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Overview of the NIST Materials Science and Engineering Laboratory

Richard R. Cavanagh
Acting Director

Materials Science and Engineering Laboratory
National Institute of Standards and Technology
Gaithersburg, Maryland

And

Boulder, Colorado

MSEL Framework

Mission

To promote U.S. innovation and industrial competitiveness ***in the development and use of materials*** by advancing

measurement science,

standards, and

technology

in ways that enhance economic security and improve the quality of life.

Vision

Creating ***critical measurement solutions*** that enable innovation and competitiveness in materials.

Materials Science and Engineering Laboratory

MSEL

R.R. Cavanagh
Acting MSEL Director

M.J. Fasolka
Scientific Advisor

151 Technical Staff
21 Administrative Staff
161 NIST Associates
$47M STRS Budget
$53M Total Budget

Ceramics

D.L. Kaiser, Chief

- Functional Properties
- Synchrotron Methods
- Structure Determination Methods
- Nanomechanical Properties

Materials Reliability

S. Hooker, Chief

- Nanoscale Reliability
- Structural Materials
- Cell and Tissue Mechanics

Metallurgy

F.W. Gayle, Chief

- Thin Film and Nano-structure Processing
- Magnetic Materials
- Materials Performance
- Thermodynamics and Kinetics

Polymers

E.K. Lin, Chief

- Electronics Materials
- Process Characterization
- Biomaterials
- Characterization and Measurement

The Technology Innovation Program
Funding Transformational Research for Critical National Needs

TIP's Mission

- Assist US businesses and institutions of higher education or other organizations, such as national laboratories and nonprofit research institutions
- Support, promote, and accelerate innovation in the United States through ***high-risk, high-reward*** research
- In areas of critical national need

America COMPETES Act (PL 110-69)
Enacted August 9, 2007

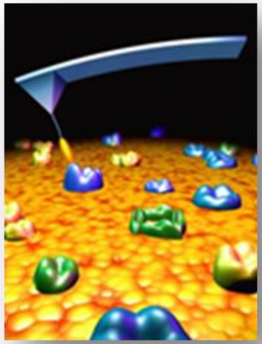
FY 2010 funding = $69.9 million

➢ *Through competitive, peer-reviewed solicitations, TIP has initiated R&D funding for:*

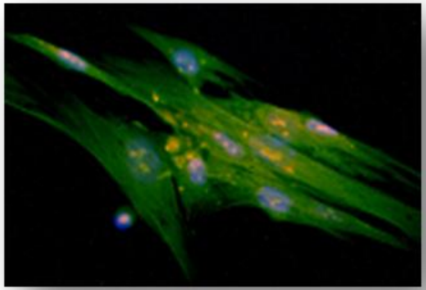
- $84 M ($41 M from TIP) for manufacturing processes for advanced materials (nano, smart materials, composites, alloys/superalloys)
- $150 M ($73 M from TIP) for advanced sensing and repair technologies for the civil infrastructure

www.nist.gov/tip

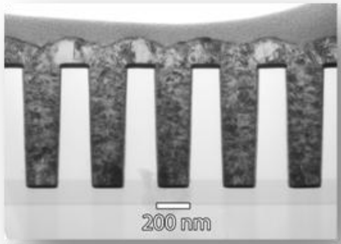
MSEL Programs



Materials at the Nanoscale



Biomaterials

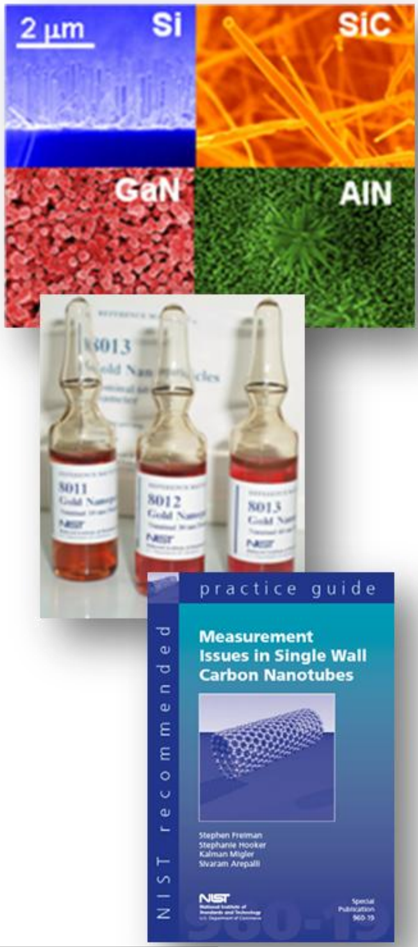


Electronic Materials

Structural Materials

Materials at the Nanoscale

MSEL works closely with key industry and government stakeholders to address the most pressing materials measurement and standards challenges that hinder the development, manufacturing, and safe, reliable use of nanostructured materials and devices.



