NISTIR 7409

NCMC Workshop Report
NCMC-10: Persistent Challenges in Combinatorial Materials Science

Michael J. Fasolka
Carol E. Laumeier
Kathryn L. Beers
Christopher M. Stafford

National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

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Combinatorial Methods Group
Polymers Division
Materials Science and Engineering Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-8542

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

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Robert Cresanti, Under Secretary of Commerce for Technology

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William Jeffrey, Director

NCMC Workshop Report

NCMC-10: Persistent Challenges in Combinatorial Materials Science

Hosted by the NIST Combinatorial Methods Center (NCMC)

October 5-6, 2006

National Institute of Standards and Technology, Gaithersburg, MD 20899

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1) Introduction and NCMC-10 Workshop Goals

Tremendous advances in the development and application of combinatorial and high-throughput methods for materials research have been accomplished over the past 15 years. Despite these accomplishments, today's combinatorial materials science is met by some of the same challenges that faced early implementers. *NCMC-10: Persistent Challenges in Combinatorial Materials Science*, was a forum to examine these "persistent" measurement needs. The Workshop addressed library fabrication, which continually emerges as a problem when Combi is applied to new materials targets; and informatics/system integration, which has always been a central barrier to workflow development. Over the course of two days, the Workshop brought together 29 industry representatives from the 21 NCMC member institutions, along with academic researchers and more than 30 NIST staff (see **Appendix C: NCMC-10 Attendee List**). The Workshop included:

- Technical symposia focused on the persistent challenges of Combi workflow integration and library fabrication. Experts from BASF Aktiengesellschaft, Cornell University, Dow Chemical, North Dakota State University, Building and Fire Research Laboratory (BFRL), and Materials Science and Engineering Laboratory (MSEL) gave plenary lectures on these subjects.
- 10 lectures detailing MSEL Combi methods for polymer formulations, nanostructured materials, organic electronics, and inorganic electronic devices.
- NCMC-led discussion sessions aimed at identifying key technologies, commercial instruments, and topics at national conferences needed to address persistent challenges to Combi implementation.
- Tours and method demonstrations in NCMC and BFRL laboratory facilities.

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-10 had the following goals:

- Discuss the persistent challenges in terms of specific Combi research scenarios
- Examine established and emerging creative solutions to these problems
- Create roadmaps for addressing persistent challenges, including
    - Priority technologies to be developed
    - Priority topics for national and international symposia
    - A statement of needs and specifications for instrument vendors

2) Summary of the NCMC-10 Technical Program

The NCMC-10 Technical Program (see **Appendix A: NCMC-10 Agenda**) provided scientific context for the Workshop goals, and introduced ideas germane to a Discussion Session (see following Sections 3 and 4). NCMC Director **Michael Fasolka** introduced and discussed the goals and structure of NCMC-10.

To kick-off the technical symposium, a group of plenary lectures illuminating technical issues in combinatorial methods were presented. The first two lectures considered the challenges to Combi workflow development. **Bret Chisholm**, of North Dakota State University, lectured on *The Importance of Understanding the Capabilities of a Combinatorial Workflow.* This presentation gave a detailed, inside account of the statistical analyses Professor Chisholm's team used to characterize the uncertainty, reproducibility, and precision of their combinatorial coatings discovery system. As the title suggests, this sort of analysis is extremely important for establishing the reliability of the system and the data it produces. **Wolfgang Schrof**, of BASF Aktiengesellschaft, discussed *Flexible All-in-One Workflows – Challenge for Combinatorial Systems Integration*. Dr. Schrof's lecture considered the factors that can complicate the integration of combinatorial workflows. These included technical and measurement issues, such as informatics infrastructure development, and the difficulty of incorporating application testing. In addition, the presentation touched on some of the management and cultural issues - from training to acceptance of Combi data - that can hamper the use of combinatorial methods in a large organization.

