



U.S. DEPARTMENT OF AGRICULTURE

— P³Nano —

Removing Barriers to Cellulose Nanomaterials Utilization

Robert J Moon
Materials Research Engineer

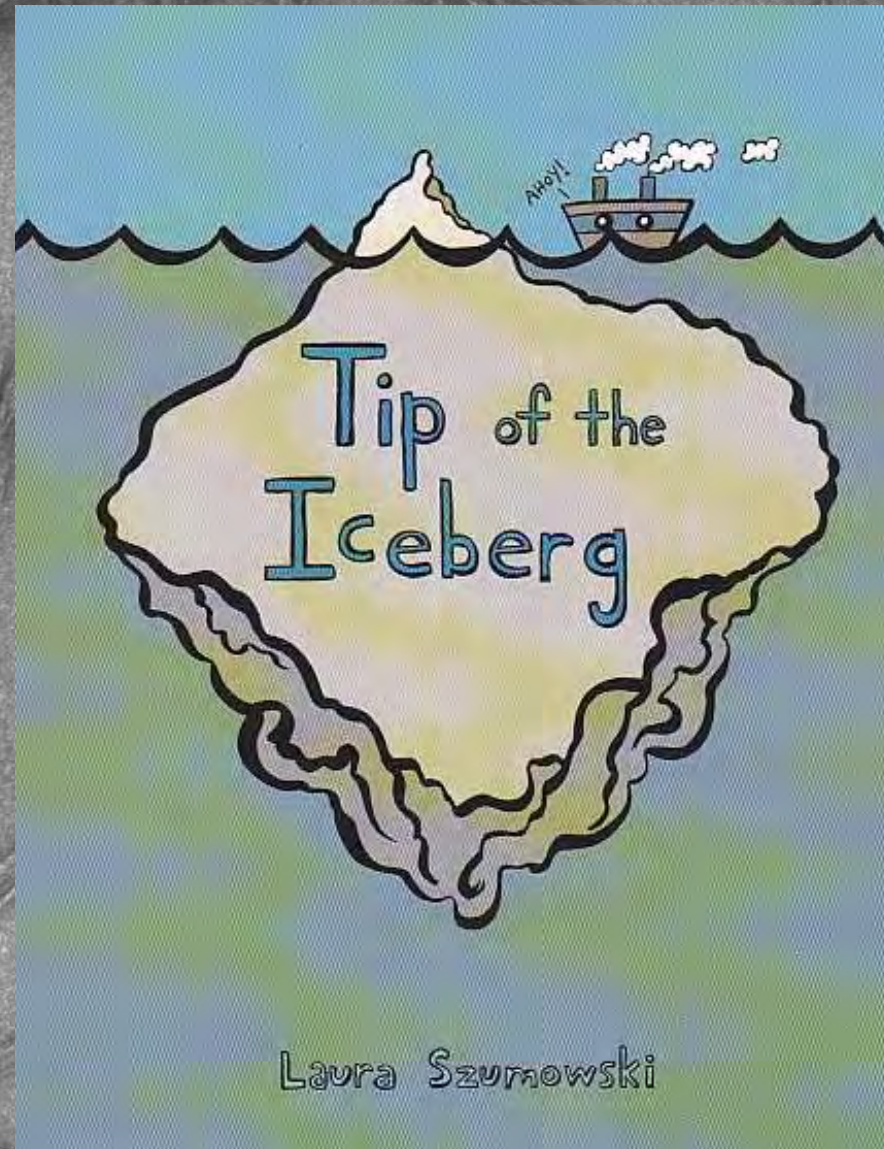
robert.j.moon@usda.gov



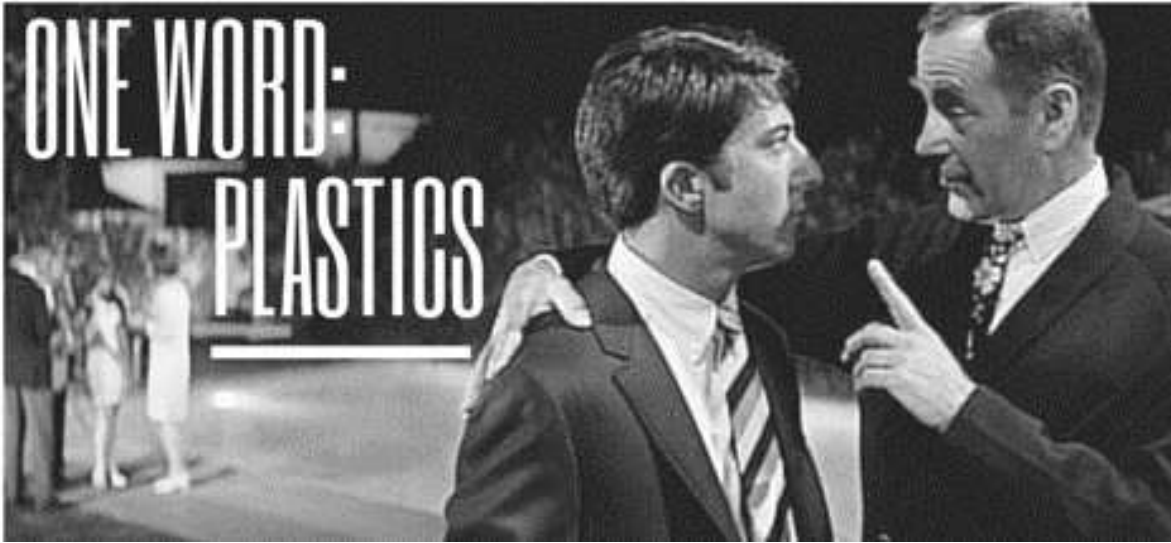
Forest Products Laboratory

2023 PDC Cellulose Nanomaterials Researchers Forum
University of Maine
24 August 2022

Nanocellulose is a Young Technology



200nm



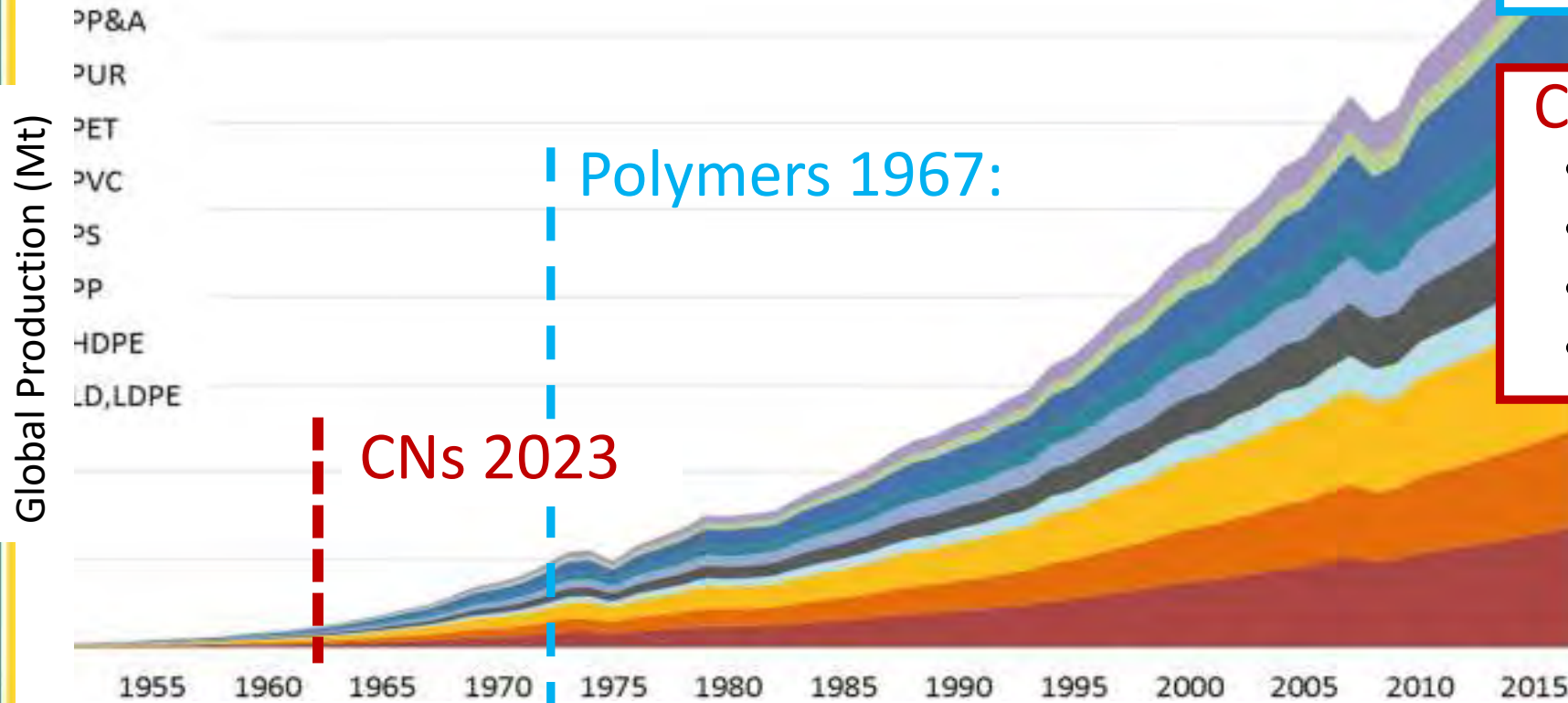
The Graduate - 1967

Polymers:

- 50+ years of R&D
- High funding levels
- High capacity
- Industry acceptance

CNs:

