NCMC Workshop Report
NCMC-9: Combinatorial Methods for Nanostructured Materials

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NCMC Workshop Report
NCMC-9: Combinatorial Methods for Nanostructured Materials

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U.S. Department of Commerce
Carlos M. Gutierrez, Secretary

Technology Administration
Robert Cresanti, Under Secretary of Commerce for Technology

National Institute of Standards and Technology
William Jeffrey, Director
1) Introduction and NCMC-9 Workshop Goals

Traditionally, industrial producers of semiconductors, microelectronics, catalysts, and biotechnology have been considered the commercial stakeholders of nanotechnology and nanostructured materials. However, recent years have seen a new focus on nanostructured materials by industries involved in a growing set of commercial goods, including personal care products and cosmetics, advanced coatings and paints, automotive components, structural plastics, and adhesives. A part of this new focus might be considered a redefinition of terms, driven by an exciting, well publicized technological theme. Nevertheless, the fact is that the materials and chemical industries are striving to incorporate nanostructured materials into more of their products, and this is because nanostructured components promise (and sometimes realize) radically improved properties and performance. In this respect, an explicit connection of this expanded set of goods with nanotechnology is essential, since it provides the appropriate conceptual, scientific, and metrology frameworks for the design, characterization, and manufacture of these innovative products.

NCMC-9: Combinatorial Methods for Nanostructured Materials was a forum to examine the application and development of nanostructured materials by industry, with a focus on polymeric products, such as films, coatings and paints, adhesives, and personal care products. Over two days, the Workshop brought together more than 35 industrial representatives, primarily from NCMC member organizations; three academic representatives; and more than 30 NIST staff (see Appendix B, NCMC-9 Participant List).

In conjunction with NIST’s mission of advancing measurement technologies that stimulate industry innovation, and the specific NCMC mission of developing combinatorial and high-throughput measurement methods for materials research, NCMC-9 had two main goals:

1. To examine key measurement needs in industrial development and application of nanostructured polymer materials; and
2. To arrive at priorities for the development of combinatorial and high-throughput measurement methods for nanostructured polymer materials.

2) NCMC-9 Technical Program

The NCMC-9 Technical Program (see Appendix A, NCMC-9 Agenda) provided scientific context for the Workshop goals, and introduced ideas germane to a Discussion Session (see following Sections 3 and 4). Opening remarks from Richard Kayser, Director of the NIST Materials
Science and Engineering Laboratory (MSEL), outlined the Workshop’s relationship to the NIST (www.nist.gov) and MSEL (www.msel.nist.gov) missions of advancing measurement science and technology. Afterwards, NCMC Director Michael Fasolka discussed the goals and structure of NCMC-9.

A set of plenary lectures illuminated scientific concepts in polymeric nanomaterials and outlined measurement methods for characterizing these systems. To kick-off the technical symposium, Fiona Case, of Case Scientific, lectured on Applications of Nanotechnology in Soft Materials: Cosmetics, Foods, Personal Care, Pharmaceutical and Oil Industries. This presentation gave an overview of how nanostructured polymer surfactants, micelles, colloids, and emulsions are used in industrial products, and the principles of designing such systems for commercial applications. In complement, Prof. Daniel Savin, of the University of Vermont, lectured on a range of light scattering techniques, which are particularly useful for measuring the structure and dynamics of fluid polymer nanomaterials. Prof. Savin’s presentation detailed the measurement fundamentals of these methods and demonstrated their application via a discussion of his current research in pH and temperature responsive block copolymer systems. In addition, a presentation by Peter Harris, of Veeco Metrology Inc., gave an overview of Scanned Probe Microscopy (SPM) techniques for characterizing polymer materials. Mr. Harris’ lecture detailed several emerging SPM methods for imaging soft nanostructures.