The next section of the Workshop considered creative solutions to the main barrier of implementing Combi for new materials sets: library fabrication. **Matt Bishop**, of Dow Chemical Company, talked about Dow's Combi effort in *Thermoset Formulations*. Dr. Bishop's lecture outlined how Dow researchers extended their existing combinatorial workflows and technology to handle these challenging systems. **Eric Amis**, Deputy Director of the Materials Science and Engineering Laboratory (MSEL) at NIST, presented *Exploiting the Innovator's Dilemma: Making Breakthroughs Happen*. Dr. Amis' vision-oriented lecture considered Combi in terms of a "disruptive technology" of the type described in C.M. Christensen's book, *The Innovator's Dilemma: When New Technologies Cause Great Firms to Fail*. **Bruce van Dover**, of Cornell University, lectured on *Combinatorial Exploration Using Cosputtered Thin Films*. Professor van Dover's presentation exemplified the power of highly flexible co-sputtering and co-deposition systems, and how they can be harnessed for Combi research of a variety of metal and oxide materials systems. **Jeff Gilman**, of NIST's Building and Fire Research Laboratory (BFRL) at NIST, lectured on *Leveraging Current Technology for Fabricating New Materials Libraries: Extrusion, Gradient Coating, and Liquid Handling and Analysis Platforms*. Through his talk, Dr. Gilman gave an overview of the equipment his group modified, in cooperation with vendor partners, to construct combinatorial workflows for polymer composites and coatings. Equipment included a spray system for multicomponent gradient film formation, modified liquid handlers, and high-throughput measurements of performance properties (e.g., flammability).

In addition, NIST staff reported on NCMC, NIST Polymers Division, and NIST Ceramics Division research. Generally, these talks focused on combinatorial and high-throughput measurement methods for new materials sets. **Kathryn Beers**, Project Leader, Polymer Formulations, discussed *Radical Polymerization in Small Spaces*. Dr. Beers' lecture gave an overview of recent work in her project, harnessing microfluidics for creating libraries of solution-based and grafted polymers, and their application for combinatorial studies of polymerization kinetics, photopolymerized products, and responsive surfaces. **Carl Simon**, Polymers Division, discussed *Combinatorial Polymer Scaffold Libraries*. Dr. Simon's talk described a new technique for making porous polymer tissue scaffolds with a gradient in blend composition. **Nathan Gallant**, Polymers Division, discussed *Universal Gradient Substrates for "Click" Biofunctionalization*. Dr. Gallant's lecture described a new, versatile approach for creating surfaces imparted with gradients in grafted biofunctional moieties. This technique harnesses the NCMC UVO gradient device and

the "click" chemistry route developed by Nobel Laureate Barry Sharpless. **Leah Lucas**, Polymers Division, discussed *Combinatorial Thermal Platform for Study of Polythiophene Thin Film Structure and Organic Field Effect Transistors*. The lecture discussed a study in which gradient techniques, in particular the NCMC gradient hotstage, were used to optimize the processing of organic electronic materials. **Michael Fasolka**, NCMC Director, presented an update on a 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. **Patty McGuiggan**, NCMC, discussed *Combinatorial Peel Measurements of Pressure Sensitive Adhesives*, completed using a modified commercial texture analyzer. The technique is able to produce peel force master curves in a single experiment. **Heqing Huang**, NCMC, discussed *Mechanics of Complex Interfaces: Interfacial Adhesion*. As a follow-up to his NCMC-9 presentation, **Thomas Chastek**, NCMC, lectured on the design and performance of instruments for *Microfluidic Light Scattering*. Case studies included mapping of surfactant phase behavior over temperature and concentration. Finally, **Makoto Otani** and **Kao-Shuo Chang,** Ceramics Division, NIST, lectured about *Development of a High-Throughput Characterization Tool for Combinatorial Thermoelectric Materials Research* and *Combinatorial Study of Metal Gate Electrodes on HfO_2 for The Advanced Gate Stack,* respectively.

NCMC-10 concluded with a series of tours and demonstrations in NCMC and BFRL laboratory facilities. Stations included the NCMC flow coater, surfactant library fabrication, microfluidic dynamic light scattering, high-throughput Transmission Electron Microscopy (TEM) specimen fabrication, 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-10 Discussion Session Goals and Guiding Questions

A central part of NCMC-10 was a Discussion Session, where Workshop attendees divided into Teams that rotated through three Breakout Sessions that were structured to:

- Further examine library fabrication, system integration, and other persistent challenges to implementing Combi methods for materials.
- Produce roadmaps that outline and prioritize:
    - Technologies and approaches that address current and emerging challenges
    - Topics for future workshops and symposia at national meetings
    - Specifications for vendor supplied Combinatorial and high-throughput instrumentation and software

