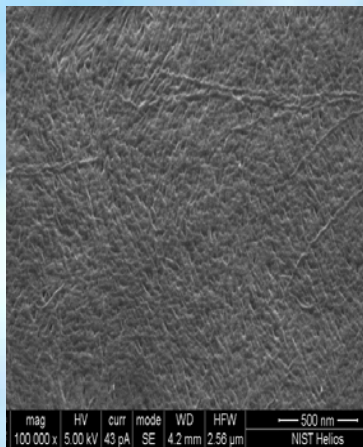


Nanomaterials Analysis and Standards

**Michael T. Postek, Dianne L. Poster, NIST,
Mark Driscoll, SUNY, Syracuse,
Jay LaVerne, University of Notre Dame
Mohamad Al-Sheikhly,
University of Maryland**



Council on Ionizing Radiation Measurements and Standards

National Institute of Standards and Technology

- **Welcome to NIST**
- NIST is the U.S. National Metrology Institute (NMI)
- Research projects in advanced manufacturing (broadly defined), nanotechnology, nanomaterials, ionizing radiation and the necessary measurements (metrology) and standards are found all across NIST
 - **Leverages the knowledge gained from work on other measurement problems, materials and structures**
 - For example: Semiconductor metrology
 - » Semiconductor companies most demanding over the last few years – measuring ever-shrinking structures
 - » Nanotechnology natural fit
- Significant need for development of both advanced, basic (research/laboratory), and manufacturing (applied/production) measurement science

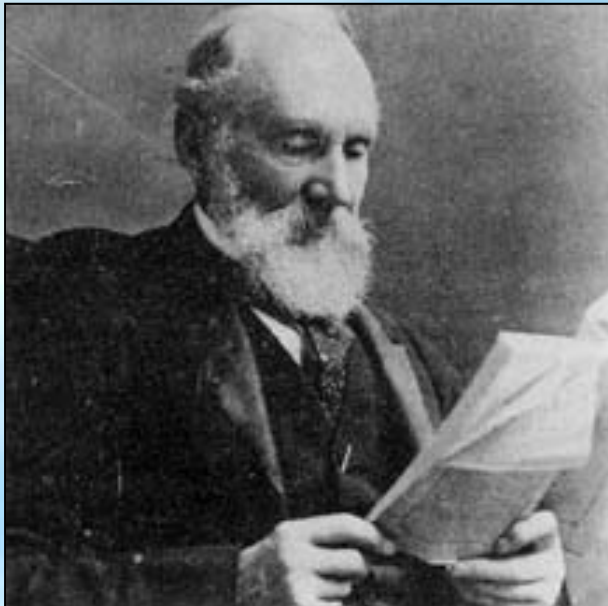


Gaithersburg, MD



Boulder, CO

Metrology: The science of measurement; a system of measures



“When you can **measure** what you are speaking about, you know something about it. But when you cannot **measure** it, your knowledge is of a meager and unsatisfactory kind. It may be the beginning of knowledge, but you have scarcely advanced to the stage of science.”

William Thomson, Lord Kelvin 1824 - 1907

In order to measure a quantity accurately, it is necessary to fully study and understand the measurement process itself
– may require multiple disciplines

NIST works closely with scientists and industry to develop the Nation’s metrology infrastructure necessary for scientific, technical, and economic advances.

National Institute of Standards and Technology

Promotes U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life through its laboratory programs and partnerships:

measurement science

Create the experimental and theoretical tools – methods, metrics, instruments, and data – that enable innovation

standards

Develop and disseminate physical standards and provide technical expertise to standards that enables comparison, ensure interoperability, and supports commerce

technology

Drive innovation through knowledge dissemination and public-private partnerships that bridge the gap between discovery and the marketplace

Measurements are Critical to Innovation

If you know how to measure something, you can design it, improve it, and compare it.

NIST measurement science provides the foundation for innovation in every industry and economic sector, from manufacturing to health care to defense.



Technical Laboratory Activities

- **Laboratory R&D:**

- Perform basic and applied research, planned and implemented in cooperation with industry that anticipates and addresses the industry's most important measurement and standards needs in a timely fashion.

One reason why the CIRMS meeting is so important is to inform and get industry needs before NIST management.