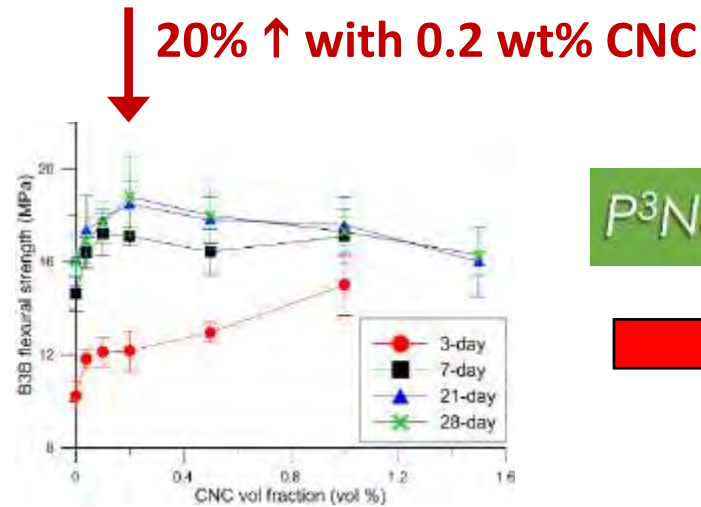
- 20+ years of R&D
- Low funding levels
- Low capacity
- Industry hesitancy



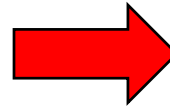
P³Nano: Accelerating Utilization

2011: CNC-Cement Discovery

2021: Bridge Deck Installation



P³Nano

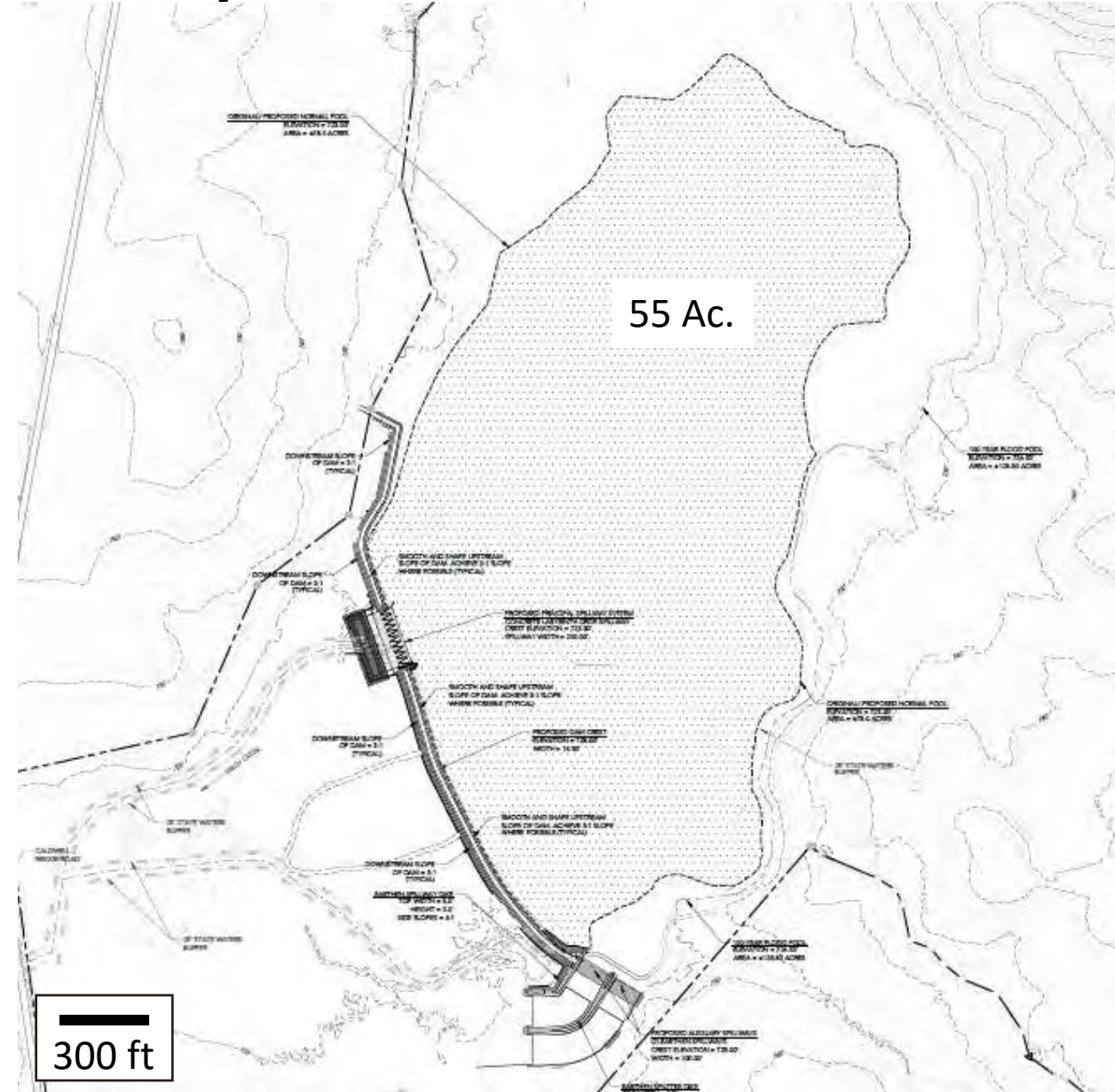
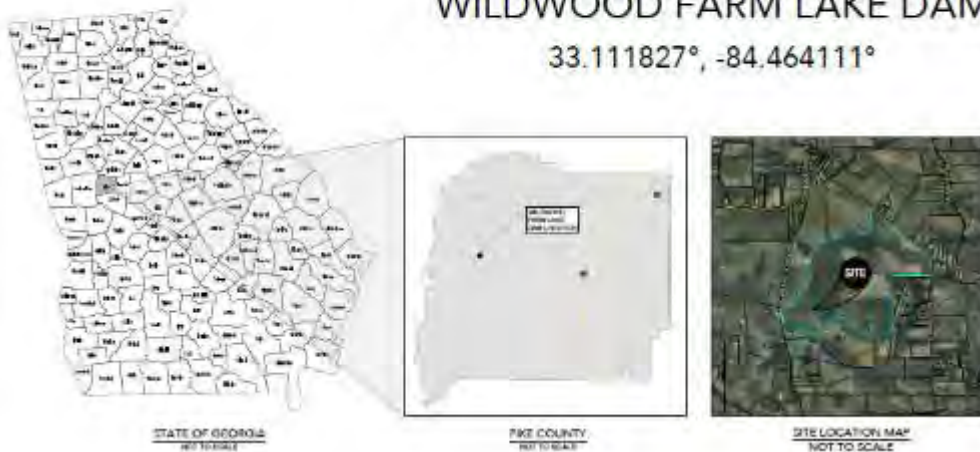


Cao, Zavaterra, Youngblood, Moon, Weiss
The influence of cellulose nanocrystal additions on the performance of cement paste.
Cement and Concrete Composites 56: 73-83. **2015.**



2024: Dam Spillway Installation

DAM RENOVATION PLANS FOR WILDWOOD FARM LAKE DAM 33.111827°, -84.464111°



P³Nano: Established in 2013

FPL Nano
Program
Timeline



2012 – CN Pilot Lines Running

- CNCs (FPL), Tempo-CNF (FPL),
- CMF (UMaine)
- 10s of Kg/week



P³Nano: a Public-Private Partnership

Government/Industry/Academia



USDA-Forest Service

Mission:

- Use *science & technology* to conserve & extend forest resources
 - Promote healthy forests
 - Promote forest-based economies

Emphasis Areas:

- Green Building/Construction
- Renewable Packaging
- Wood to Value
- Sustainability & Forest Carbon
- ..and more....

New Markets: *Cellulose Nanomaterials*

- Low quality wood → high value uses
- Many useful properties & function
- Reinvigorating utilization R&D



US Endowment

Mission:

- Keeping forests healthy and productive for people who depend on them

- Markets help us meet our mission
- New markets are essential for forest products

Project Areas:

- Mass Timber
- BioChar
- Wood to fuel
- Nanocellulose
- ..and more....

New Markets: *Cellulose Nanomaterials*

- Capture people's imagination of wood
- Excited about their myriad uses
- Reinvigorating Forest Service R&D → FPL

Partnership Contributions

USDA-FS FPL:

- Funding
- Strategic Planning
- Road Mapping
- Complete the R&D
 - Scientists, Technicians
 - Connections to academia
 - Connections to industry

US Endowment:

- 25-50% Funding Match
- Contracting & Management
- Advocate for FPL
- Connections: DC, State, Local
- Encouraging CN utilization
- Push to higher TR levels



FPL's Cellulose Nanomaterials Team

May 2022



19 years of CNM research



Forest Products Laboratory



Remove Barriers to Commercialization



**Fundamental
Science**
(Cool Stuff)

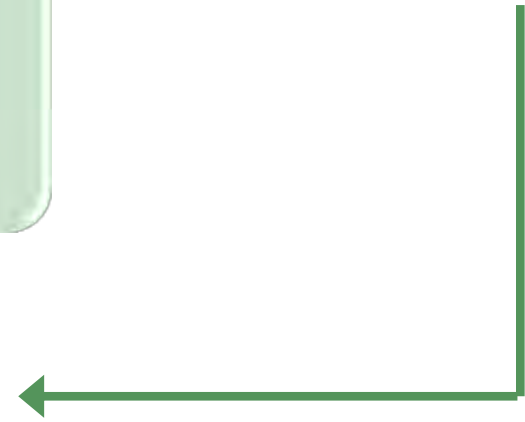
**Commercial
Product**
(Useful Stuff)

Technology Development:

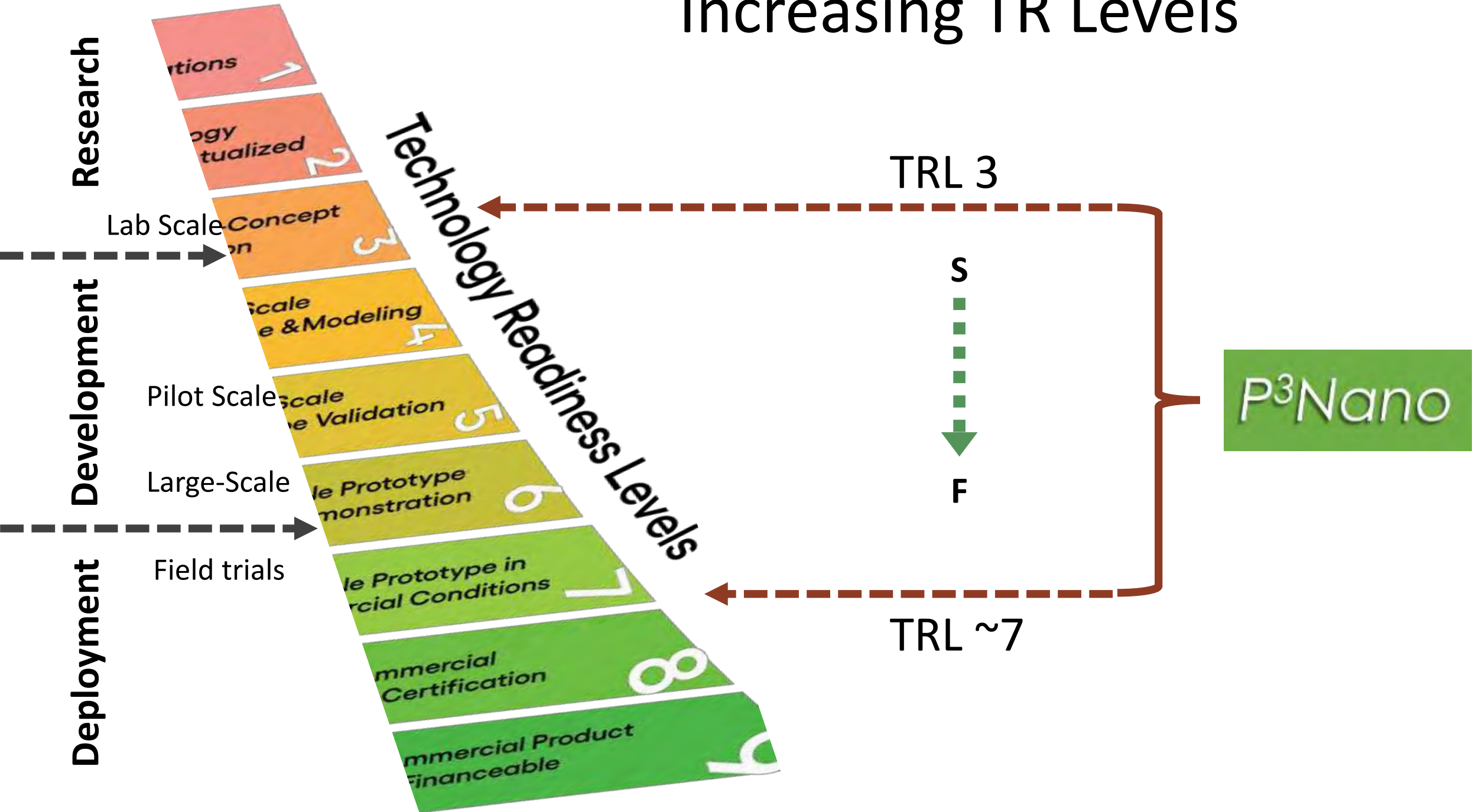
1. Develop proof of concepts
2. Develop applied technologies
3. Develop pilot scale capabilities
4. Risk Reduction
 - EHS
 - TEA/LCA
5. New application areas

**Needs
Assessment**

Increase Technical Readiness Levels



Increasing TR Levels



Drivers



USFS -Washington Office

- Need to show products & Jobs
- Need to show FPL having impact



↑ P3 Funding



US Endowment

- Need to show growing markets
- Need large volume applications



P³Nano by the Numbers: 2013-2023

Funding:

- USFS:
- US Endowment:
- Industry:
- Project Institutions:

~\$24 million in Total

~\$15 million

~\$8 million

~\$1 million

+ 20% cost share

P³nano Projects:

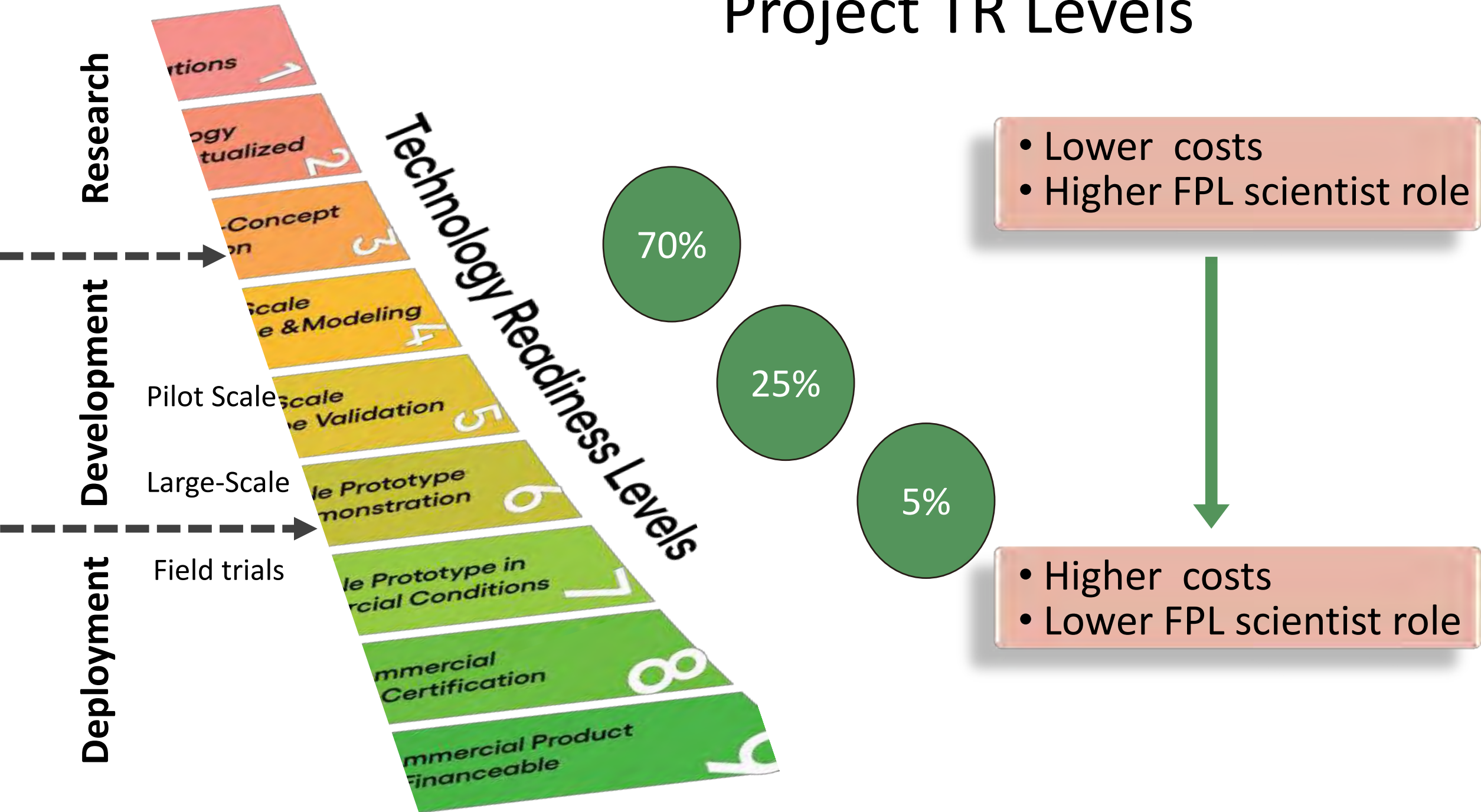
~60

Collaborating Institutions:

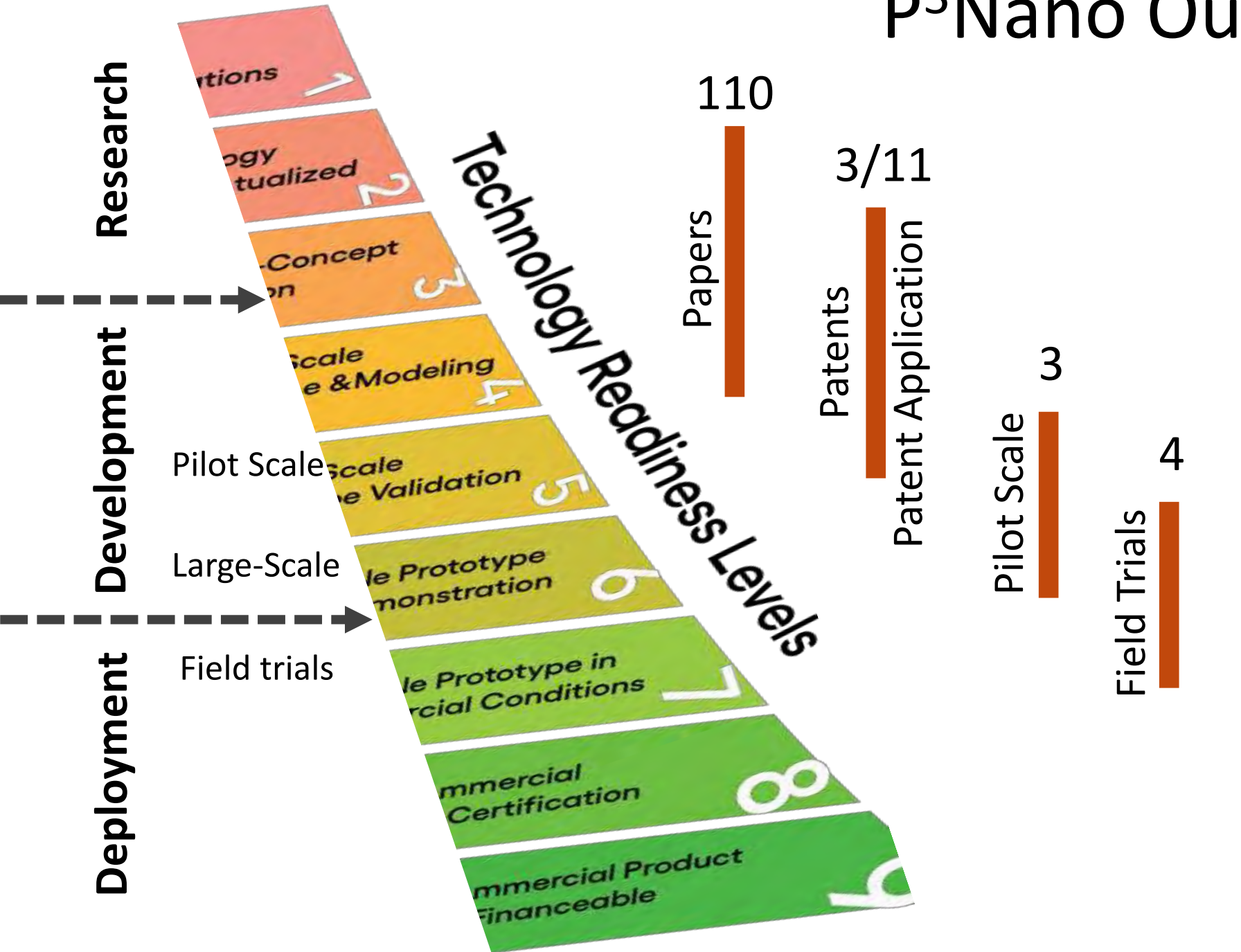
~40

- Academia: 21
- Industry: 15
- Federal Labs: 2
- Local gov: 2

Project TR Levels



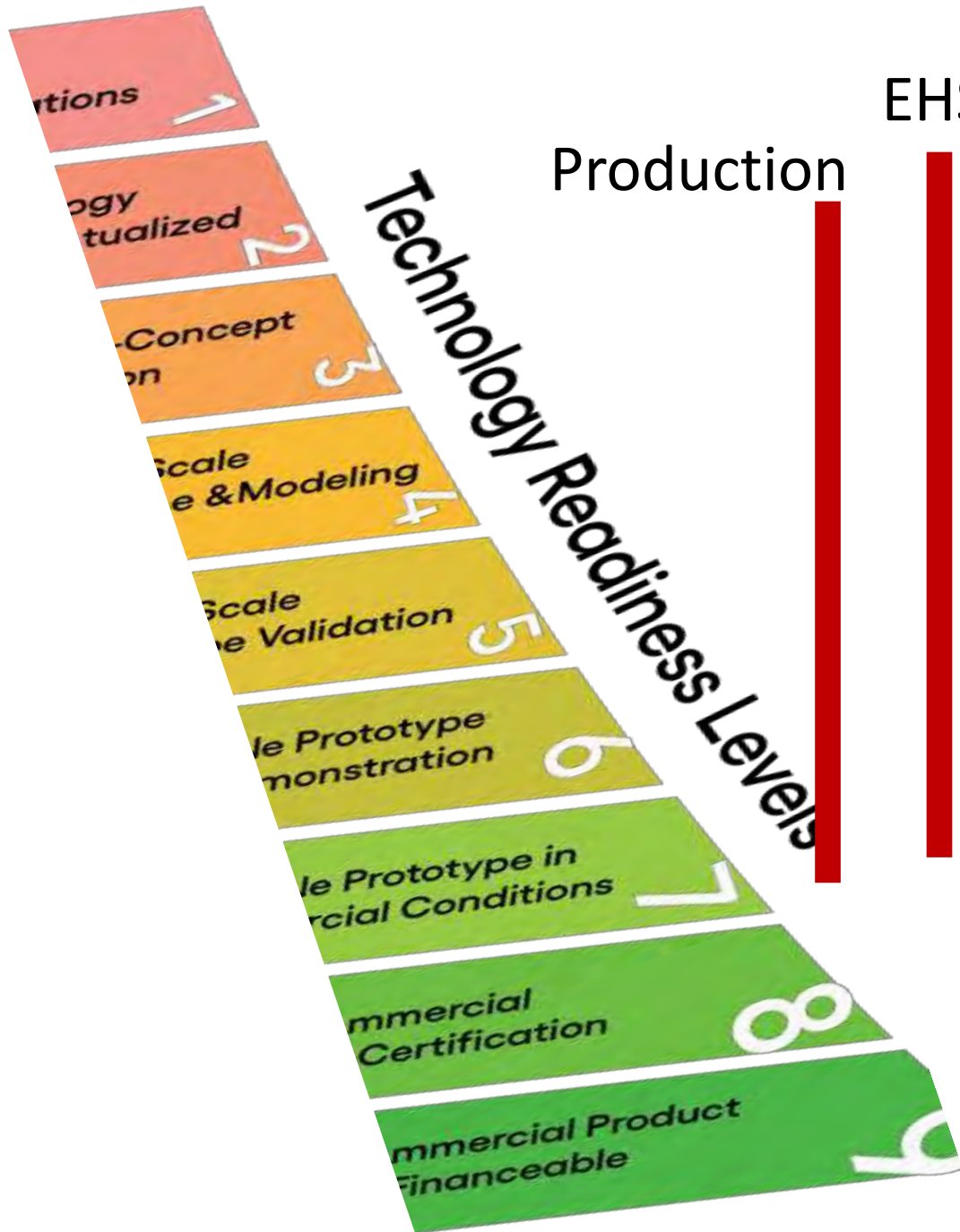
P³Nano Output



P³Nano – Focus Areas

1. Production
2. Risk Reduction:
 - a. EHS (Environment, Health, Safety)
 - b. TEA (Techno Economical Analysis)
 - c. LCA (Life Cycle Analysis)
3. Composites, Processing & Structures
 - coatings, films, fibers, laminates, etc.
4. Applications:
 - a. Cement/Concrete
 - b. Construction Materials (foams, boards, etc)
 - c. Packaging
 - d. Innovative

P³Nano Project TRL Overview



Production

EHS

Composites

TEA/LCA

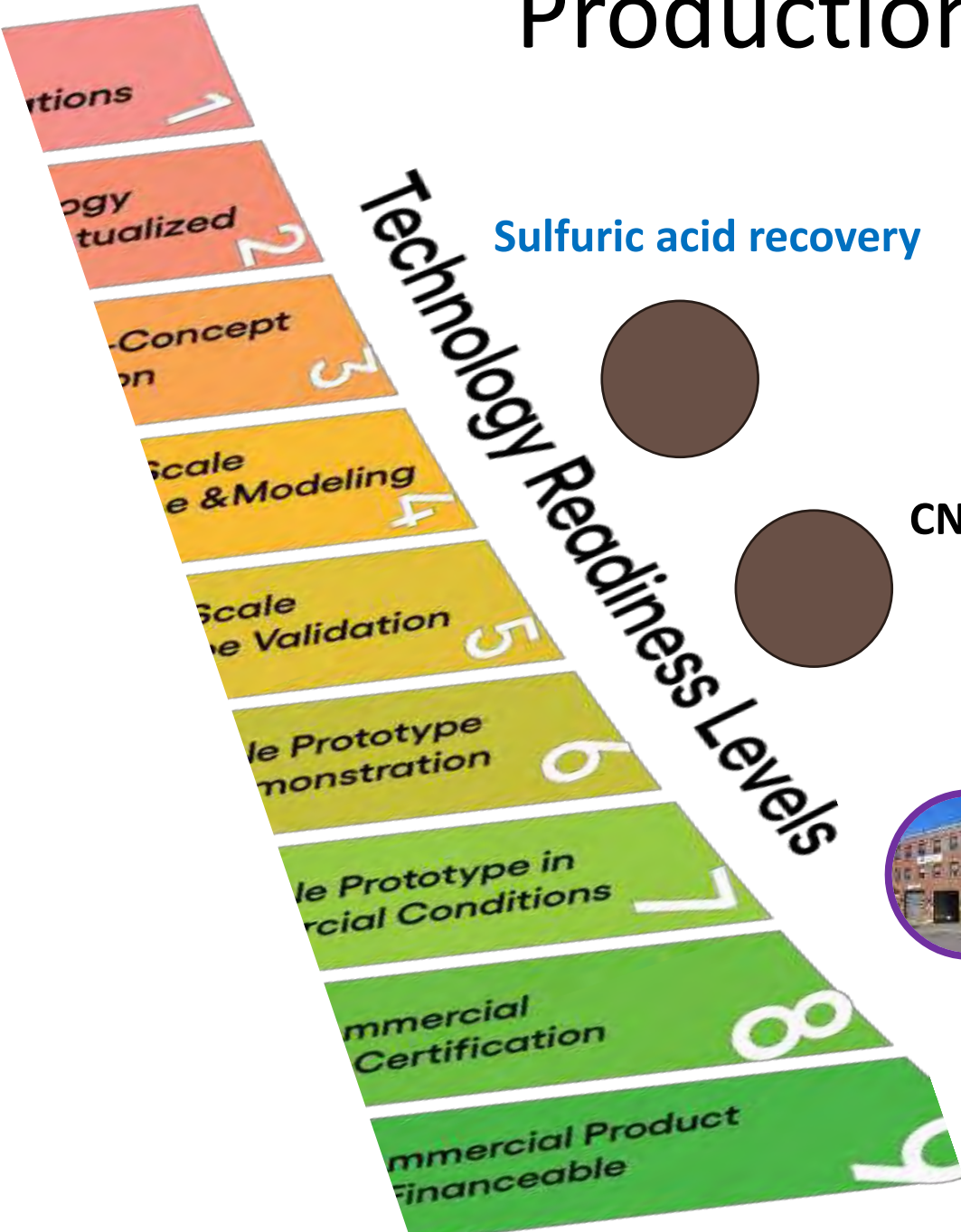
Packaging

Construction

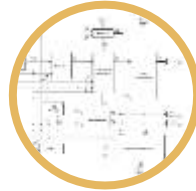
Cement

Production: CN, CN-Polymer

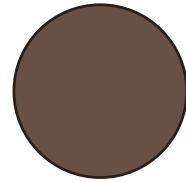
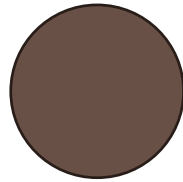
Technology Readiness Levels



Sulfuric acid recovery



50mt/day CNC & CNF
Engineering study (Class 4 & Class 5)