The Breakout Sessions were moderated by **Michael Fasolka, Kathryn Beers,** and **Christopher Stafford**, with assistance from **Carol Laumeier**, NCMC Outreach Coordinator. To guide the Sessions, the major themes and specific questions for consideration were distributed to attendees in advance of the Workshop, and are as follows:

Breakout Session 1. *Technologies Needed to Meet Persistent Challenges*

A. What are the persistent challenges to your implementation of Combi today? What challenges to you foresee in two years? Consider, for example:
    - Design/fabrication of libraries for new materials sets. For what *types* of materials? For what kinds of processing parameters?
    - System/workflow integration. Where are your informatics "bottlenecks"?
    - Automation of existing equipment. What kinds of instruments?
    - High-throughput measurements. For what materials properties?
    - Measurement reliability. For what materials properties?
    - Database development
    - High-throughput data analysis. What kinds of data?

B. What specific technologies are needed to meet your challenges? Consider, for example:
   - Library approaches and instrumentation
   - Measurement approaches and instrumentation
   - Informatics tools for system integration and data analysis
   - Standards and reference materials

Deliverable from Session 1: A published NCMC report with prioritized lists of key technologies needed now and in two years.

Breakout Session 2. *Vendor-Supplied Instrumentation and Software*

A. What Combi-related products you would be inclined to purchase if they were commercially available today? What would be your required specifications for these products? Examples to consider:
   - Library fabrication equipment. For what materials systems? Over what variables? Required specifications?
   - High-throughput measurement/screening instruments. For what properties? Required tolerances and specifications?
   - Software systems. Examples:
     - For high-throughput data analysis. For what kinds of data?
     - For system integration. For what kinds of instruments/components?
   - Reference materials. For what kinds of Combi measurements?
   - Standard file formats for integration. For what kinds of instruments and data?

B. What types of commercial instrumentation and software do you currently use for Combi? What types of currently available instruments have caught your eye? In each case, how could these products be improved? Consider, for example:
   - Throughput
   - Ability to automate
   - Versatility
   - Reliability
   - System integration
   - Your key specifications for improvement

C. What vendor companies would benefit from hearing these messages?

Deliverable from Session 2: A NCMC document outlining opportunities for vendors to develop or improve products for Combi. This report will be distributed to vendors, including those identified in 2C above, and will be posted on the NCMC Web site.

Breakout Session 3. *Workshops and National Symposia*

A. What topics should be addressed in future NCMC Workshops?
B. What topics for Combi symposia at national conferences would draw your attendance and contribution?
C. What emerging topics in Combi should be addressed through focused symposia at national conferences?
D. At what national meetings would Combi symposia be useful?

Deliverable from Session 3. Your input will be reflected in plans for future NCMC Workshops and symposium proposals submitted to conference committees.

4) Discussion Session Summaries and Conclusions

Breakout Session 1: *Technologies Needed to Meet Persistent Challenges*
Michael J. Fasolka, Discussion Leader

Discussion Summary
Two major themes emerged from this discussion. The first was related to combinatorial methods for high-viscosity organic fluid products. Here, major barriers still exist in terms of both library fabrication and high-throughput measurements. Current formulation and dispensing robots are still mainly geared for aqueous and low viscosity materials. To handle coatings, personal care, cleaning, and other consumer products, industry requires Combi liquid handling systems designed specifically for high-viscosity products. These must be able to accommodate viscous liquids, and ideally, powder fillers and additives. Integrated parallel mixing of samples is necessary, and thorough mixing is essential. Processing protocols must be flexible, with changing mixing times and speeds, temperature soaks, wait times, and order of component addition. Dispensing must likewise handle viscous, complex fluids, with solid additives. The last point is considered to be a huge barrier: "powders are extremely complicated," stated one participant. Dispensing needs to be high precision (perhaps microliter resolution), and amenable to fabrication of dots and film patch libraries, as well as vial or plate arrays.

The main high-throughput measurement barrier for fluid products remains complex rheology. Here, industry requires means to measure the complex viscosity of hundreds of formulations in a short period. Data quality and frequency range (perhaps 10^{-5} Hz to 5 Hz) needs to be comparable to a traditional rotational viscometer. Needs to measure so called "functional viscosity," i.e., assessed in situ in a processing or application condition (e.g., a drying film), were also noted. Parallel/high-throughput measurements of the extent of mixing of complex fluids were also highlighted as a need.