Standard development takes time

- **The National Measurement System:**

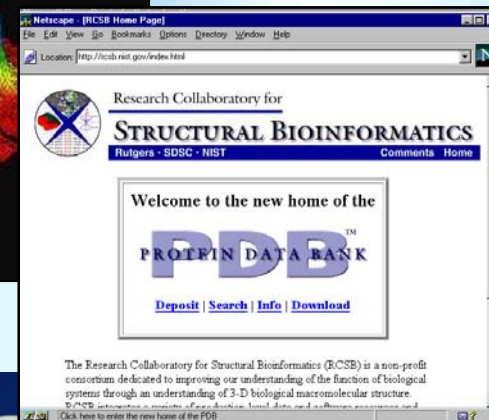
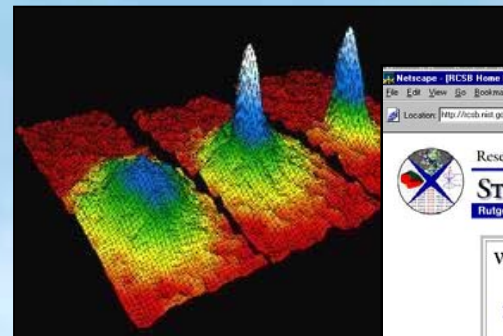
Strengthen the national system of standards, measurement, measurement traceability, and conformity assurance.

- **The International Measurement System:**

Provide leadership in harmonizing international measurements and standards to facilitate trade.

NIST Laboratories, Products, and Services

- **Measurement Research**
>>2,200 publications/year
- **Standard Reference Data**
>65 types available
>20 on-line databases
virtually all NIST databases available free on-line



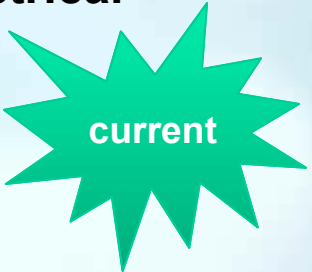
Standard Reference Materials

- >1,300 types available*
>32,000 units sold/year
- **Calibrations and Tests**
>3,000 items/year
- **Standards Committees**
>400 NIST staff, 900 committees

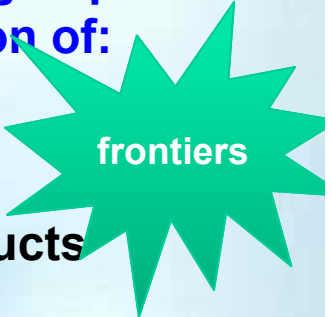


CIRMS, Ionizing Radiation and Manufacturing (1)

- NIST and its academic partners have been pursuing:
 - Fundamental and applied research in the physical interactions of ionizing and non-ionizing radiation with matter.
 - Efforts include the synthesis and characterization of advanced nanomaterials, including:
 - soft nanomaterials for drug delivery applications
 - magnetic nanocomposites for imaging and electrical functions
 - adsorbents for environmental products
 - membranes tailored for fuel cell uses
 - Etc.
 - Demonstration of applying ionizing radiation processing to plant materials for the effective manufacturing characterization of:
 - Novel nanomaterials
 - Advanced paper and printing applications
 - Coatings and inks for secure printing and new products



current



frontiers

CIRMS, Ionizing Radiation and Manufacturing (2)

- Opportunities with other agencies within the Federal Government
 - US Government Publishing Office & Bureau of Printing and Engraving
 - Inks – paper, money, 3D printing (Braille)
 - Secure printing applications (passports, visas, holograms)
- In all aspects, the key NIST component is:
 - The development of measurement methods and standards to support the precise and accurate characterization of the products
 - Understanding and optimizing product **manufacturing and performance**.
 - Development of standards, technology & data for accurate measurements & characterization –



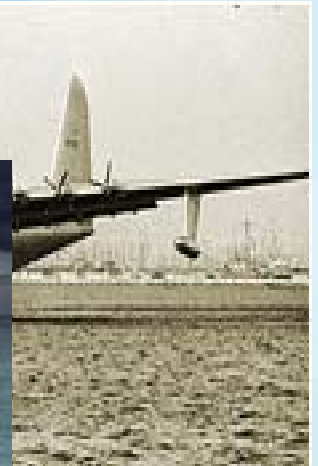
Understanding what is made

So... what do strategic alliances look like?