Another set of invited lectures demonstrated the application of combinatorial and high-throughput (C&HT) methods to nanomaterials discovery and development. Karl Maurer, of CombiMatrix Inc., described a micro-array platform for the rapid synthesis and screening of small biomolecules. Mr. Maurer’s presentation exemplified the wide success C&HT approaches have had in the biotechnology sector. Returning to the Workshop’s focus on polymers, Prof. Sergiy Minko, of Clarkson University, described approaches for fabricating combinatorial libraries of functional surface-grafted polymers. As Prof. Minko demonstrated, such libraries enable high-throughput assessment of interfacial interactions, which are critical to the engineering of thin nanomaterials and nanoparticles. In the next presentation, Prof. Thomas Epps, of the University of Delaware, lectured on gradient combinatorial methods for measuring the effect of surface and interfacial interactions on the self-assembly of network forming block copolymer films. Prof. Epps’ presentation was an overview of his post-doctoral work with the NCMC, and included new gradient methods for varying governing interfacial interactions at both the substrate and the free surface of films.

A number of presentations reported on NCMC and NIST Polymers Division research. Generally, these talks focused on new combinatorial and high-throughput measurement methods for nanostructured polymer materials. Thomas Chastek, NCMC, presented a new microfluidic device for dynamic light scattering measurements of fluid borne nanoparticles and nanostructures. This “lab on a chip” instrument enables rapid, continuous assessment of nanostructure size, while using very small sample volumes – two prerequisites for a viable C&HT measurement platform.

Chris Stafford, NCMC, discussed new gradient combinatorial approaches for measuring mold adhesion in nanoimprint lithography. Such methods will enable more rapid development of processing routes and mold release agents for this emerging nanomanufacturing route. In addition, Dr. Stafford presented a recently published work concerning the NCMC Flow Coater device. This instrument fabricates continuous gradient libraries of polymer film thickness, which provide a foundation for rapid, thorough measures of the effect of thickness on film materials and device performance.

Michael Fasolka, NCMC Director, presented a new NCMC technique for the high-throughput preparation of samples for transmission electron microscopy (TEM) analysis. This method harnesses a “peel off” approach to simultaneously harvest many samples from a combinatorial film library, and suggests a first step towards C&HT TEM of polymer materials.
Chang Xu, NCMC, lectured on his work in developing micropatterned gradient libraries of grafted polymers. When complete, these combinatorial surfaces will enable high-throughput assessment of film properties and reference substrates for advanced SPM methods and other microscopies.

Alamgir Karim, Group Leader of the Polymers Division’s new Nanostructured Materials Group, gave an overview of research and projects in nanoimprint lithography, defect metrology, and thermal management in active nanodevices. The new Group’s efforts include the development and application of C&HT methods, as illustrated by Brian Berry, NCMC, who presented his measurements of domain orientation in block copolymer thin films.

The final set of talks highlighted new interactions the NCMC is building with other institutions. The first presentation was given by InsightFaraday, which joined the NCMC this year. Steve Fletcher, Director of InsightFaraday Partnership, gave an overview of the goals and structure of the institution, which serves UK industries interested in C&HT methods much in the same way that the NCMC works with US firms. In addition, InsightFaraday associate Jawwad Darr, of Queen Mary University, London, outlined his work in the combinatorial synthesis of nanoparticles useful in applications such as fuel cells, photocatalysts, and sunscreens.

Celesta Fong, CSIRO (Commonwealth Scientific and Industrial Research Organisation, Australia), summarized molecular technologies and health research at CSIRO, and described her work examining surfactant nano- and meso-phases. Dr. Fong also introduced a current NIST-CSIRO collaboration aimed at developing high-throughput measurements for surfactant phase behavior.

Dan Cutbirth, of nScrypt Inc., described a prototype fluid deposition system developed recently through a NIST Small Business Innovation Research (SBIR) Grant. Capable of blending and depositing small quantities of up to three input fluids, including viscous polymer solutions and nanoparticle laden liquids, the nScrypt instrument suggests a promising route for fabricating combinatorial libraries of nanostructured polymer materials.

NCMC-9 concluded with a series of tours and demonstrations in the NCMC laboratory facilities. Demonstration stations included the NIST flow coater, the nScrypt deposition system prototype, deposition of epoxy libraries, microfluidic dynamic light scattering, static light scattering, buckling measurements of modulus, the NIST CD-SAXS (Critical Dimension Small Angle X-Ray Scattering) instrument, and gradient libraries of organic electronics materials.