Program Objectives:

- Methods to quantify the size, shape, physiochemical characteristics and performance of fabricated nanostructures and nanostructured materials that are critical elements of cutting-edge electronic devices.

- Measurement methods to quantify, with nanoscale resolution, mechanical properties, and mechanical states such as stress and strain, of materials and devices; reference materials and standards that advance the widespread application of nanoscale mechanical properties measurements.

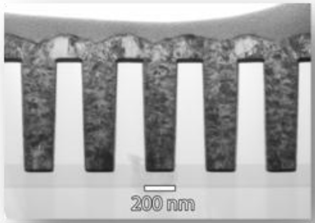
- Measurement methods necessary to advance both state-of-the-art and emerging routes for fabricating nanostructured materials and devices.

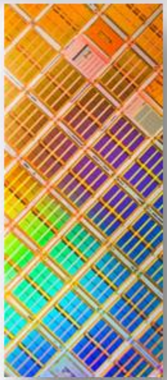
- Measurement approaches, standard test methods, and reference materials that enable the safe and reliable use of nanomaterials in current and emerging technologies.

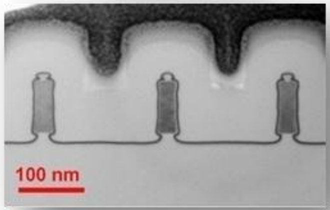
Electronic Materials

MSEL

MSEL works closely with the U.S. semiconductor and electronics industries to address the most pressing materials measurement and standards challenges that hinder development and utilization of advanced materials and material processing for cutting-edge electronics technologies.







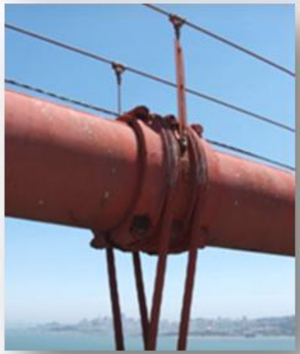
Program Objectives:

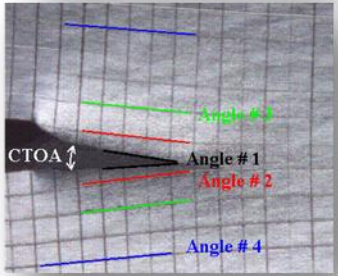
- Measurement methods and standards to quantify the size, shape, thickness, and properties of the fabricated nanostructures and nanostructured materials used in cutting-edge electronic devices.
- Measurement methods needed to help industry introduce new materials into electronic devices in order to increase performance or enable new applications.
- Measurement methods and standards needed to assess electronic device reliability due to stresses and strains at microscale and nanoscale dimensions.

Structural Materials

MSEL

MSEL is developing the next generation of measurement tools, sensors, predictive models, data, and standards needed to accurately assess and predict the safety of structural materials now and in the future.





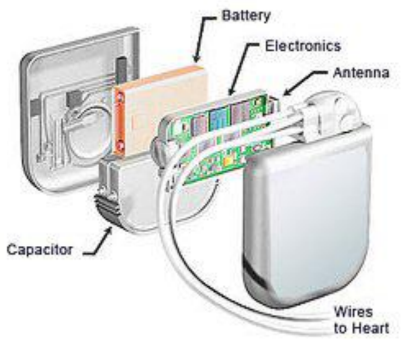
Program Objectives:

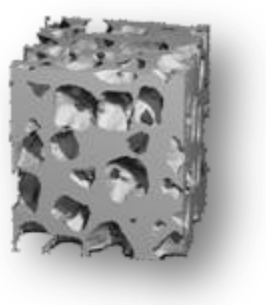
- *Measurement methods, standards, and data required to ensure safe operations of the nation's oil and gas pipelines and enable the safe and widespread distribution of alternative clean fuels, e.g., hydrogen and biofuels.*
- *Standards and measurement methods to guide the development and use of new materials in structures and components for infrastructure and automotive applications.*

Biomaterials

MSEL

MSEL is developing cutting-edge instrumentation and measurement methods that illuminate complex interactions between biological systems and synthetic materials. These tools advance the development of quantitative medical imaging technologies, emerging therapeutic approaches like tissue engineering, and reliable implantable medical devices.