CN Dryer at FPL



Trees to Tires
Nanocellulose Dispersion Composite



CNF Manufacturing Plant:
Turner Falls mills



CNF/Validia
Sappi

EHS



Techn

Air Sampling CN detection

Toxicity testing

Thursday, August 24, 2023 Wells Conference Center, Room 100	
8:00 – 8:30 AM	Continental Breakfast, Coffee
8:30 – 9:15 AM	Opening Keynote: “Safety and Regulatory Aspects of Cellulose Nanomaterials: Challenges and Needs” Jo Anne Shatkin, Ph.D., President, Vireo Advisors



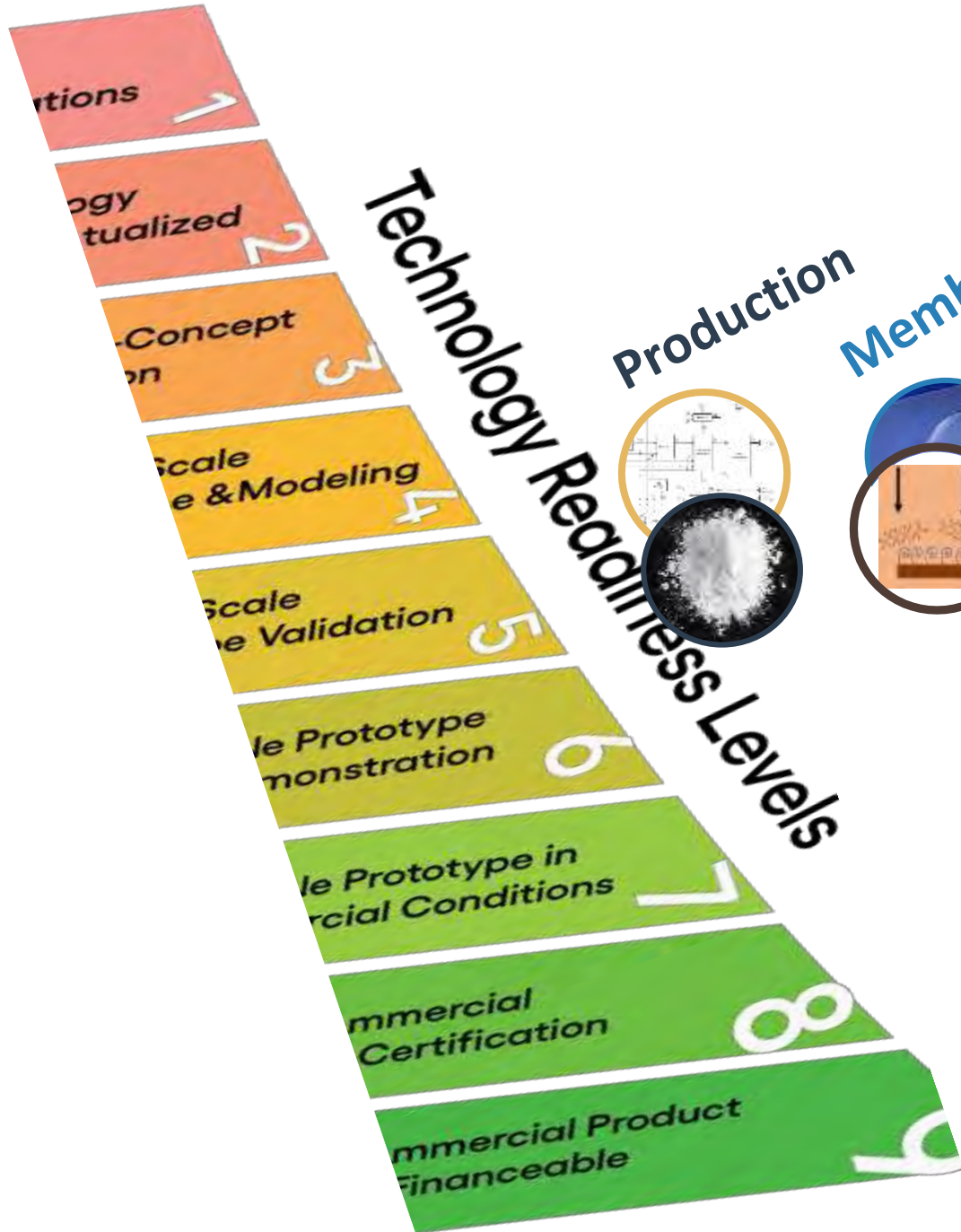
CN Materials Safety Data Sheet Templates