3) NCMC-9 Discussion Session Goals and Guiding Questions

A central part of NCMC-9 was a Discussion Session, which was structured to:

- Examine key measurement needs in industrial development and application of nanostructured materials; and
- Determine priorities for the development of combinatorial and high-throughput measurement methods for nanostructured materials.

This Discussion was moderated by Michael Fasolka, Alamgir Karim, and Kathryn Beers, and flipchart notes were compiled by Carol Laumeier, NCMC Outreach Coordinator. To guide the Discussion, the major themes and specific questions to be considered were distributed to participants in advance of the Workshop, and are outlined as follows:

Theme I: Scope of Metrology Needs

- In your company, what aspects of nanomaterials development or application require new or improved measurement methods? Examples to consider:
  - Basic R&D: design and testing of custom fabricated materials
  - Evaluation of raw materials from internal or external suppliers
Formulation of products with nanostructured components
- Performance of new products
- Testing of manufacturing and/or processing routes
- Quality control and/or failure

- Of these aspects, where would new combinatorial and high-throughput measurement capabilities be most important?

Theme II: Key Materials Properties

- What new materials’ property information is critical for the development or application of nanomaterials in your company?
- Of these properties, for which is it critical that new combinatorial/high-throughput measurement methods be developed or improved?

Theme III: Key Test Methods

- For nanomaterials analysis, are existing test methods and strategies adequate?
- What measurement tools and instruments do you currently find most useful for nanomaterials testing and analysis?
- Of the tools you use for nanomaterials analysis, which one would be most valuable if it were developed into a combinatorial/high-throughput measurement technique?
- What entirely new test needs to be developed? What entirely new combi/high-throughput test needs to be developed?

4) Discussion Results and Workshop Conclusions

The Discussion Session, participant responses to presentations, and informal interactions over the two days of the NCMC-9 Workshop yielded several points of consensus relating to the Workshop goals. These conclusions, outlined as follows, are organized in terms of the guiding Discussion themes and questions.

Workshop Goal 1: Discuss key measurement needs in the industrial development and application of nanostructured polymer materials.

I. Scope of Metrology Needs

With respect to polymer-based nanomaterials, industrial Workshop participants noted that improved measurement methods are required throughout their R&D, manufacturing, and quality control processes. However, this industrial community emphasized that nanostructured materials are generally part of formulated products, such as personal care goods, cosmetics, coatings, polymer blends and composites, and adhesives. Formulated products are very complex; they typically contain a large number of components, and have structure and properties that are highly sensitive to composition and processing routes. In formulations, nanostructured materials can be:

1) Specialty additives. Often, polymer or inorganic nanoparticles are added to a formulation to improve performance. In this case, measurements of raw nanomaterial additives are important, as stocks are often from outside suppliers, or fabricated in-house using new synthesis routes. Moreover, once added to a formulation, measurements of nanoparticle distribution are critical.

2) The goal of the formulation. In many formulations, organic molecular components such as surfactants and block copolymers are added with the goal of creating nanostructured products. In these cases, measurements of the formation and structure of nanoscale emulsions, micelles, colloids, and blends in formulations are essential.
In addition, in both of these cases, industry requires measurement approaches that illuminate relationships among the nanoscale structure of products, processing routes, and their performance properties. To accommodate the large number of experiments needed to determine these multivariate relationships, industry needs measurement approaches that are both rapid and thorough.

II. Key Materials Properties

Industry Workshop participants identified three types of property information as critical for the development or application of polymer nanomaterials:

1) Nanostructure Morphology. Measurements of the morphology of nanostructured components are needed to gauge the success of formulation designs or processing routes, and to build structure-property relationships.
   • In the case of nanoparticle additives, measurements of the size, size distribution, and shape/shape distribution of raw materials are necessary. Particular concerns of the quality of carbon nanotube stocks were noted. Moreover, after these particles are added to a polymer formulation, measurements of the particle dispersion, flocculation, and stability are essential.
   • Similar verification measurements are required in the case of polymer fluid and solid formulations. Here, assessment of the size-scale and morphology of emulsions, micelles, and other polymer nanophases are critical.