- **UMd - Core long-term collaboration in materials innovation**
 - Training fundamental radiation processing, chemistry and dosimetry – Dr. Mohamad Al-Sheikhly
 - Acquisition and setup of the asymmetric flow field flow fractionation (AF⁴) system at NIST targeting near field scanning optical microscopy and atom probe.
- **Government Publishing Office**
 - Guest Researcher was placed on assignment to NIST to investigate new methods for fiber analysis and innovations for their agency
- **SUNY**
 - Long term collaboration with Dr. Mark Driscoll
- **nanoC-Technology** - Dr. John Cowie
- **Ebeam** - Michael Fletcher
- **Other National Laboratory** interactions to study electron interactions with cellulose and nanocrystalline structures
 - Notre Dame – Dr. Jay LaVerne
 - Brookhaven National Laboratory – Dr. James Wishart

???Question???

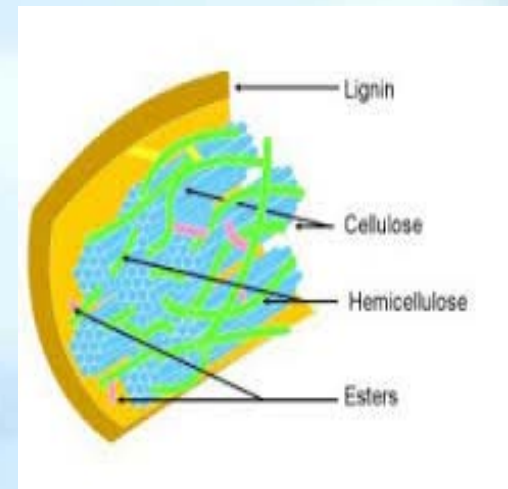
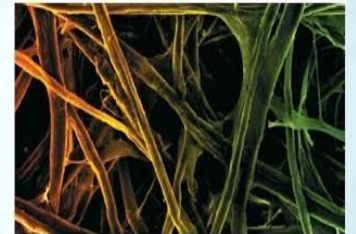
What do: ancient Japanese armor, V
biplanes, the Hughes H-4 Hercules -
a proposed "unsinkable"



of ice
it

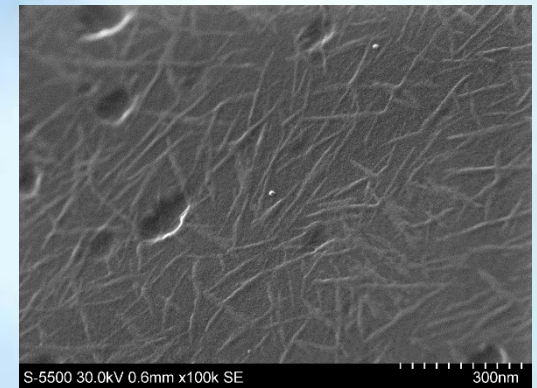
Wood and Cellulose

- **Wood is a natural composite composed of cellulose, hemicellulose and lignin (and other components).**
 - strong interaction of these three components makes wood incredibly strong and durable.
 - this leads to high cost to process wood for uses, other than a structural material.
- **Cellulose: major structural component of wood and plant fibers, most abundant polymer synthesized by nature**
- **Despite its great abundance, cellulosic biomass has seen only limited application beyond its historical uses:**
 - Paper
 - Construction materials
 - etc.
- **Forest Products (wood) industry is very “conservative”**
- **Leads to new and exciting \$\$ opportunities**