TEA/LCA



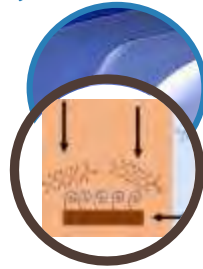
Technology Readiness Levels



Production



Membranes & Filters



Packaging



Fruit Coating



CN Concrete

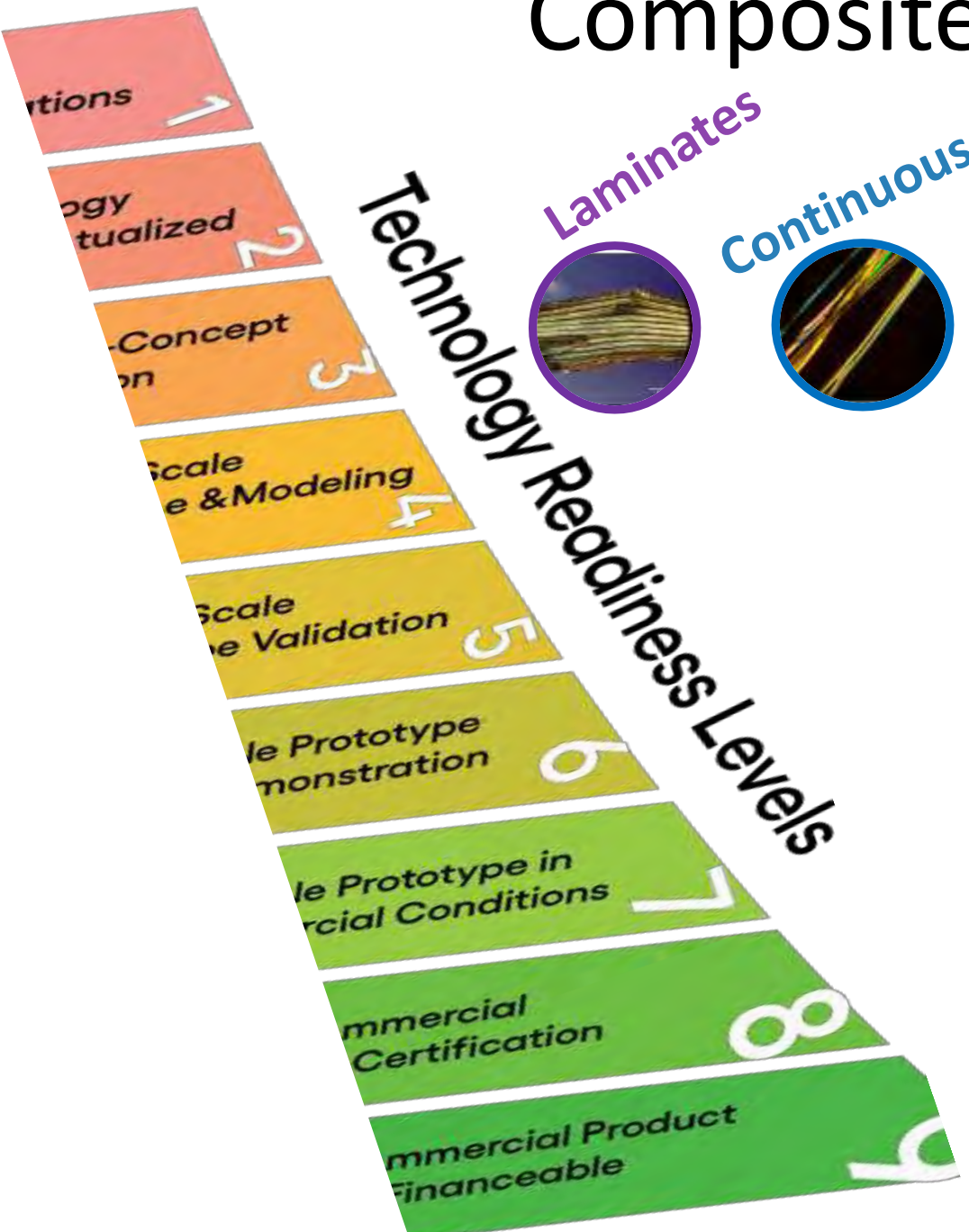


Equipment



Composites, Processing & Structures

Technology Readiness Levels



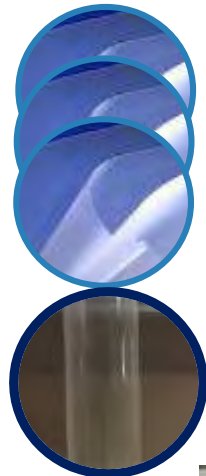
Laminates



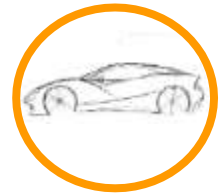
Continuous Fibers



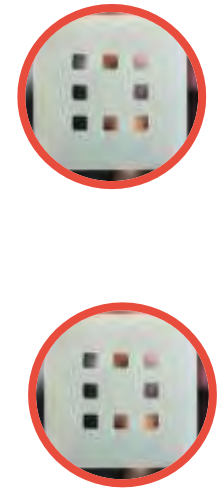
Coatings & Films



Automotive

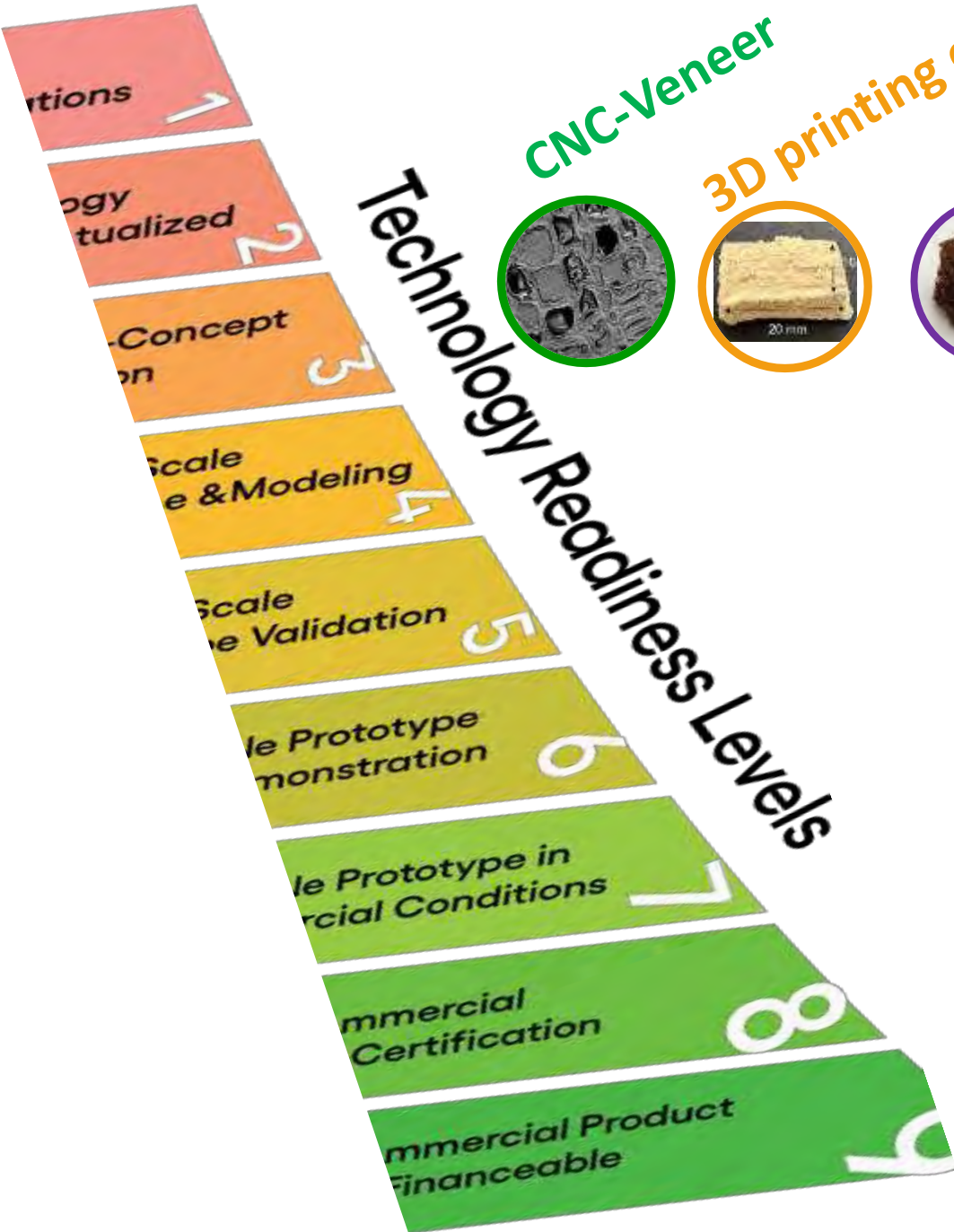


Fiber Glass

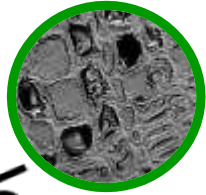


Construction Materials

Technology Readiness Levels



CNC-Veneer



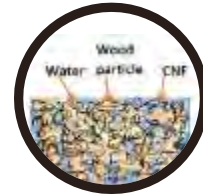
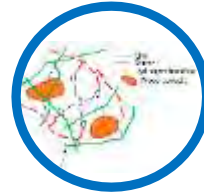
3D printing of CNF



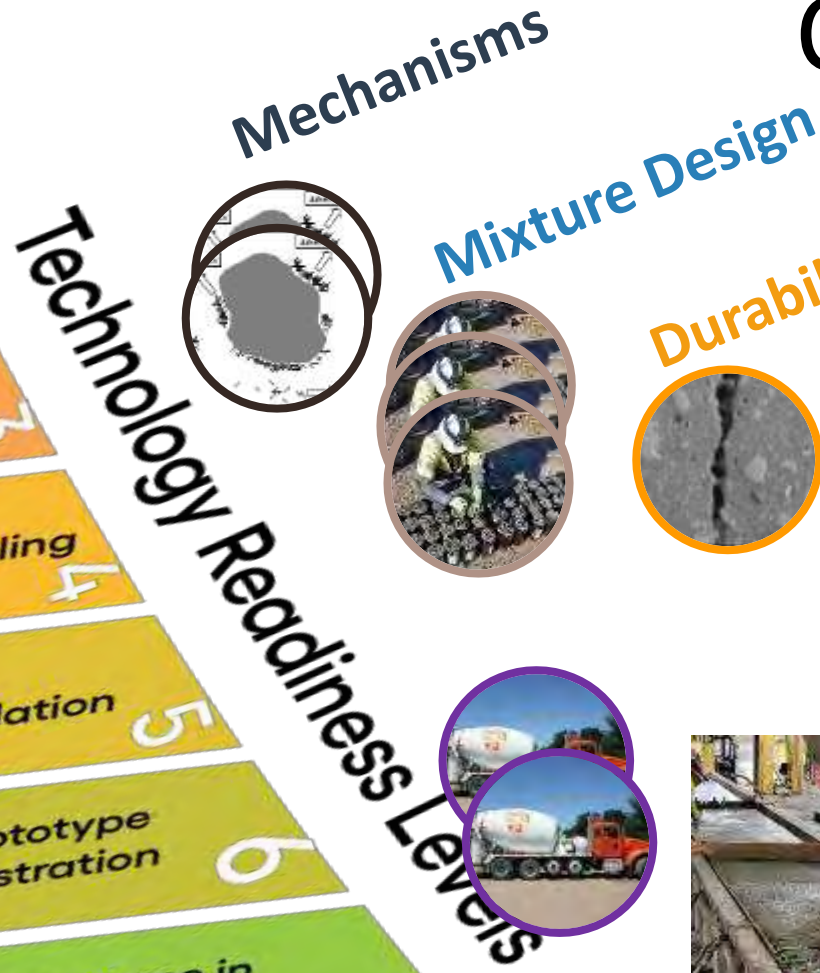
CN-PU Foams



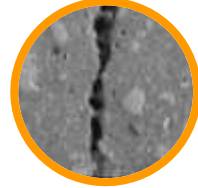
CNF-Fiberboard



Cement/Concrete



Durability



CO₂ Reduction



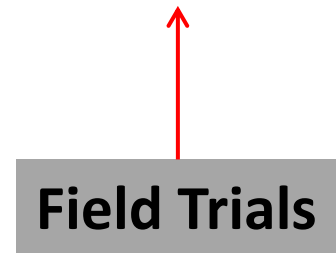
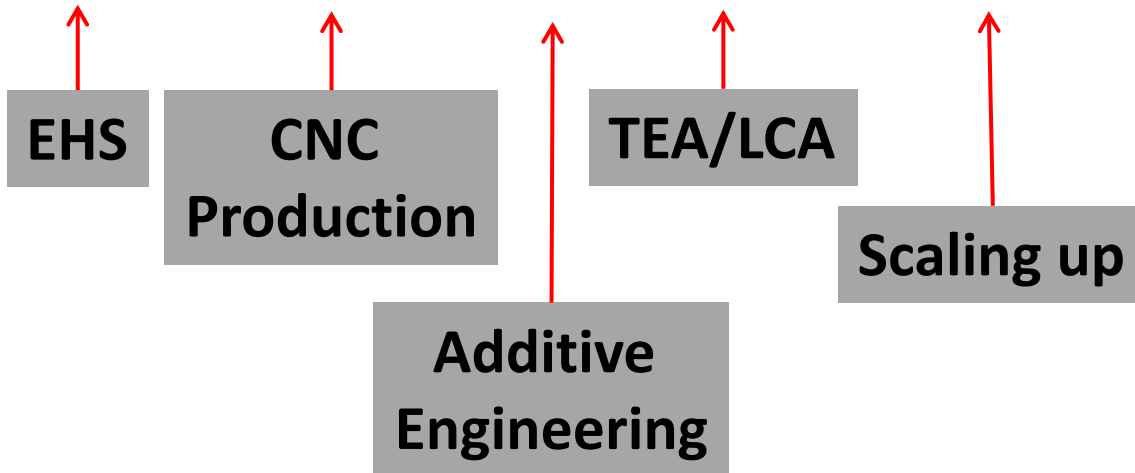
3D Printing