2) Nanostructure Chemistry/Composition. As with morphology, information about the chemistry and/or composition of nanostructures is needed to verify formulated products:
   • For nanoparticle additives, industry requires chemical measurements to verify the quality of particle stocks, or the routes they use to functionalize particles for more effective deployment in formulations, e.g., proper exfoliation of clay nanoparticles. In this latter case, surface-sensitive chemical information is essential, as functionalization layers can be less than 1 nm thick.
   • For nanostructured formulations, information on the spatial distribution of organic modifiers, such as surfactants and block copolymers, are necessary. In this respect, chemical analyses amenable to organic materials and with nano-scale spatial resolution are needed.

3) Performance Properties. Ultimately, performance properties determine the success of nanostructured products. Workshop participants identified two critical performance measures:
   • Rheological Properties. Many participants identified rheological properties (viscosity, complex viscosity, and flow characteristics) as the most important set of information they require about nanostructured polymer products. For nanostructured fluids and melts, rheological information allows industry to optimize processing parameters. In addition, rheology data enables prediction of a variety of related, but often more complex, performance properties; for example, spreadability and film forming in coatings; and texture and “skin feel” in personal care products and cosmetics. Moreover, in industry, rheological measurements are an indirect, but widespread means to verify the morphology of fluid formulations. In all of these cases, complex rheological information (i.e. storage and loss data) is needed over a large range of shear rates and flow conditions.
   • Mechanical Properties. For polymer materials such as coatings, adhesives, and structural plastics, nanoparticle additives or a nanostructured formulation can give enhanced or tailored mechanical properties. Accordingly, measurements of modulus that verify product mechanical property goals are important. Moreover, some industrial parties highlighted the need for in-situ measurements of the mechanical properties of individual nanoparticles, single nanodomains, or ultra-thin film systems. This, of course, requires more sophisticated, more sensitive measurement techniques with nano-scale resolution.

III. Key Test Methods

Workshop participants identified five key measurement methods they currently use for characterization of nanostructured polymer products:
1) **Electron Microscopy (EM).** Transmission Electron Microscopy (TEM) is the primary tool industry uses to measure the morphology of nanostructures in solid products. TEM provides information on the size and shape of raw nanoparticle stocks, the internal structure of these materials, and the distribution (dispersion, flocculation, exfoliation, etc.) of nanoparticles deployed in polymer formulations. TEM also provides measurements of the morphology of polymer nanophases, e.g., micelles, emulsions, and microphase separated block copolymers. However, as noted below, TEM relies on slow, meticulous preparation of ultrathin samples, and is only appropriate for solid specimens. To a lesser extent, Field-Emission Scanning Electron Microscopy (FESEM) is also used for these purposes, and similar challenges to sample preparation are apparent. Generally, nanoscale chemical information provided by some EM instruments (e.g., via Energy Dispersive Spectrometry) are not applicable to polymer specimens.

2) **Scanning Probe Microscopy (SPM).** A basic SPM, i.e., Atomic Force Microscopy (AFM), is employed widely by industry to assess the morphology of nanostructures in solid polymer materials. AFM can be easier to use than EM, and it is more amenable to automation. However, for of bulk specimens, SPM analysis requires production of a flat, smooth surface, which like EM sample preparation, can be slow and difficult. Advanced SPM techniques, such as Chemical Force Microscopy and Atomic Force Acoustic Microscopy, which promise nanoscale assessment of chemical and mechanical and other properties, generated considerable interest from industrial participants. However, since they generally provide unproven, qualitative data, these new methods are rarely used in industry at this time. Nanoindentation is highly regarded for its quantitative measurements of local mechanical properties in inorganic materials, but this technique tends to be unreliable for soft polymer substances.