The second major theme concerned informatics barriers. Here, there were two types of needs. The first barrier was in automation of instrumentation and system integration. Participants noted that most vendor instrumentation (and software) is not designed to be automated or integrated with other devices or data structures. Given this, they pointed out needs for device independent data-passing protocols, interchange standards, and flexible data management systems. These areas were seen as good opportunities for vendors to improve their products or for new products (see **Section 2, Summary of the NCMC-10 Technical Program**). The second informatics needs were in datamining and modeling. According to participants, the lack of chemistry- and materials-specific tools for datamining of spectra or micrograph libraries, for example, were a serious and long standing impediment (see also **Section 3**, **NCMC-10 Discussion Session Goals and Guiding Questions**).

In addition, needs for high-throughput measurements of surface properties were discussed. Here measurements of surface tension or surface energy during processing steps (e.g., film forming), were considered important. Surface area and surface energy measurements of porous products and particulate additives were also deemed important.

Conclusions and Response
Through its research projects, the NCMC is continuing to address high-viscosity library fabrication issues. For example, novel approaches to deposition are currently being advanced through the development of a combinatorial "direct write" system (with vendor nScrypt) that can handle and mix viscous and particle laden fluids. This year, the NCMC invested in the second generation of this system, which includes precision translation stages, and will be developing and testing it for library deposition. On the measurement front, the NCMC is sponsoring a new Focus Project in combinatorial melt rheometry. Contact Kalman Migler (kalman.migler@nist.gov) for more information on this opportunity. In addition, the NCMC will continue to exploit microfluidics and other approaches for high-throughput measurements in fluid viscosity.

In terms of informatics, the Center plans to focus future Workshop activities on the issues illuminated in these discussions. In particular, NCMC-12 will have sessions related to practical informatics development, interchange protocols and standards, and the newest advances in datamining methods. In addition, in 2007 the NCMC will create a new Web page on the Member's Only Web site that will be a central resource for learning about and accessing the best open source informatics tools available to the community.

In addition, the NCMC will continue to develop surface- and interface-related Combi tools through the new Mechanics of Complex Interfaces Project (contact Christopher Stafford, christopher.stafford@nist.gov) and the new Combi Nanotechnology effort (contact Michael Fasolka, michael.fasolka@nist.gov).

Breakout Session 2: *Vendor-Supplied Instrumentation and Software*
Christopher M. Stafford, Discussion Leader

Discussion Summary
There was some discussion as to whether modifying commercial equipment ("Combi-fying") in-house would nullify any warranty that the vendor offered. Many attendees noted incidences where it did in fact void the warranty. Others mentioned that they have approached certain vendors about partnering together to provide a unique solution that would enable that instrument to incorporate libraries and high-throughput data collection and output. This could be an area where the NCMC could educate instrument vendors about combinatorial and high-throughput methods, and company's eagerness to purchase such instruments.

On a related note, there was a consensus among all three groups that informatics is a critical and pivotal element that always surfaces when implementing C&HT (combinatorial and high-throughput) approaches. Inherent to informatics is instrument communication (input) and data standards (output). There needs to be cost-effective solutions to these challenges. Instrument vendors need to gain awareness of C&HT so that they can provide simple but effective solutions, such as having a series of DLLs or ActiveX drivers on hand for those who may request them.

The ability to handle high-temperature applications in a variety of instruments was a common theme. Many instruments that are already on the market for C&HT are designed for the bio- and pharma- fields, where water is the primary component/carrier, and as such are relegated to temperatures below 100 ºC. The material science community requires that much higher temperatures be accessible. This also highlights the need for these instruments to be engineered to handle volatile (and often flammable) solvents at these high temperatures.

There appears to be a real void (and thus opportunity for vendors) in instruments for handling/dispensing high viscosity materials and solids. This continues to be brought up in personal discussions and at national meetings. Additionally, it is pervasive that the polymer community be able to measure complex rheology in a high-throughput manner. Finally, high-throughput high-resolution imaging (Scanning Electron Microscopy, TEM, Scanning Probe Microscopy, etc.) needs to be addressed, as these analytical tools are seen as a bottleneck to completing the Combi process.

Finally, an interesting discussion centered on affordable, portable instruments for education purposes. These instruments would help educate the next generation of scientists on C&HT and provide them with real-life experience with this new toolset. This would be most beneficial to the academic community, but also has an interesting place in the industrial sector, as they embark on educating management, customers, and suppliers on the power of Combi, and how it will help drive business.