Program Objectives

- Measurement methods and standards that quantify the properties and performance of synthetic materials, natural biomaterials, and biomedical devices in the presence of biological media.
- Measurement methods and standards that quantify the response of biomolecules, cells and tissues to such materials and devices.
- *Measurement techniques and instrumentation for imaging the morphology, chemistry and biological function of cells, tissues and biomaterials.*

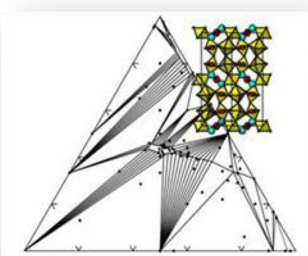
Measurements and Standards

Standards—reference materials, documentary standards, calibrations, and reference data—are used in commerce to lower trade barriers, in manufacturing to ensure quality control, and in research and development to enable discovery and optimization of new materials and devices. Materials Standards activities are a significant part of each of the other MSEL Programs.

Outputs:

Standard Reference Materials **-** *MSEL provides over 120 reference materials that are used to evaluate and qualify methodology and instrument performance related to the physical and chemical characterization of materials, and to conduct inter-laboratory test comparisons.*

Documentary Standards **-** *MSEL leads and participates in numerous committees and working groups in national and international standards developing organizations (SDOs). These SDOs develop, coordinate, promulgate, and maintain voluntary-consensus, documentary standards—test methods, technical specifications, and technical reports—that directly impact businesses in nearly every sector.*



Reference Data **-** *MSEL provides standard reference databases that are compilations of evaluated, high-quality technical data for use in materials and process development and optimization, materials forensics, and product design and development.*

Standards: Charpy Impact Verification

Motivation

- Predicting the reliability of structural materials is essential to avoid critical failures.
- Charpy impact testing measures fracture toughness of construction steels, providing an effective quality control parameter.

Objectives

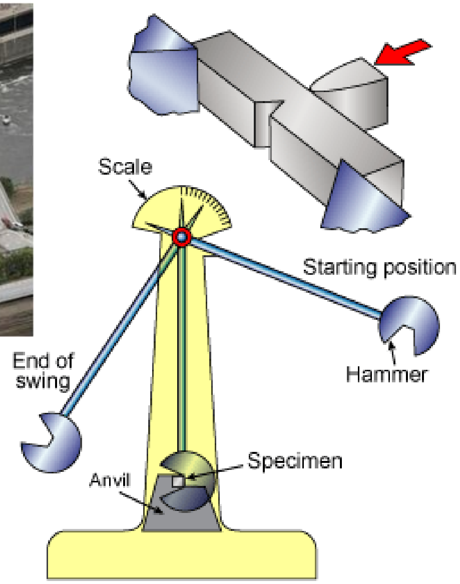
- Provide standard reference materials (SRMs) to verify impact test machine performance
- Conduct post-test analysis to identify corrective measures
- Ensure customer compliance with national and international test standards (ISO, ASTM, ASME, ***U.S. military procurement specifications***)
- Support harmonization of international standards

Achievements and Impact

- Certify 10,000+ SRMs per year
- Evaluate 1000+ machines per year (worldwide)
- Ensure the US has the most accurately characterized population of impact machines in the world

Collapse of I-35W bridge (2007)

NIST Charpy SRM 2098



Charpy impact machine and associated reference specimens

Partners

Measurement Programs of Interest

Vehicle Light-Weighting

Manufacturability of lightweight alloys for fuel efficiency

Materials Performance Under Extreme Conditions

High Temperature and High Loading Rates

Rates: Quasi-static → Crashworthiness → Explosion

Soft Body Armor

Failure mechanisms and mitigation in ballistic vests

Energy Storage

Fuel Cells and Battery Measurements

NIST Center for Automotive Lightweighting

Motivation

- Facilitate incorporation of lighter, advanced alloys and components into cars, improving fuel efficiency, saving car companies wasted development time and expenses by improving design models.