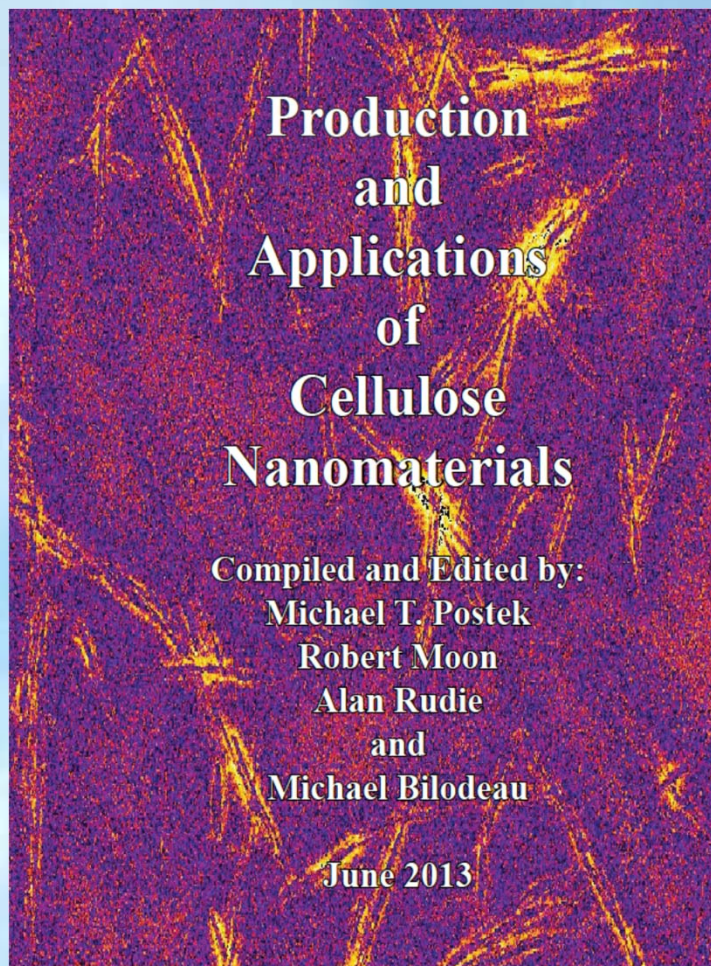


Even Smaller: Cellulose Nanomaterials

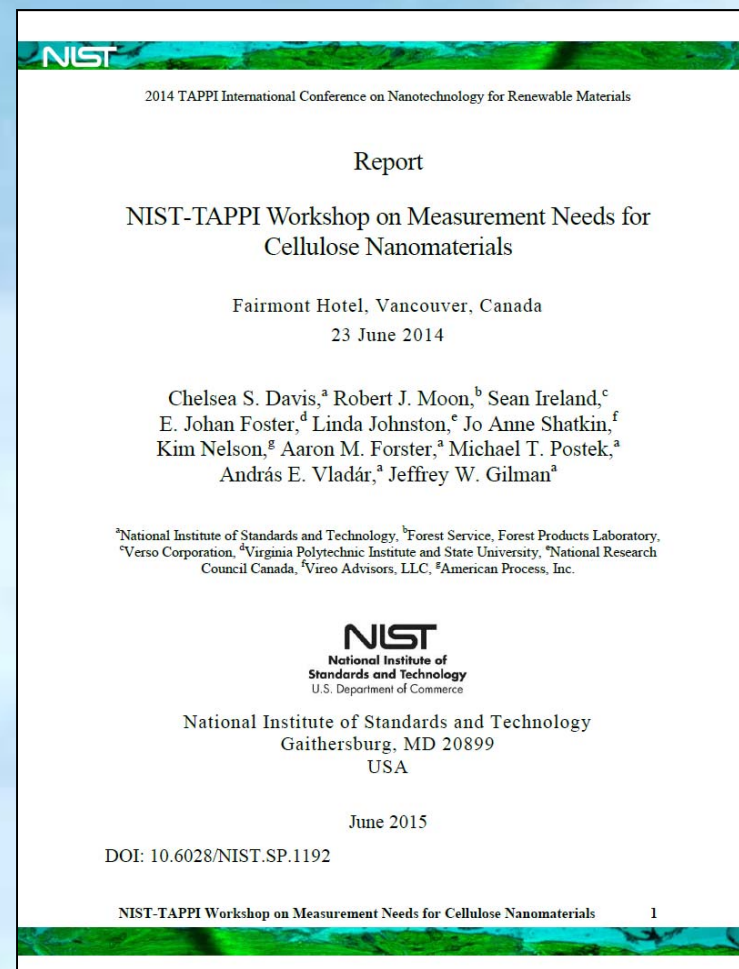
- **Cellulose, manufactured to the smallest possible-size (~ 2 nm x ~ 100 nm), is a high-value \$ material that:**
 - Enables products to be lighter and stronger
 - Has less embodied energy
 - Can require no catalysts in its manufacturing (in stark contrast to Carbon Nanotubes)
 - Biologically compatible – key for disposal
 - Come from a readily renewable resource