Conclusions and Response

The main conclusion is that instrument vendors need to be educated about the rigor, flexibility, and specifications required from users who wish to integrate devices into combinatorial workflows. In addition, there are key opportunities for vendors to develop and produce new needed measurement tools. To this end, the NCMC will create a "Message for Vendors" Web page on the NCMC Web site that outlines key requirements for existing tools, especially software interfaces, and opportunities in complex rheology, high temperature processing, and automated electron microscopy. In addition, vendors will be invited to a future Workshop (e.g., NCMC 12) to take part in a forum to discuss these issues. Moreover, this year we will make a concerted effort to recruit instrument manufacturers into the NCMC consortium. Current NCMC member organizations are encouraged to suggest appropriate vendor companies; please email suggestions to combi@nist.gov.

Breakout Session 3: *Workshops and National Symposia*

Kathryn L. Beers, Discussion Leader

Discussion Summary

During the three subgroup discussions, a number of topics and themes emerged. With regard to NCMC Workshops, all subgroups stressed that some form of tutorial or short course would be valuable for new members. Suggestions included adding a half-day or full-day review into the Workshop, and producing materials that could be used as outreach to the academic community for training graduate students. One participant used the term "Creative Combi 101" to describe the NCMC contribution to the community.

All three groups discussed the topic of statistical methods and data analysis. There was a lot of interest in data modeling; however, suggestions ranged from requesting a tutorial in the field from world-class experts, to concerns that such a session might not be appropriate for the typical NCMC audience, and that levels of application and strategy similar to Bret Chisolm's talk were more valuable.

Modeling in general, and computational methods as a compliment to combinatorial experimentation were also a top interest. In particular, the identification and understanding of descriptors for data sets, as well as development of surrogates and predictors which could substitute for testing, were of interest.

Understanding the relationship between fundamental and performance properties was another popular topic. Challenging cultural resistance to change and acceptance of new tests in place or complimentary to established performance metrics (e.g., scratch tests), discovering real correlations, and sharing experiences among various member companies were all interesting topics.

Shared experiences were a popular request as well. Details relating to success stories, including start-up plans, costs to market and impact information, as well as bringing in vendors to discuss future opportunities for C&HT R&D were all of interest.

The general consensus was that Workshops that focus on specific application areas (e.g. Nanotechnology, Formulations, Coatings, etc.), were more successful/interesting than those focused on more general themes. Other suggested topics for future meeting included Electronics (Combi device development for chips, circuits, and sensor development), Bio (including bringing in C&HT veterans from Pharma to share experiences from a more developed Combi community), Energy (alternate feedstocks, fuel cells, hydrogen storage), and Consumer Products and Alloys.

More information on the pros and cons of a variety of new forums for Combi symposia can also be found in the transcribed notes (see **Appendix B: Raw Discussion Session Notes**), including AIChE, a replacement for the defunct Combi Gordon Research Conference and other issues.

Conclusions and Response

As organizers of Combi-related symposia at several upcoming national and international conferences, NCMC staff members are positioned to shape some of the directions and content of these meetings. Two examples include the Fall 2007 Materials Research Society meeting (Boston, Fasolka co-organizer), and the 5th International Workshop on Combinatorial Materials Science and Technology (Fall 2008, site in Germany TBA, Fasolka co-organizer). The MRS symposium has a traditional focus in Combi applications, and in this sense the NCMC is dedicated to advancing consortium interests. Accordingly, organic formulation science will be forwarded as a focus of the event, as well as "emerging" areas of organic electronic devices, bio, and so called "complex materials" that contain both organic and inorganic components. Given its size and international attendance, the International Combi Workshop series seems poised as a replacement for the Combi Gordon Research Conference (GRC). The challenge is to grow the soft materials components (polymers, coatings, bio) of this event, which historically has had a strong focus in catalysis and inorganic materials. Headway was made in this respect at the 4th Workshop (December 2006, Puerto Rico), and the NCMC is committed to ensuring that consortium interests are represented and advanced in these Workshops.

With respect to future NCMC Member Workshops, the advice culled from these discussions will be continually and directly implemented in these events. Our goal will be to keep the Workshops focused on current issues and practical knowledge. Priority topics will be a review seminar or short course ("Creative Combi 101") for new users, practical informatics and modeling issues and development, a "Veterans Forum" (including representatives from Pharma), and Combi for emerging materials systems, such as biomaterials/healthcare, organic electronics, and complex materials.