Objectives

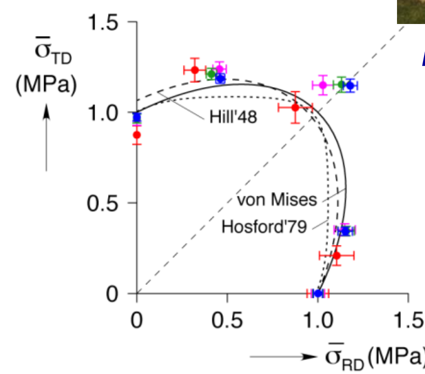
- Provide next-generation material tests, metrology and data to speed incorporation of light-weight materials and components.

Achievements and Impact

- New Sheet Metal Springback Cup Test
  - ASTM E-2492, ISO pending
- New multiaxial stress-strain law that is tied to evolving crystallographic texture during forming
- Discovery - TRIP effect being impeded by texture of conventionally processed sheet
  - US Steel is modified its thermomechanical processing based on these insights

ASTM E-2492

Lightweight Body-in-White



Multiaxial Flow Surfaces

In Situ Stress Measurement

Partners

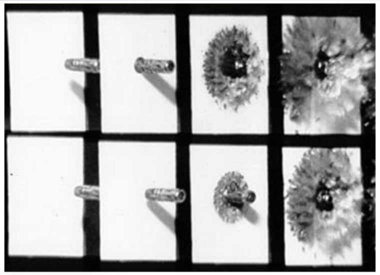
Mechanical Performance: Extreme Conditions

Project Goal: To develop new high strain rate measurement techniques and to provide more accurate and more robust data for modeling material behavior under extreme conditions.

Motivation: Reliable manufacturing, transportation safety, and ballistic armor, require data for constitutive models to accurately predict material behavior under rapid loading & heating conditions.

Approach: Develop instrumentation and measurement science underpinning a "Kolsky Bar" approach that combines ultra high-rate loading and in-situ high-temperature capabilities.

Partners: NRL, Auto Industry, OLES, PHMSA, DHS



Deformable bullet impact for Soft Body Armor Standards

"Gel-Man" for Force Protection Measurement Naval Research Labs

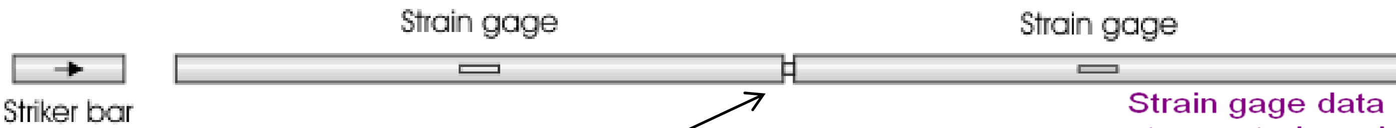
PHMSA

Project is Anticipated

NIST Kolski Bar Instrumentation

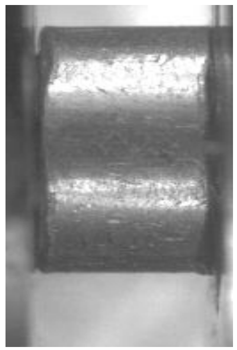
(aka Split Hopkinson Pressure Bar)