3) **Scattering Methods.** Dynamic and static light scattering are ubiquitous tools in industry for basic particle and domain sizing in nanostructured polymer products. To a lesser extent, x-ray scattering techniques are used to determine the internal structure of inorganic nanoparticle stocks and the distribution of molecular species in organic formulations. For both light and x-ray scattering measurements, industry generally employs traditional instrumentation, which is geared for single specimens. Neutron scattering is recognized by industry as highly powerful for measuring nanostructures in polymer materials. However, industrial access to these measurements is limited.

4) **Spectroscopy Methods.** Basic Infra-red, Raman, and UV-vis spectroscopies are used to measure average compositions of polymer formulations and nanoparticle stocks. To a lesser extent, industry employs these, and more surface-sensitive techniques such as x-ray photoelectron spectroscopy, to assess nanoparticle functionalization. As with scattering methods, industry generally employs traditional spectroscopy instruments, which are aligned toward single specimens.

5) **Rheometry.** As previously noted, complex rheology information is highly valued by industry for gauging the performance, and often morphology, of nanostructured polymer products. To gain this information, industry researchers typically employ traditional rotational rheometers. These instruments provide the shear rate range necessary for product evaluation, but are severely limited in their capacity to accommodate the large numbers of formulations they wish to measure.

**Workshop Goal 2:** *Arrive at priorities for the development of combinatorial and high-throughput measurement methods for nanostructured polymer materials.*

For the rapid development of innovative nanostructured materials, industrial researchers are faced with two major problems:
First, industry is challenged by the large, complex variable spaces formulated nanomaterials present, and thus the huge number of measurements required to discover and optimize these new products. Of course, in many companies, a degree of combinatorial and high-throughput (C&HT) infrastructure is already in place for traditional formulated products. However, this instrumentation often is not suitable for C&HT analysis of the new set of nanostructured polymer products considered in this Workshop. Indeed, this challenge mirrors the situation of perhaps 5- to-7 years ago, when the materials and chemicals industries started to adapt C&HT approaches geared for biotechnology and genomics to formulations problems relating to traditional coatings and adhesives.

The second problem involves the methods industry uses to measure the morphology and performance of nanostructured products. As discussed previously, industry currently relies on a number of key measurements and instruments (e.g., EM) to conduct this analysis. While these methods generally provide appropriate data, instruments are aligned most often with traditional, one-at-a-time experimental paradigms that are not commensurate with the C&HT approaches industry strives to adopt for nanostructured systems.

With respect to these broad challenges, industry Workshop participants identified five priority areas for the development of C&HT measurement methods for nanostructured polymer materials. For each of these areas, existing or possible NIST/NCMC responses to the problem are outlined.

1) **Library Preparation Methods.** Advancement of C&HT measurement methods for nanostructured materials hinges on the ability to produce appropriate specimen libraries. With respect to this primary barrier, Workshop participants emphasized the following needs:
   - Routes for preparing multivariate libraries that systematically vary the formulative and processing parameters relevant to nanostructured materials. These libraries should vary multicomponent composition with precision that reflects the sensitivity nanostructured materials have for this parameter. Means for processing include temperature, mixing, and order of component addition. Automated preparation approaches need to effectively handle and combine both fluids and solids, accommodate particle-laden materials, and higher viscosity liquids. Significant reliability testing of such systems needs to ensure that library elements are comparable to single-specimens prepared by traditional means.
   - Specimen libraries must be compatible with analysis by key measurement tools such as EM, SPM, scattering, spectroscopy and rheometry. Specific issues related to each of these techniques are noted in the following Points 2-5.

   **Response:** The NCMC staff has prepared three related Measurement Need Statements, entitled Nanomanufactured Components in Complex Fluids, Advanced Specialty Materials, and Adhesion and Performance of Underfill Materials for the U.S. Measurement System (USMS, www.usms.nist.gov). These statements will be included in the USMS measurement needs assessment, which is to be published later in 2006. NCMC has extensive experience in library design, and in the preparation of libraries for new types of materials. Indeed, current NCMC work in adhesives and microfluidics technologies is either suited for nanostructured polymer products, or could be modified to accommodate these materials.