5) For More Information

- For more information about the **NCMC-10 Workshop, NIST Combinatorial Methods Center, NCMC research, or NCMC membership**, please visit the NCMC Web site at www.nist.gov/combi, or contact:

  Carol Laumeier, NCMC Outreach Coordinator
  carol.laumeier@nist.gov
  301-975-6093

  Michael Fasolka, NCMC Director
  michael.fasolka@nist.gov
  301-975-8526

Appendix A: NCMC-10 Agenda

NCMC-10: Persistent Challenges in Combinatorial Materials Science

October 5 - 6, 2006 ◊ Bldg. 101 / Lecture Room B

Thursday, October 5, 2006 - Morning

9:00 am Welcome and Workshop Introduction
Michael Fasolka, Director, NIST Combinatorial Methods Center

Workflows and System Integration

9:30 am Invited Lecture
Bret Chisholm, North Dakota State University
The Importance of Understanding the Capabilities of a Combinatorial Workflow

10:30 am Invited Lecture
Wolfgang Schrof, BASF Aktiengesellschaft
Flexible All-in-One Workflows – Challenge for Combinatorial Systems Integration

11:20 am Invited Lecture
Matt Bishop, Dow Chemical Company
Thermoset Formulations

Thursday, October 5, 2006 - Afternoon

Library Design and Fabrication

1:20 pm Invited Lecture
Eric Amis, Deputy Director MSEL, NIST
Exploiting the Innovator's Dilemma: Making Breakthroughs Happen

2:10 pm Invited Lecture
Bruce van Dover, Cornell University
Combinatorial Exploration Using Cosputtered Thin Films

3:10 pm Invited Lecture
Jeff Gilman, Building and Fire Research Laboratory, NIST
Leveraging Current Technology for Fabricating New Materials Libraries: Extrusion, Gradient Coating, and Liquid Handling and Analysis Platforms

Discussion Sessions – Lecture Room B and Room B-111

4:00 pm ***Introduction***: Michael Fasolka, Director, NIST Combinatorial Methods Center

4:10 pm ***Discussion Moderators***: Fasolka, Beers, and Stafford

5:15 pm ***Adjourn***

Friday, October 6, 2006 – Morning

NCMC Update

8:45 am **Michael Fasolka**, NCMC
Welcome Back

8:50 am **Kathryn Beers**, NCMC
Radical Polymerization in Small Spaces

9:20 am **Carl Simon**, NIST
Combinatorial Polymer Scaffold Library

9:40 am **Nathan Gallant,** NIST
Universal Gradient Substrates for "Click" Biofunctionalization

10:00 am **Leah Lucas,** NCMC
Combinatorial Thermal Platform for Study of Polythiophene Thin Film Structure and Organic Field Effect Transistors

10:30 am **Michael Fasolka,** NCMC
Update: High-Throughput Preparation of Specimens for TEM

10:50 am **Patty McGuiggan**, NCMC
Combinatorial Peel Measurements of Pressure Sensitive Adhesives

11:10 am **Heqing Huang**, NCMC
Mechanics of Complex Interfaces: Interfacial Adhesion

11:30 am **Tom Chastek**, NCMC
Microfluidic Light Scattering

11:50 am **Makoto Otani**, Electronic & Optoelectronic Materials Group, MSEL
Development of a High-Throughput Characterization Tool for Combinatorial Thermoelectric Materials Research

12:10 pm **Kao-Shuo Chang**, Electronic & Optoelectronic Materials Group, MSEL
Combinatorial Study of Metal Gate Electrodes on HfO_2 for The Advanced Gate Stack

Friday, October 6, 2006 – Afternoon

NCMC Tours and Demonstrations

1:30 pm ***Lab Tours*** – convene in the NCMC Labs, Building 224, Room B204

3:30 pm ***Adjourn***

Appendix B: Raw Discussion Session Notes

Breakout Session 1: *Technologies Needed to Meet Persistent Challenges*

Group 1

1) Roadblocks
    a) Money
    b) Less expensive technologies
        i) Dispensing
        ii) Higher viscosity
        iii) Parallel Mixing
        iv) Integration (hardware)
        v) Design with automation in mind
2) "Automationware"
    a) Software (many types)
    b) XML?
        i) NIST support/working group
    c) Windows flaws, viruses
        i) Device independent protocols
3) Hardware
    a) Flexible mixing & processing
    b) Dye
    c) Rheology (complex)
4) Informatics for chemistry
5) NIST ask vendors