MSEL



specimen

Wave Direction

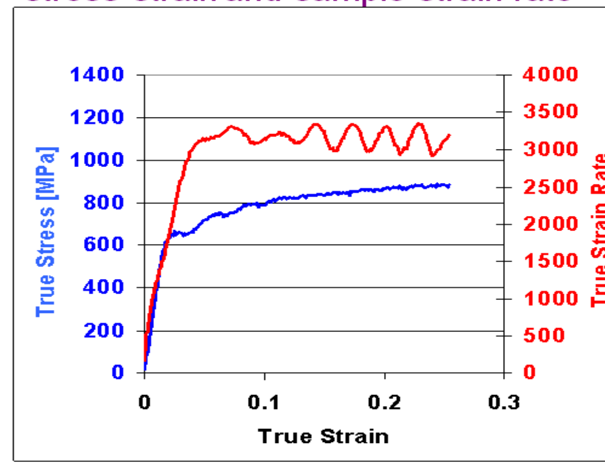


Max T: 800 ºC

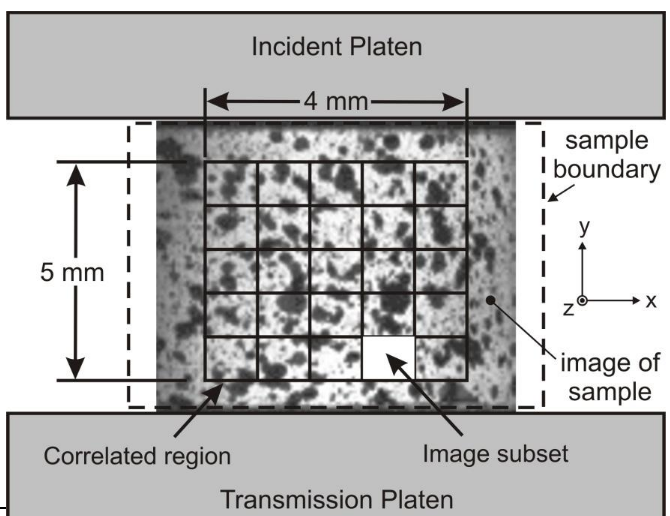
ΔT Rate: ~ 14K ºC/s

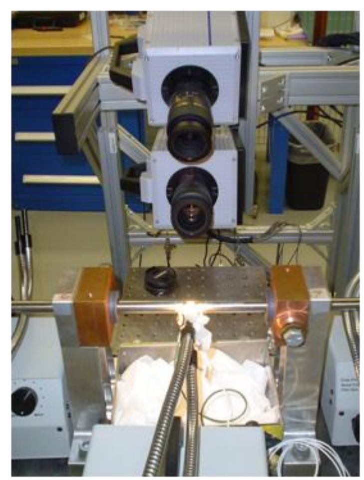
Strain Rate: 10^3 - 10^5

Strain gage data used to calculate stress-strain and sample strain rate



3D High-Speed Digital Image Correlation System





3D photogrammetry using random dot pattern

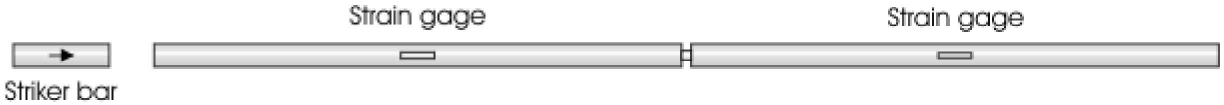
Cameras measure specimen shape up to 180,000 times/second

Tensile Pulse-Heated Kolsky Bar

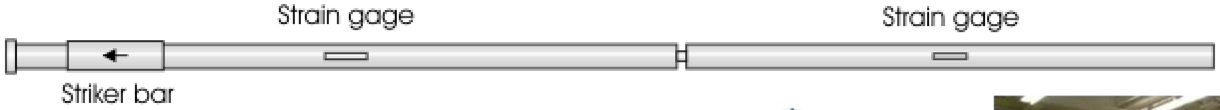
New Facilities:

1) Pulse-heated tension Kolsky bar for ductility and fracture studies (auto crashworthiness & impact studies)
2) Fast servo-hydraulic test frame for intermediate strain rates (up to 10^3 per sec to overlap with Kolsky bar $10^3 – 10^4$ per sec)

Compression Kolsky bar arrangement:



Tension Kolsky bar arrangement:



The new equipment will be acquired in FY2010 (ARRA) and operational in FY2011

Soft Body Armor Project

MSEL provides data, measurement methods and new instrumentation to characterize the processing, structure, mechanics and long-term reliability of high performance polymeric fibers used for ballistic protection. These services support development and enforcement of regulation by partner agencies and they inform decision tools for municipalities and other customers of the technology.

Program Objectives

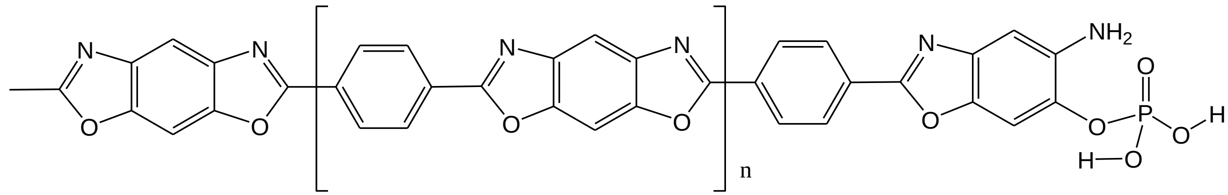
- Early data identified mechanisms of field failures in established materials. Both chemical and mechanical routes to weakened products were identified in polybenzoxazole (Zylon) fibers. [This work included testing on materials which had failed in the field.]
- New measurement methods to characterize mechanical properties at application relevant strain rates: High Strain Rate Kolsky Bar Apparatus, modified for fiber testing
- *Measurement techniques and instrumentation for characterizing next generation hybrid materials which impregnate ballistic fibers with shear thickening ("stab-resistant") materials.*