2) **Specimen Libraries for EM and Basic SPM.** The key measurement tool for nanostructured polymer identified by industry participants was EM, particularly TEM. Currently the preparation of suitable specimens is the bottleneck process in TEM analysis, and the major barrier to the development of the higher-throughput EM measurements needed to accelerate testing of nanostructured formulations. The ≈50 nm thick polymer samples needed for TEM are generally prepared using cryogenic ultramicrotomy, which is very slow and difficult. Preparation of specimens for SPM or FESEM can be equally slow if ultramicrotomy is needed to create flat sample surfaces for analysis. Alternative methods to generate arrays of TEM or SPM samples, drawn from combinatorial libraries, would greatly speed the analysis of potential new products.
Response: The NCMC-9 technical presentations gave preliminary data on a new NCMC method for the high-throughput preparation of TEM specimens. This work is currently in progress. In addition, at NCMC-6, a NCMC Focus Project was proposed for the development of HT methods for SPM or FESEM analysis. Contact Michael Fasolka at mfasolka@nist.gov for more information on this research.

3) Scattering and Spectroscopy Methods. While these are used widely by industry for nanomaterials analysis, library fabrication and manipulation barriers hinder their full realization as C&HT measurement tools. In particular, industry requires:

- New apparatus designs that enable delivery of rapid series of fluid specimens across scattering or spectroscopic source beams. In this respect, designs that harness milli- and micro-fluidic technologies are of great interest.
- Instrument designs that enable automated scattering and spectroscopic measurements of combinatorial specimen arrays. There is a general paucity of these designs among commercial light-scattering and x-ray scattering instrumentation. In addition, industrial participants stressed that it would be extremely useful if basic automation capabilities were added to the advanced neutron and x-ray scattering equipment found in the National Laboratories. Basic automation tools would allow industry (indeed all users) to analyze combinatorial libraries at these facilities, and thus increase the number of measurements they are able to complete in an experimental session.
- Guidance on conducting scattering and spectroscopic measurements on small specimen volumes, which are common to combinatorial libraries.

Response: NCMC is currently working towards the integration of both scattering and spectroscopic measurements with microfluidic device technologies. Indeed, a presentation at NCMC-9 demonstrated a new microfluidic device for performing dynamic light scattering measurements on nanostructured fluids. Contact Kathryn Beers (kathryn.beers@nist.gov) for more information about this work. In addition, the NCMC will continue to work with industry participants towards a series of recommendations regarding automated array analysis to be distributed to National Laboratory user facilities, including the NIST Center for Neutron Research. A USMS measurement need statement can be prepared in this area.

4) Rheology Methods. To accelerate the analysis of nanostructured polymer formulations of industry, new rheometry instrumentation and designs are needed. For C&HT measurements, rheometers need to:

- Provide complex rheology information over a large range of shear rates.
- Conduct parallel measurements of many specimens simultaneously, or conduct serial measurements in a rapid, automated manner.
- Measure the small liquid volumes inherent to most C&HT methods.

In addition, some Workshop participants expressed interest in simultaneous measurement of both rheometry and scattering.

Response: NCMC has published designs for a complex rheometer that conducts parallel measurements of multiple specimens (Walls et al, Measurement Science and Technology 16: 137-143 (2005). Contact Kathryn Beers (kathryn.beers@nist.gov) for more information about this work. A USMS measurement need statement can be prepared in this area. Moreover, this appears to be a prime area for a future NCMC Focus Project.

5) Advanced SPM techniques. SPM instrumentation is amenable to automation, making it viable for inclusion in a C&HT measurement workflow. However, as discussed previously, industry only uses basic SPM, since more advanced modes are unproven and not quantitative. Development of quantitative advanced SPM measurements, e.g., Chemical Force Microscopy, or Atomic Force Acoustic Microscopy, would allow industry to accomplish
nanoscale measurements of chemical and mechanical properties, while leveraging the instrumentation they already have in-house.

Response: NCMC researchers prepared a related USMS measurement need statement, *Advanced Scanning Probe Microscopies* (see above). In addition, a NCMC project was defined in this area, and first papers were published in 2005 (e.g., Julthongpiput et al, *Nanoletters* 5(8): 1535-1540 (2005)).