Group 2

1) Roadblocks
    a) Price vs. throughput (information for vendors)
    b) Automation
2) Dispensing – high precision
    a) Solids, liquids, high viscosity (additives)
    b) Complex issue – powders are complicated
3) Rheology performance testing
4) Data management system
    a) Quantitative, data mining

Group 3

1) Measurement
    a) Surface tension (during process in situ, during film forming)
    b) Function viscosity (during process in situ, during film forming)
    c) Tg (glass transition temperature)
    d) Surface area – pores (quantitative and high-throughput)
    e) Surface energy – high surface area systems (dust), particles in pores
    f) ICP, AAS
2) Rheology
3) Interchange standards
4) NIST/vendor working group
    a) Hardware integration

Breakout Session 2: *Vendor-Supplied Instrumentation and Software*

Group 1

1) Does "Combi-fying" null warranty?
    a) Flexibility/reconfiguring

   b) Partnership?
   c) Legal agreements
2) High viscosity/solids
   a) LHR (liquid handling robot)
3) More versatile plate readers
   a) High temperature (>100 degrees C)
4) Informatics packages
   a) Cost effective
   b) Data standards
      i) Symyx
      ii) Waters
      iii) Mettler - Toledo

Group 2

1) Communication standards
   a) Importing sample information
2) Analytical tools
   a) Handle high temperature/volatiles/flammables
3) Multichannel solid dispensing
4) Product recovery/isolation
5) Complex rheology
6) Sample preparation for high-resolution imaging
   a) SEM (scanning electron microscopy)
   b) TEM (transmission electron microscopy)
   c) AFM (atomic force electron microscopy)

Group 3

1) Coordinated sample loading/fiduciary
2) Vendor awareness to high-throughput
   a) DLL, ActiveX, etc.
3) Affordable, simple academic combinatorial and high-throughput tools for education purposes
4) STANDARDS
5) 10 mL equivalent of microtitre (“macro”)
   a) 24 or less
   b) Bottom visibility

Breakout Session 3: *Workshops and National Symposia*

Group 1

1) Cross-fertilization is good, but associated “training” is also valuable
   a) Tutorials
   b) ½ day or 1 day tagged onto normal meeting
   c) Short Course?
   d) Discussions / Reaching out to Academia (training the next generation)
2) with tutorials and short courses
3) Statistics Important
4) Overview of general approaches
5) How to Apply in Combi?
6) Basic Language of the statistics community
   a) Who are the speakers?
7) Wolfgang can suggest (gave names of several German professors: Honerkamp from Freiburg; Hamprecht from Heidelburg)
8) Computational
   a) Modeling – emerging technology?
   b) Interfacing with data management (for descriptors?)
   c) Improving use and understanding of descriptors

9) GRC: canceled and sorely missed?
10) In Europe – a new model, possibly sponsored by DeChema (maybe early 2008) to help replace GRC (Gordon Research Conference) with worldwide audience (100 to 150 attendees)
11) AIChE (American Institute of Chemical Engineers)
12) Only if pushing into processing (missing and a variable, curing, sputtering, layering)
    a) Scaling problems?
13) CRS (Controlled Release Society)
14) Colloid Science Area / Societies? (Simon Gibbon)
    a) Need applications
    b) Expertise demand defined
    c) Extension to phys. schem.
15) Formulations Community
    a) Again phys. chem.

Group 2

1. Tutorials / Short Courses good
2. Combi Device Development
    a) Circuits, chips, OLEDs (organic light emitting diodes) (NIMS [National Institute of Materials Science Japan], Philips, etc.)
    b) All encompassing topic
    c) A whole new level of library fabrication
    d) Sensor Discover / Development (New Sensor Technology
    e) Symyx oil gauge
3. Consumer Products
    a) Characterization, Rheology, Formulation
4. Maybe Alt. Energy (alternate feedstocks, e.g. biodiesel?)
5. General application areas as topics tend to work better (nano, formulation, elec., etc.)
6. Statistics may be too “dry” for a whole session
    a) Maybe NCMC not right forum
    b) Bret Chisholm’s talk was a good level and very valuable
    c) Applications and strategies more appropriate for this audience
7. More success stories
    a) Start-up costs ($$)
    b) Marketed successes (with statistics data)
8. Performance / Property Correlations
    a) Overcoming cultural prejudices
    b) Brining in external validation sources
9. Modeling
    a) In data mining
    b) Descriptors / design
10. Interdisciplinary communication
11. Alloys? (possible topic) – ternaries and higher (Hysitron)