Full Scale Installations



Strategic Targeted Effort

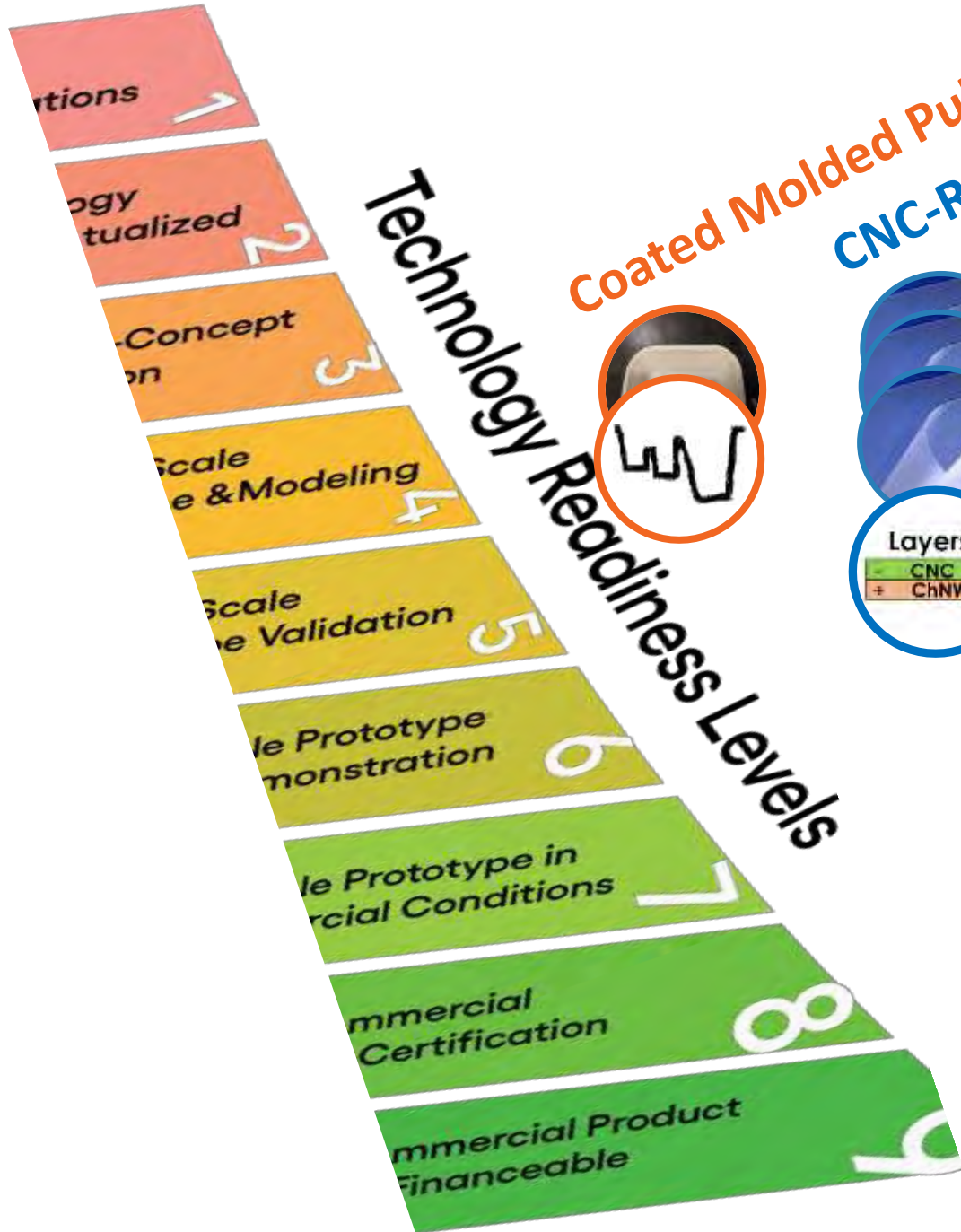


Ramifications

- Faster progress to TRL 7 for the application

Packaging

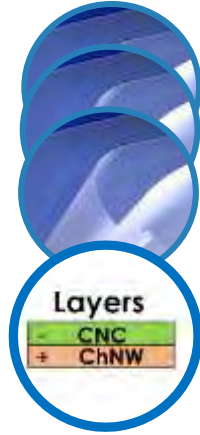
Technology Readiness Levels



Coated Molded Pulp



CNC-Resin films



Layers
- CNC
+ ChNW

Lidded Trays



Fruit Coating

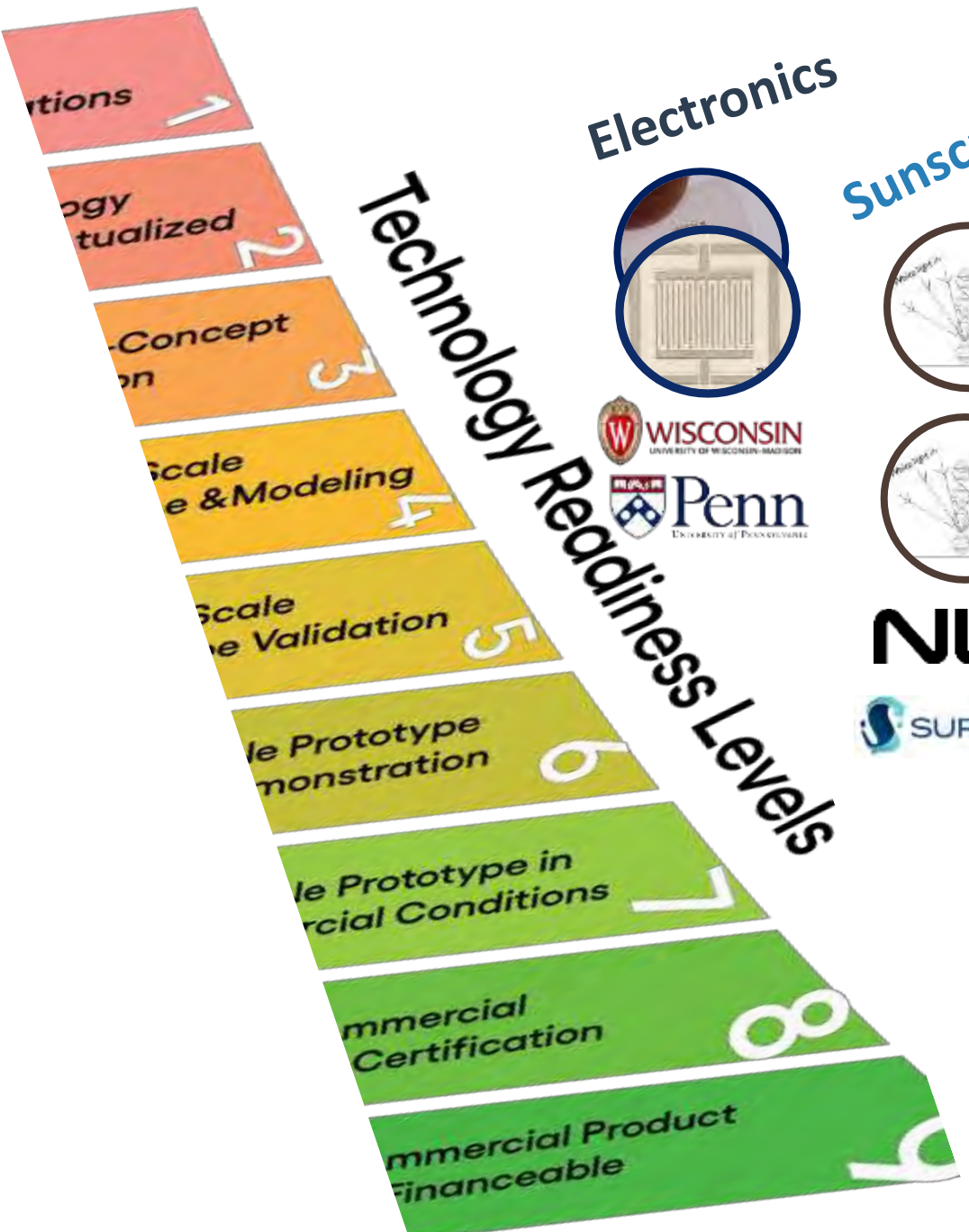


Paper Additive

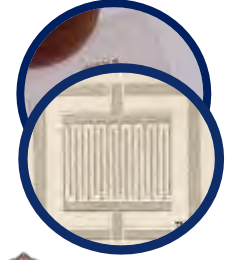


Unique Applications

Technology Readiness Levels



Electronics



Sunscreen



NIST



Batteries



Tires



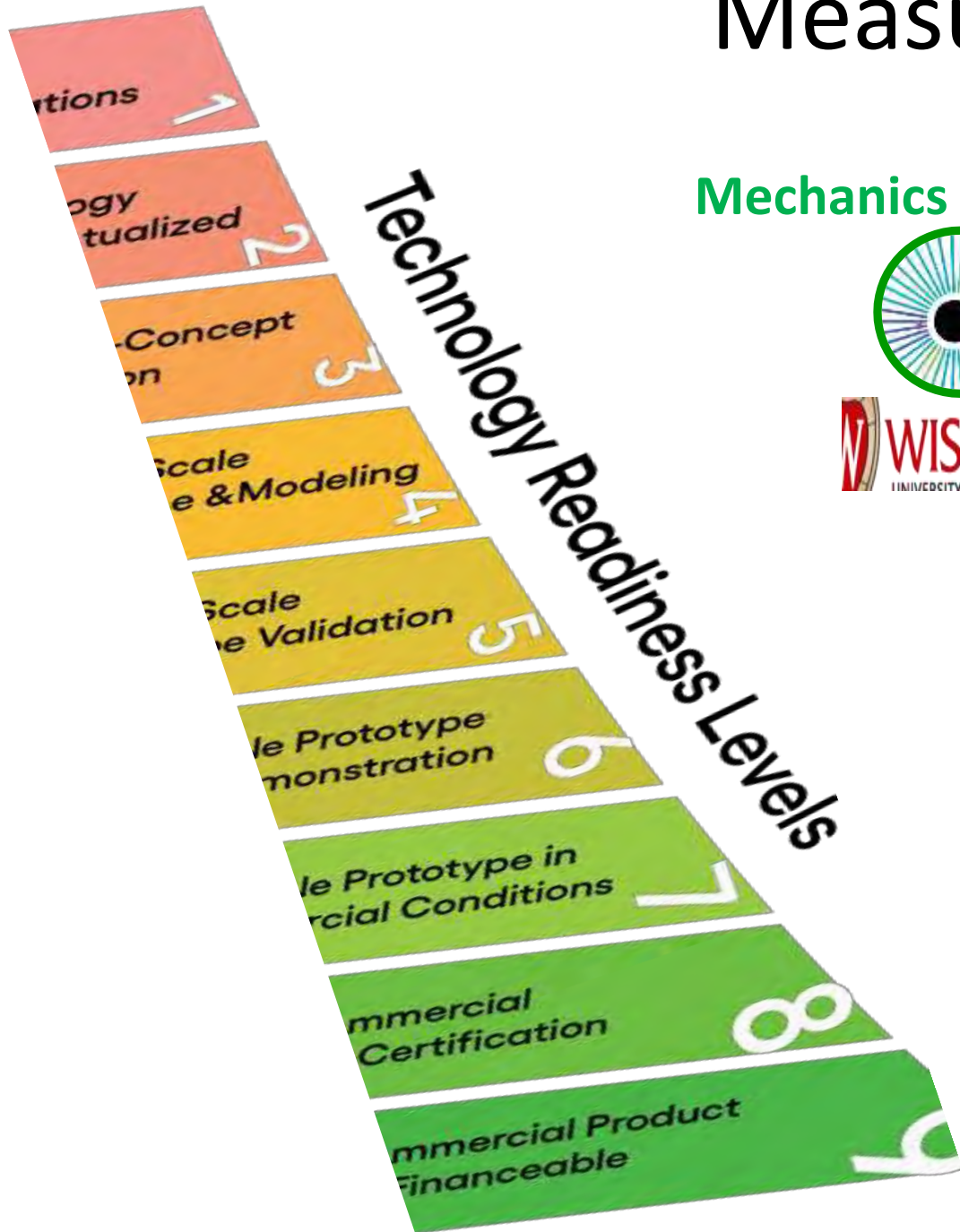
Drilling Fluids



Frost Protection



Measurement Development



Mechanics of CN-Hydrogels



CNC Particle Size



2022 Funding: \$1.75M USFS + \$400K En

Development (~\$500K for 3 years)