National Institute of Justice

The Research, Development, and Evaluation Agency of the U.S. Department of Justice

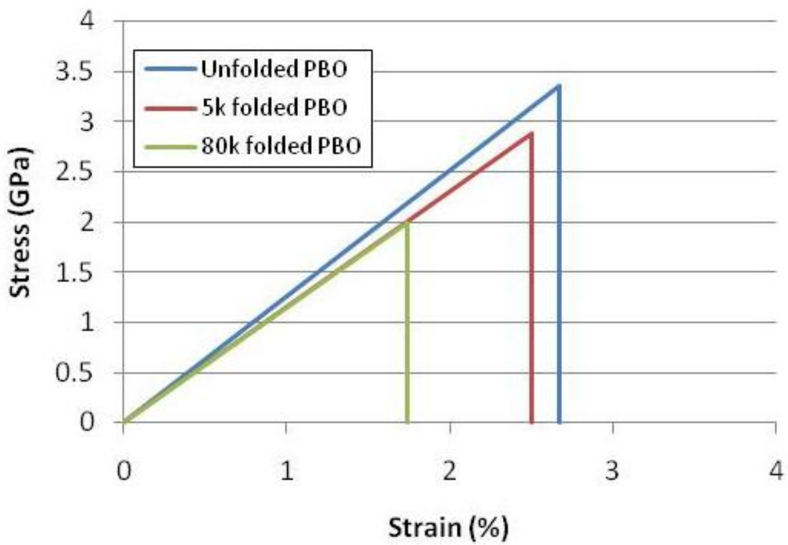
Soft Body Armor Project

MSEL

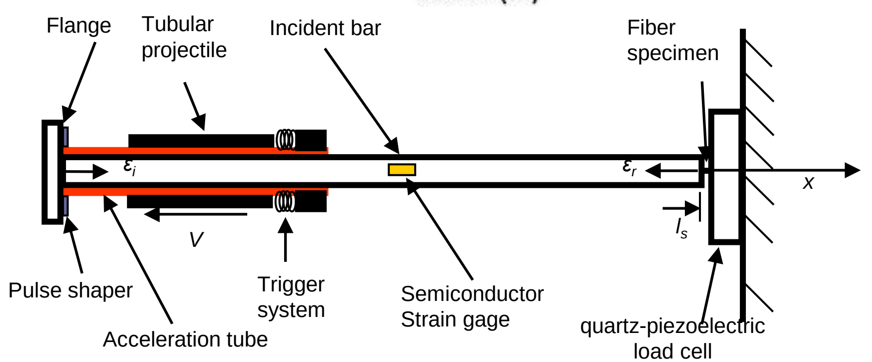


phosphoric acid attached to PBO oligomer as monoaryl phosphate ester

- **Chemical Degradation**
  - Residual phosphoric acid contaminants from both synthesis and processing of PBO fibers
  - *Key tools: GC-MS and MALDI-TOF-MS*



- **Mechanical Degradation**
  - Bend tests indicate mechanical weakening and performance lifetime effects
  - *Key tools: NIST-built folding apparatus and commercial high throughput single fiber tensile test*



- **High Strain Rate Testing**
  - Kolsky Bar with modified grip technology

UNCLASS

Energy Storage and Delivery Materials

MSEL

Motivation

- Renewable energy sources require improved electrochemical energy storage & delivery technologies, but the pathway to improve the rate limiting ion transport materials that underpin these technologies is poorly defined