Research in Cellulose Nanomaterials



**2012-2013 Over 100 Research
Projects Documented**



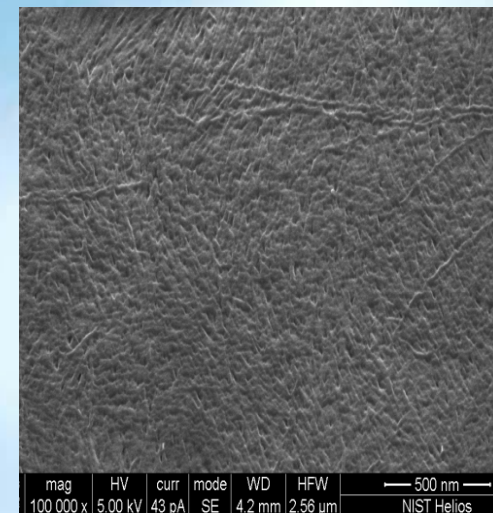
**NIST 2014 Measurement
Needs Workshop**

Metrology in the Nanoworld

- In the nanoworld, we have no first-hand experience so we must rely on “well functioning and calibrated” instruments to provide reliable measurements for process control
 - Instruments are fallible
 - This must always be remembered
- Role of accurate measurements more important today than any other time because the error budget is so small and we cannot rely only on past data since properties (related to size) might change
- Metrology is often the last thing considered, but often turns out to be a limiting factor.
 - If you can't measure it, you can't manufacture it**

Potential Impact of Cellulose Nanomaterials

- **Potential for a dramatic impact on the national economy – early estimates to be as much as \$250 billion worldwide by 2020**
- **Cellulose-based nanotechnology creates a pathway for expanded and new markets utilizing these renewable materials.**
- **Cellulose nanomaterials are a natural by-product of a Biorefinery.**