Group 3

1) Creative Combi 101
2) Sharing Company Experiences
3) Vendors (Research-Oriented Discussions)
    a) Where is the area going? Symyx, HTE, Avantium, Accelerys, Waters, Spotfire, smaller start-ups
4) Porous Materials
5) C&HT (combinatorial and high-throughput) Success Stories / Impact
    a) More depth
    b) Start-up Plans
6) Statistical Methods / Analysis (Yadu can suggest speakers)
    a) Modeling Data

   b) Visualization
7) Relationship between fundamentals and performance properties
   a) Challenging cultural acceptance
   b) Discovering real correlations
   c) Experiences / Successes (e.g. surf. energy and biofouling example of Bret Chisolm)
8) Pitfalls and Warnings
9) Property / Performance Relationships in general
   a) Which ones work, which ones don't?
10) Testing Substitutions
   a) Predictors / Surrogates
11) Focus on Specific Material Areas?
   a) Nano, Coatings,
12) Photovoltaics, Gen. Electronic Devices (organic, composite electronics)
13) Next Generation Audience?
   a) Review previous ground for newcomers
14) Alternate Energy (Fuel cells, H-storage, etc.)
15) Bio
   a) Reach out to the veterans
   b) Pharma (correlating experience with drugs to materials?)
16) AIChE (American Institute of Chemical Engineers): community has not been included to date and has HUGE knowledge base / history (MechEng., too) in mixing, heat transfer, transport phenomena in general

Appendix C: NCMC-10 Attendee List

NCMC Member Attendees	
Air Products & Chemicals	David R. Hubble
Air Products & Chemicals	Svetlana M. Ivanova
Air Products & Chemicals	Michael S. Lowry
Air Products & Chemicals	Menas S. Vratsanos
Arkema Inc.	Christopher A. Bertelo
Arkema Inc.	Stephan Moyses
Avon Products, Inc.	John Brahms
Avon Products, Inc.	Prithwi Maitra
Avon Products, Inc.	Derrick McKie
Bayer Material Science	Nancy Hessler
Bayer Material Science	Peg Kendi
Chemistry Innovation Knowledge Transfer Network	John Whittall
[The Dolomite Centre Ltd.]	Harold Swerdlow
[Univ. of Liverpool]	Andrew Cooper
Dow Chemical Company	Don Patrick
Honeywell International	Mickey McDonnell
[UOP]	Frank Modica
Hysitron Inc.	Ryan Major
Hysitron Inc.	Oden Warren
ICI/National Starch & Chemical	Yadunandan Dar
ICI/National Starch & Chemical	Simon Gibbon
PPG Industries	Kevin Gallagher
Rhodia Inc.	Bertrand Pavageau
Symyx	Michael Smith
NIST Attendees	
Jae Hyun Kim	Joseph Antonucci
Carol Laumeier	Matt Becker
Brian Okerberg	Thuy Chastek
Kirt Page	Jun Young Chung
Derek Patton	Aaron Forster
Stephanie Scierka	Sheng Lin-Gibson
Chad Snyder	Marty Green
Christopher Stafford	Kazunori Iida
Scott Stanley	Alamgir Karim
Chengqing Wang	Chang Xu
Invited Speakers	
BASF Aktiengesellschaft	Wolfgang Schrof
Cornell University	R. Bruce van Dover
Dow Chemical Company	Matthew T. Bishop
North Dakota State University	Bret Chisholm
NIST Speakers	
Eric J. Amis/MSEL	Jeffrey W. Gilman/BFRL
Kathryn Beers/NCMC	Heqing Huang/NCMC
Kao-Shuo Chang/MSEL	Leah Lucas/NCMC
Tom Chastek/NCMC	Patty McGuiggan/NCMC
Michael Fasolka/NCMC	Makoto Otani/MSEL
Nathan Gallant/NIST	Carl Simon/NIST