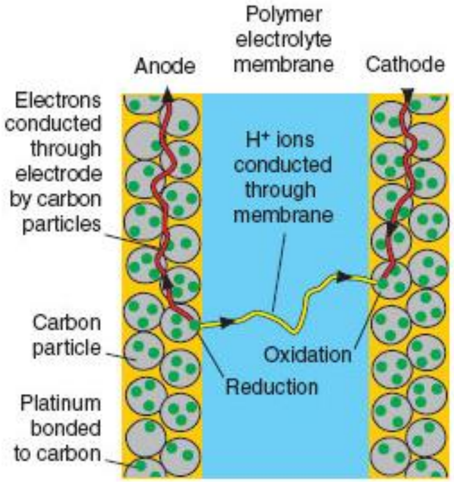
Objectives

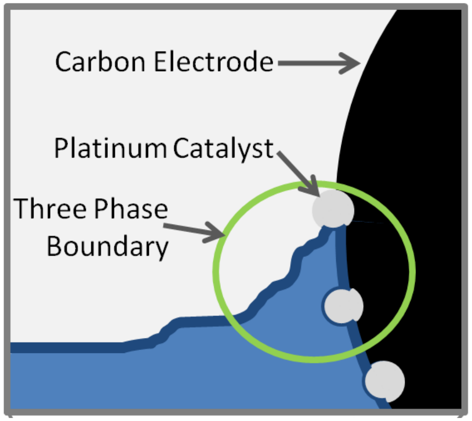
- Deliver measurements that identify the structure and dynamics in the ion transport media, especially at the electrode interfaces, that hinder charge transport and device performance.

Accomplishments

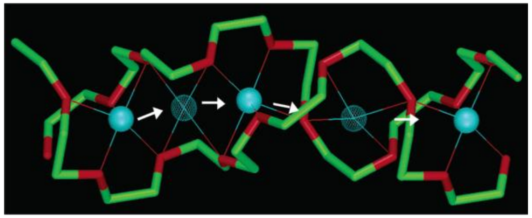
- Perceived an interfacial structure in fuel cell membrane and are demonstrating that this interface can lead to strong deviations in the transport properties of the critical ion channels.
- Quantified a strong coupling of the high frequency polymer electrolyte dynamics to the Li ion mobility through the electrolyte for model battery systems.

Structure and Transport at Interfaces





Ion dynamics in solid-polyelectrolytes



Stoeva et al, *J. Am. Chem. Soc., 125(15), 4619,* ***2003****.*

Customers and Partners

- NIST Center for Neutron Research (Dura)
- Seoul Nat'l U (Yoon); Uni-Mainz (Frey); U Mass (Center for Hierarchical Manufacturing); Waseda U (Nishide); SUNY Binghampton (Wang)
- General Motors (Xie)

UNCLASS

Interfaces in Energy Storage & Delivery Materials

Measurement Challenge

- Ions must pass through several interfaces between the ion transport media/electrodes/catalysts to extract charge
- Interfacial structure and properties can be very different from the bulk and impose an interfacial resistance to charge transport
- Interface sensitive structure, property, and performance measurements are critically needed

Accomplishments and Impact

- Substrate surface energy can induce lamellar-like water/ion transport channels in Nafion that are sub-optimal for charge extraction – oriented parallel not into the electrodes
- Established phase-modulated infrared spectroscopy method that is sensitive to the kinetics & the molecular state of the water/ion in Nafion interfaces as thin as 10 nm
- Quantified a dramatically reduced H_2O diffusivity in the thin film interfacial regions of a fuel cell membrane material

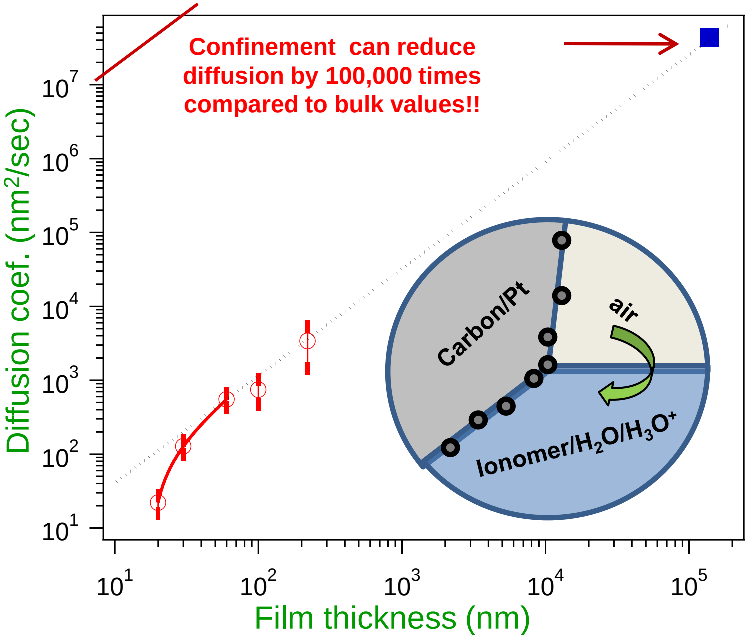
Future

- Correlate interface morphology with water/ion transport kinetics on the range of surfaces relevant for fuel cells. Use to validate parameters used fuel cell modeling.
- Leverage these platforms to corroborate the suspected role of interfaces in the fuel cell service life failures.