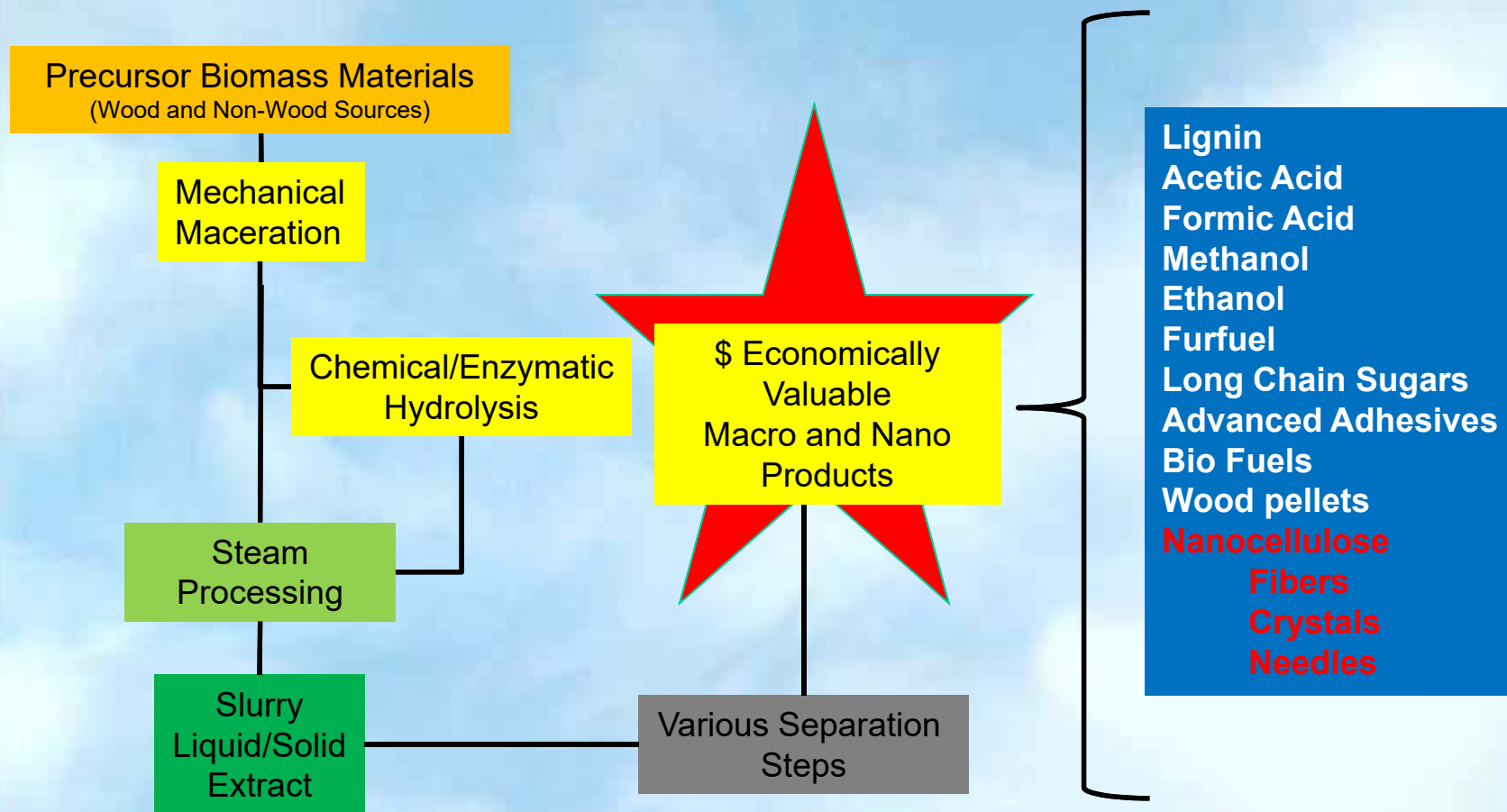
Nanocellulose paper



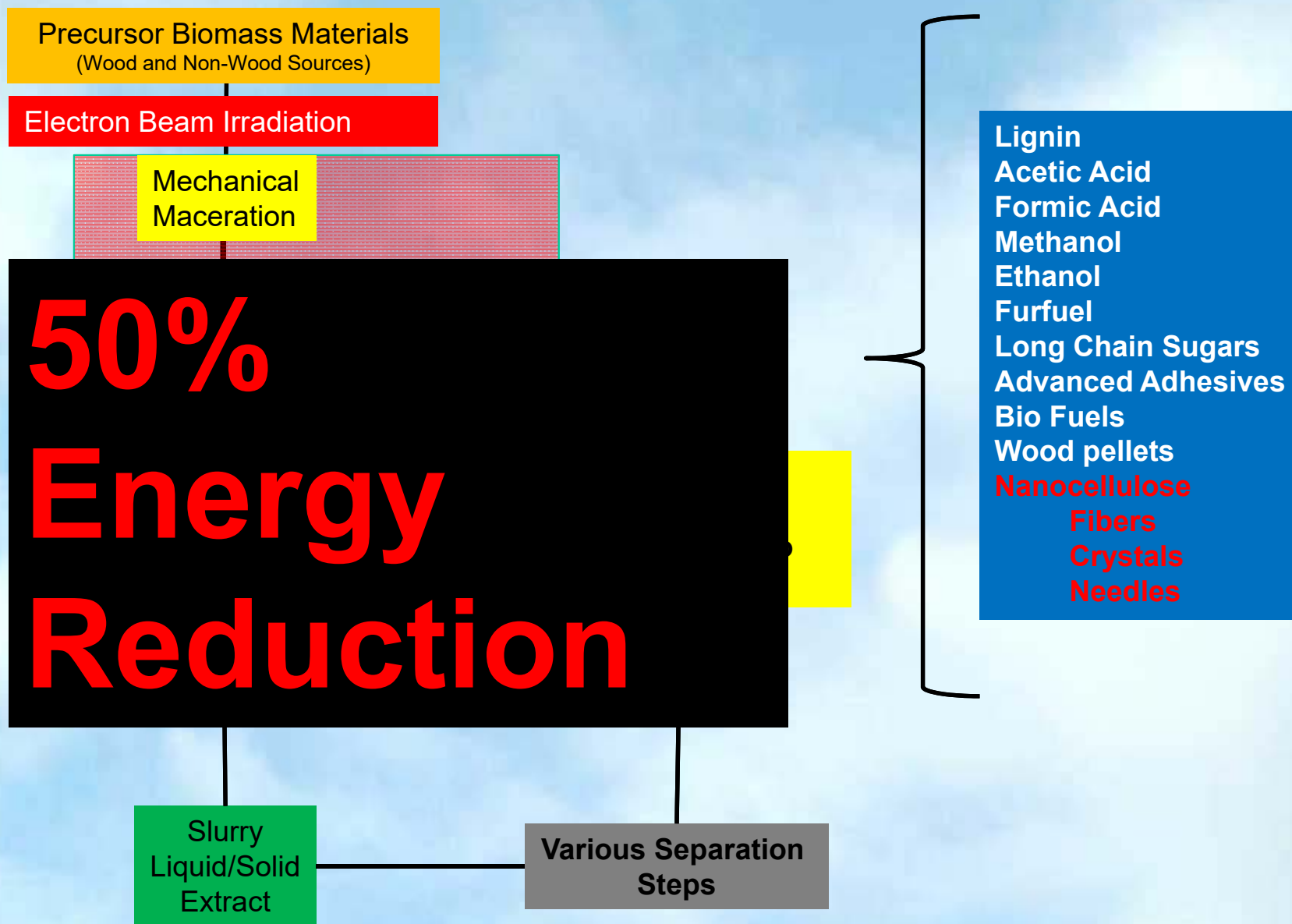
Lignocellulosic - based Biorefinery

- Utilizes wood, grasses and agriculture waste to produce economically beneficial materials and chemicals.
 - Initial chemical target for the Biorefinery was glucose for the production of ethanol.
 - More recently, other economically valuable chemicals and materials are being studied and produced, including nanocellulose.
- Pre-treatment of the lignocellulosic material with electron beams can be used to reduce the recalcitrance of cellulose