5) **For More Information**

- For more information about the *NCMC-9 Workshop, NIST Combinatorial Methods Center, NCMC research, or NCMC membership*, please visit the NCMC Web site at [www.nist.gov/combi](http://www.nist.gov/combi), or contact:

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- For more information about *specific NCMC measurement technologies*, contact:

  Kathryn Beers, Project Leader, Polymer Formulations
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  Michael Fasolka, Project Leader, SPM Reference Specimens and Nanomaterials
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- For more information on the *U.S. Measurement System Project*, see [www.usms.nist.gov](http://www.usms.nist.gov), or contact:

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301-975-3463
Appendix A: NCMC-9 Agenda

NCMC-9: Combinatorial Methods for Nanostructured Materials
April 24 - 25, 2006 ♦ Bldg. 101 / Lecture Room B

Monday, April 24, 2006 - Morning

8:45 am Welcome and Introduction
Richard Kayser, Director, Materials Science and Engineering Laboratory, NIST
Michael Fasolka, Director, NIST Combinatorial Methods Center

Industrial Application of Nanomaterials

9:00 am Plenary Lecture
Fiona Case, Case Scientific Inc.
Applications of Nanotechnology in Soft Materials: Cosmetics, Foods, Personal Care, Pharmaceutical and Oil Industries

10:00 am Karl Maurer, CombiMatrix Corp.
Building addressable molecular libraries: combinatorial synthesis and analysis of small molecules on a 12,000 feature microelectrode array

11:00 am Plenary Lecture
Prof. Daniel Savin, University of Vermont
Light Scattering Techniques for Polymer Materials

11:45 am Thomas Chastek, Polymers Division, NIST
High-Throughput Light Scattering Measurements

12:05 pm Peter Harris, Veeco Instruments
Advanced Scanning Probe Microscopy Techniques

Monday, April 24, 2006 - Afternoon

Combi Methods for Thin Nanostructured Materials

1:30 pm Invited Lecture
Prof. Sergiy Minko, Clarkson University
Combinatorial approach to the problem of interfacial interactions via gradient polymer brushes

2:15 pm Invited Lecture
Prof. Thomas Epps, University of Delaware
Combinatorial Studies of Block Copolymer Interactions with Surfaces

3:15 pm Alamgir Karim, Polymers Division, NIST
Nanostructured Materials Group

3:45 pm Christopher Stafford, Polymers Division, NIST
Combinatorial approaches to nanoimprint lithography
Discussion Session

4:15 pm  Introduction: Michael Fasolka, Director, NIST Combinatorial Methods Center

4:20 pm  Discussion: Fasolka, Beers, Karim Moderators
Goals:

• Discuss key measurement needs in industrial development and application of nanostructured materials.
• Arrive at priorities for the development of combinatorial and high-throughput measurement methods for nanostructured materials.

5:15 pm  Adjourn

Tuesday, April 25, 2006 – Morning

Interactions and Update

9:00 am  Michael Fasolka, NCMC
Welcome Back

9:10 am  Steve Fletcher and Jawwad Darr, InsightFaraday Partnership, UK
High Throughput Nanomaterials Discovery - a UK perspective

9:45 am  Daniel Cutbirth, Nscrypt Inc.
Novel Deposition System for Combinatorial Libraries

10:40 am  Celesta Fong, CSIRO, Australia
CSIRO Overview and NCMC Interaction

10:55 am  Christopher Stafford, NCMC
NIST Gradient Flow Coater

11:15 am  Michael Fasolka, NCMC
High throughput preparation of specimens for TEM

11:30 am  Chang Xu, NCMC
Combinatorial Surfaces of Grafted Polymers

11:45 am  Brian Berry, Polymers Division
Orientation in Nanostructured Thin Films

12:00 pm  Michael J. Fasolka
Wrap Up and Initial Discussion Summary

NCMC Tours and Demonstrations

1:45 pm  Convene in NCMC Labs – Building 224, Rm. B204
See Lab Tours Handout for Schedule

3:30 pm  Adjourn – See you at NCMC-10! (October 5-6, 2006)
## Appendix B: NCMC-9 Participant List

### Speakers

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