Lamellar H_20 transport channels in thin Nafion interfaces

Dura et al, *Macromolecules, 42(13), 4769,* ***2009****.*

Reduced H_20 diffusivity in thin Nafion interfaces



Hydrogen Storage

MSEL

Motivation:

Industry requires methods to accurately assess and compare the performance of candidate materials for hydrogen storage and battery electrodes.

Measurements of hydrogen content needed to assess performance are challenging.

Objective:

High-throughput measurement of the hydrogen content of novel materials proposed for hydrogen storage and for electrodes in Ni-MH batteries.

NIST Program:

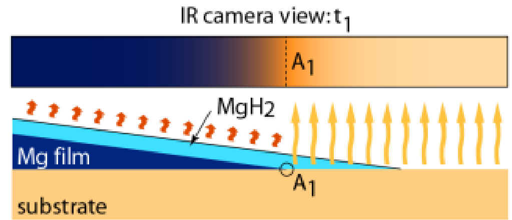
Methods to screen storage materials:

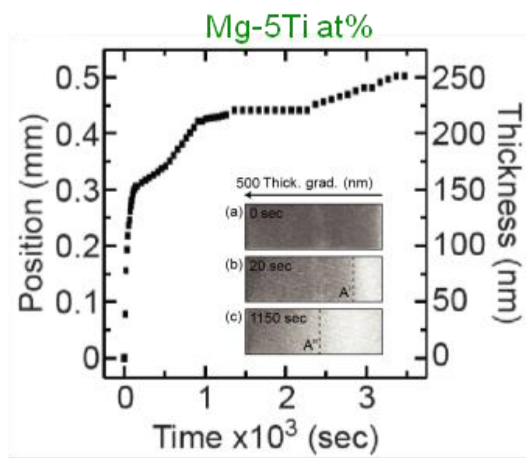
- IR, FTIR and Raman imaging/spectroscopy methods widely usable by industry
- High spatial resolution needed for combi

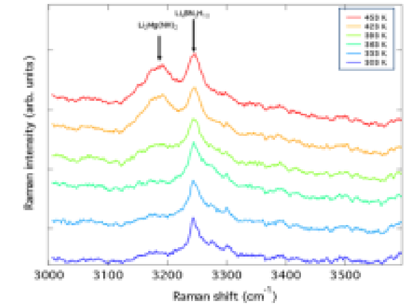
Direct measurements of hydrogen content:

- To calibrate screening methods
- Neutron reactor-based Prompt Gamma Activation Analysis (w/ CSTL/NCNR)
- Volumetric Sievert PCI measurements w/ Raman

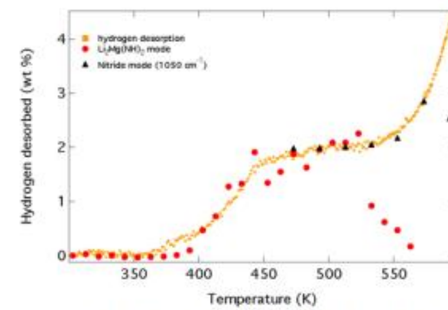
Screening for combi/High Throughput





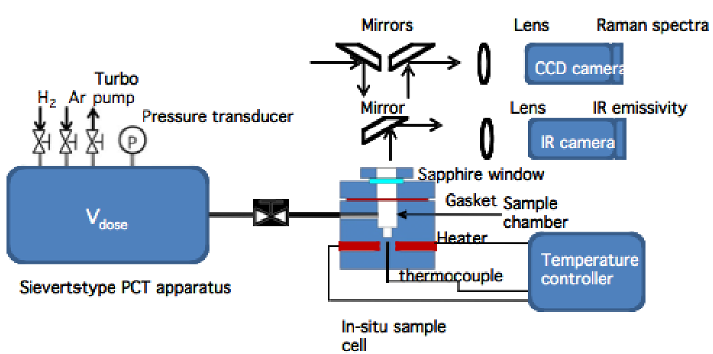


Raman intensities of $Li_2Mg(NH)_2$ and $Li_4BN_3H_{10}$ modes during heating from room temperature to 453 K



Desorbed hydrogen (wt.%) and Intensities of $Li_2Mg(NH)_2$ and nitride modes as a function of temperature

Direct Measurements



Key Partners: Energy Conversion Devices (Ovonic), UMD, OSU, PNL