High-level Biorefinery Concept I



High-level Biorefinery Concept II

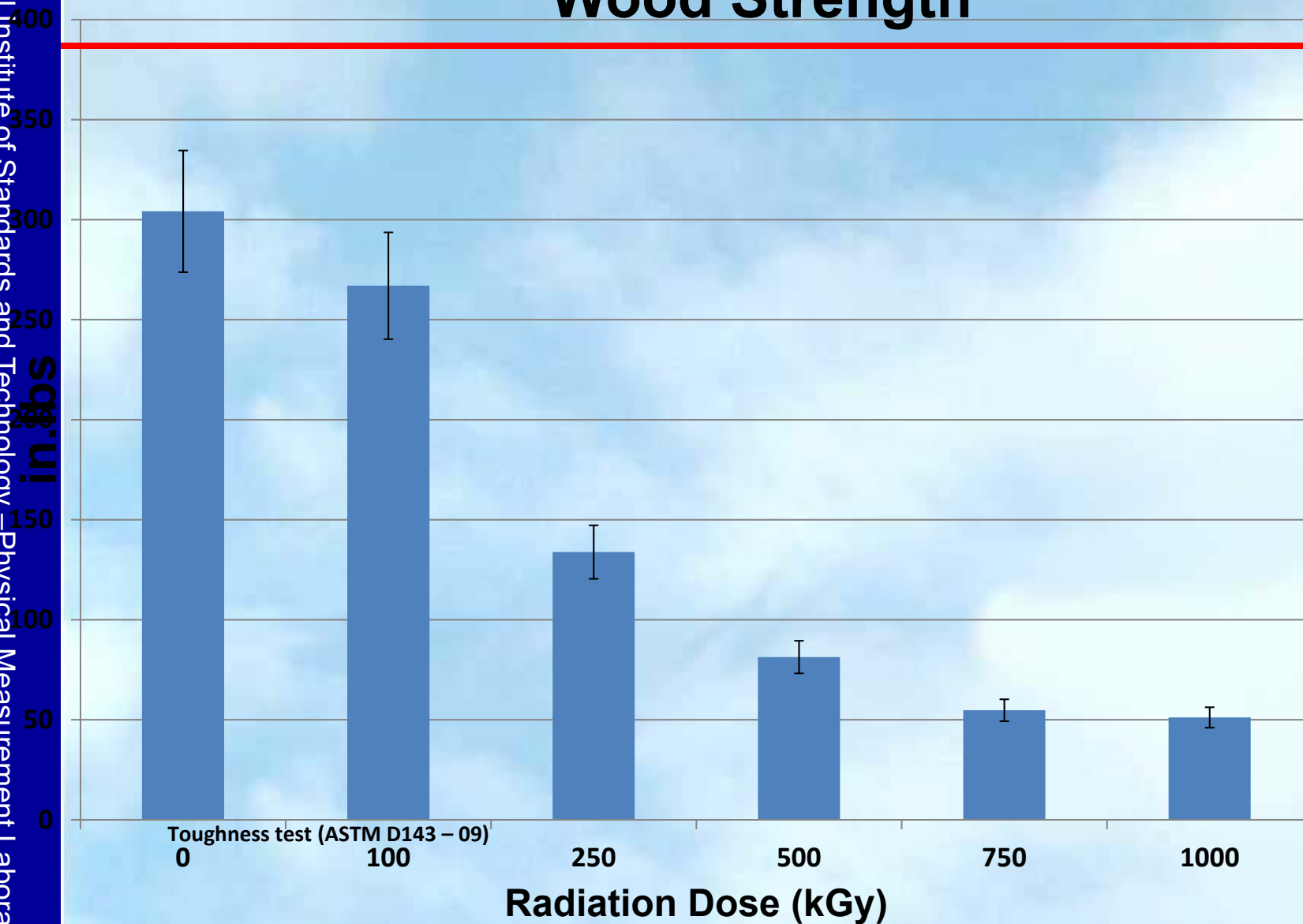


Electron Beam Irradiation of Wood

- **Electron beam irradiation of wood reduces the molecular weight and crystallinity of the cellulose and thus reduces the strength of the wood and the energy required to process wood for other uses.**



Effects of Electron Beam Irradiation on Wood Strength

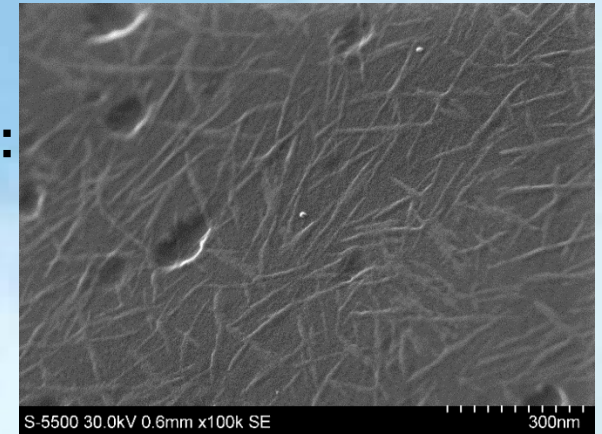


Advantages of Electron Beam Irradiation

- **Approximately 50% reduction in energy required**
 - Energy needed for maceration reduced due to reduced milling
 - Hot water/steam processing time reduced!
- **Removal of chemical/enzymatic step for some applications – e.g., biorefining:**
 - No additional sulfur groups added to the product
 - Provides the ability to control dose, to customize the particle size

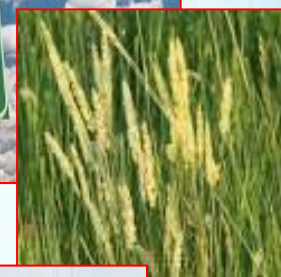
Nanocellulose

- **Nanocellulose is a high value material**
- **Gained increasing attention because of its:**
 - High strength,
 - Stiffness,
 - Unique Photonic and Piezoelectric Properties,
 - High stability,
 - Uniform structure.
- **Nanocellulose can be produced in large volumes from wood either by contemporary wet extraction or by application of radiation processing**
- **Ionizing radiation processing causes significant break down of the polysaccharide and, in parallel, leads to the production of potentially useful gaseous products such as H₂ and CO.**



Opportunities Abound!

- Application of radiation processing to the production of nanocellulose from:
 - wood
 - field grasses
 - biorefining by-products
 - industrial pulp waste
 - agricultural surplus materials
- **Remains an open field, ripe for innovation and application**
- Researching the mechanisms of the radiolytic decomposition of cellulose and the mass generation of nanocellulose is vital to tapping into nanocellulose for the growth of product development.
- **Successful application of ionizing radiation processing is key.**



Conclusion (1)

Electron beam irradiation

- **An environmentally friendly pretreatment method for bio-refining, production of biofuels, economically viable chemicals and nanomaterials from wood and non-woody sources.**
 - Decreases the energy needed
 - Improves the hot water extraction of lignin and hemicellulose.
 - Reduces the recalcitrance of cellulose.
 - Increases the rate of enzymatic hydrolysis.
 - Reduces the energy needed for milling.
 - Can be used to sterilize wood chips.

Conclusions (cont.): Strategic Alliances Imperative

- **Success requires strategic alliances between: Government, University, and Industry**
- **Advanced Manufacturing and Nanotech strong fit for such strategic alliances**
 - Multi-disciplinary nature
 - Enabling technology
 - Broad industry implications
 - Federal funding opportunities
 - Commercialization challenges
- **Cannot be done alone ...**
- **We welcome any further interactions**

THANK YOU!

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