sigma—what are the things we can measure and how well do we need to measure them?
- BTO has less focus on QC or metrology as a subject even though it might be used or needed for a product
    - Focus is on final product
    - Webinars might be good to keep community informed
- Catalogues needed of QC techniques available
- Translation of laboratory techniques to production sites difficult—understanding lacking on the laboratory side about manufacturing needs
- Technology driven partnerships might be inhibited by competitive considerations in expert groups and partnerships

Focus of program needs to be on manufacturer:

- Manufacturer does not want metrology on their line
- Only accepted if there is an impact on their productivity and bottom line (i.e., if manufacturer has a problem and it hurts)

Needs that are not being met by commercially available technology:

- Might be available but too expensive
- Example: time-resolved photoluminescence
- Current techniques signal problem but do not give guidance for correction

Needs to be addressed by EERE:

- How to improve collaboration with manufacturer? Understand manufacturers' challenges in a competitive environment?
- Correlation of inspection to process control via classification through data analysis software

General considerations:

- Expertise in manufacturing is critically low—manufacturing R&D and scientists are not with companies anymore
- Manufacturing brain drain—switch to services organization
- Problem of profit center profitability from day one in United States vs. global long term vision in Eastern Asia
- Loss of workers—everybody wants to be a manager and accountant

Table C-10. Notes from Breakout 3C

Remaining R&D Needs and Recommendations

R&D needs for QC:

- Vision systems that replicate the processing ability of the human brain and eye
    - The human eye is a great screening tool, but people get tired and make mistakes
    - Ability to quickly differentiate between features like the human eye can, but by camera
    - Hardware improvements and/or software improvements will be needed to achieve this
    - It would be helpful for the vision system to detect things smaller than the diffraction limit of what the human eye can detect
- The ability to process and detect defects over large scales (looking at small things over large areas or detecting one defect over a large area)
    - There is no current method to look at physical features in intermediate scales over large surface areas
    - We have good optical techniques (e.g., microscopic) that work at short enough wavelengths for small areas, but it is very hard to measure defects (around 0.5 micron) over square meters of materials
- Create a large database and data mining effort from which the data can be analyzed to determine what is statistically different
    - Use big data mining techniques to accumulate a lot of data and help apply this to the problem
    - Oversample and then try to look back at the data (see why samples were degrading or further analysis… etc.).
    - Go back in time and see what is statistically significant. Large data set may show trends, but we need the data set first
    - Three problems (initial data collection, storage of data, and going back to look at the data)
- Define what a real versus perceived defect is
    - What is a variation vs. an actual defect?
    - Try to identify what the actual characteristics are of a defect
    - Need measurement techniques that can identify defects that are relevant to the specific application
    - We throw out a lot of material from being too conservative with perceived defects
- Process integration issues with equipment and the need for standardization (manufacturers need to be working hand in hand with the QC vendors)
    - Every application of sensors is different from the equipment vendor perspective
    - Would be good to have standardization. A lot of custom applications for standard equipment
    - Generally use offline analysis to show issues with equipment (labs, etc.)
    - A lot of issues come up with data collection and control systems
- Generate a table that summarizes QC techniques and their capabilities (defect size, line speeds, etc.)
    - Make a table for renewable energy technologies for defect definition, irregularities, how to detect them and different synergies
    - A table showing QC techniques used and how they are applied and then have equipment vendors and others come in and fill gaps for areas of QC that aren't well defined
    - A speed-dating type activity where QC equipment vendors could help identify ways to look at the defects identified by manufacturers
- More customizable products = less tolerances
    - Continue to improve yield by reducing cost by building in tolerance variations
    - Need a good feel for what variables matter
    - Issues surrounding poor build quality products. Whether to ramp up QC and inspection or get liability insurance (due to bad design)

Questions on next steps:

- What could DOE do to help?
    - Metrology webinar series under the AMO umbrella so that it can cross multiple technologies
        - If the topic is specific to “solar” as opposed to “metrology” then people may not tune in if they are in the fuel cell industry
        - Make sure to add contact information for the presenter and organization for people to follow up with after the webinar
    - DOE could get lots of end users at meetings where QC options are presented
    - Finding a way of bringing people that “cross pollinate” across multiple industries to attend meetings and workshops
    - Meetings to minimize travel. Hold events at well attended events. Invite people to manufacturing meetings or conventions
    - More breakout sessions
- Improve buy-in from and tech transfer to industry
    - Taking technology from the laboratories and demonstrating it at real companies for a brief period
    - Helping companies identify and then resolve issues; convince companies of the payoff of the technology
    - Could make companies hesitant because of QC issues that get sampled—manufacturers do not necessarily want their defects or manufacturing issues to be known by the outside