- | | |
|---------------------------|--------|
| 1. CNF-tableware | UMaine |
| 2. Lidded trays | Purdue |
| 3. CNC-barrier packaging: | GT |



Exploratory (~\$100K for 1 year)

- | | |
|---------------------------|------------|
| 1. CNF hydrogels | UW-Madison |
| 2. CNC-based filter media | SUNY |
| 3. CMF-paper packaging | SUNY |
| 4. CNC-veneer | MSU |



2023 Funding: \$1.75M USFS + \$400K End

Development (\$300 -\$500K for 3 years)

1. CNC-PU Foams at industrial scale
2. CNC size measurement
3. CN-paper substrates for electronics
4. Scale up CNC-composite food Coatings

Uni Delaware

GaTech

UPenn

Rice



CNF-biopolymer PFAS-free barrier thermoformed fiber tableware





Coated Molded Pulp



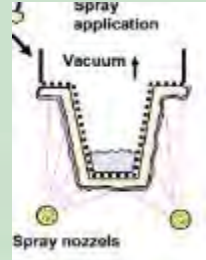
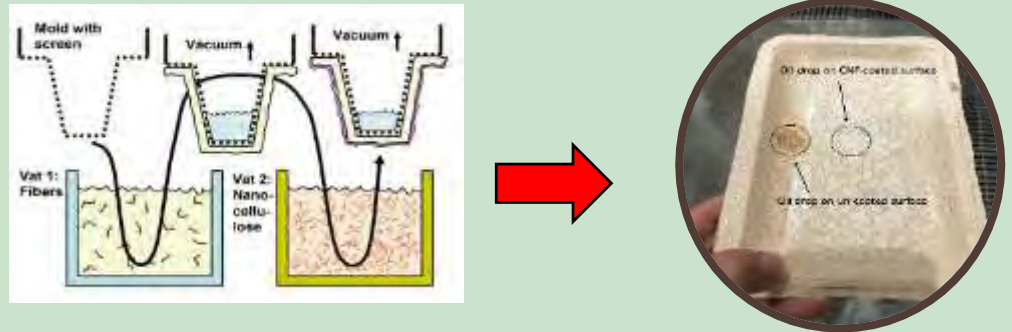
- Packaging
- TEA/LCA

Development Project:

- Jinwu Wang (FPL)
- Colleen Walker (UMaine)

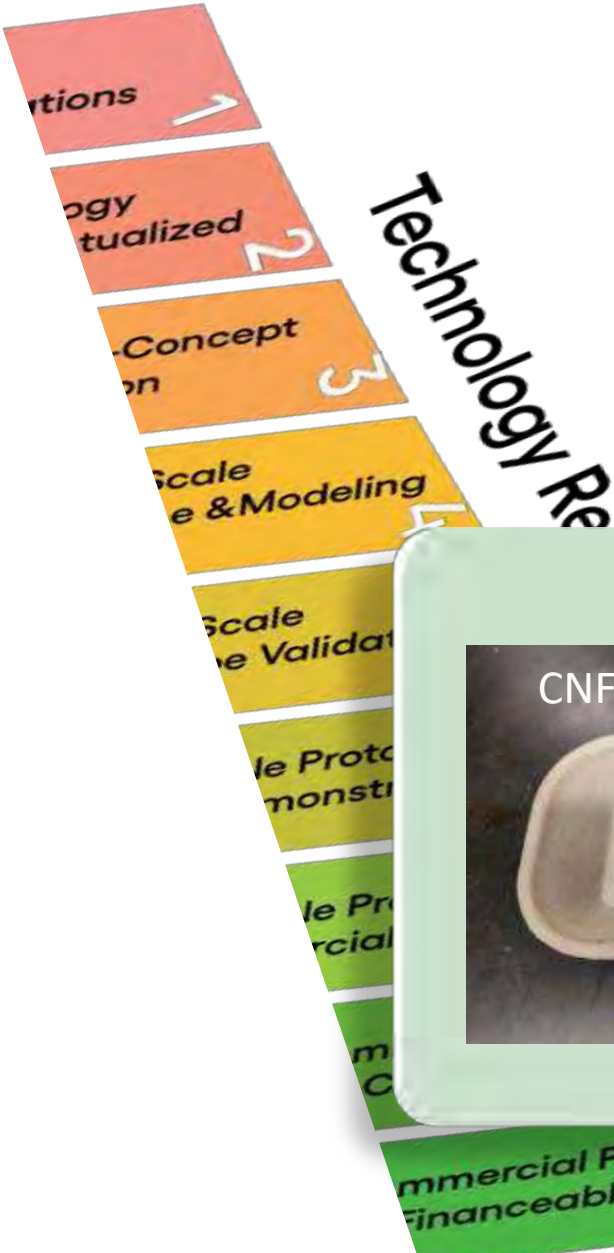


Kiefel pulp molding machine



- Optimize CNF coating
- Pilot-System
- OTR, WVTR
- TEA/LCA

Sustainable Lidded Rigid Trays for Food Packaging



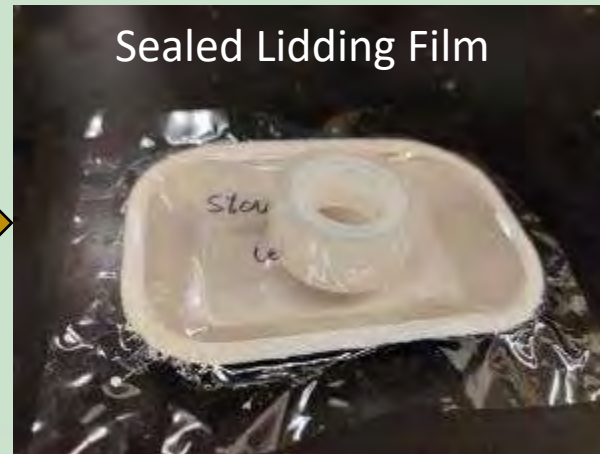
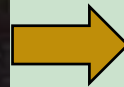
Coated Molded Pulp



- Packaging
- TEA/LCA

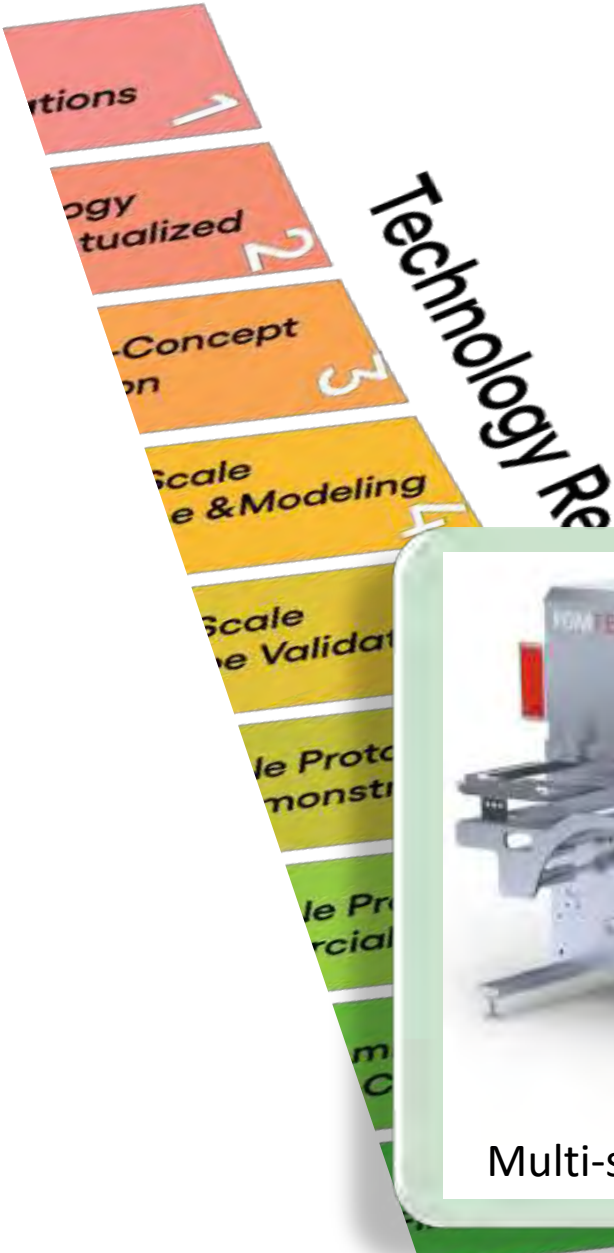
Development Project:

- Jeff Youngblood (Purdue)
- Greg Schueneman (FPL)

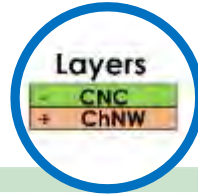


- Optimize CNF coating
- Optimize CN lidding film
- Optimize lidding seal
- OTR, WVTR
- Food preservation testing
- TEA/LCA

CNC Utilization in Renewable Barrier Packaging



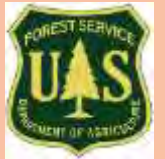
Coating/Films



- Packaging
- TEA/LCA

Development Project:

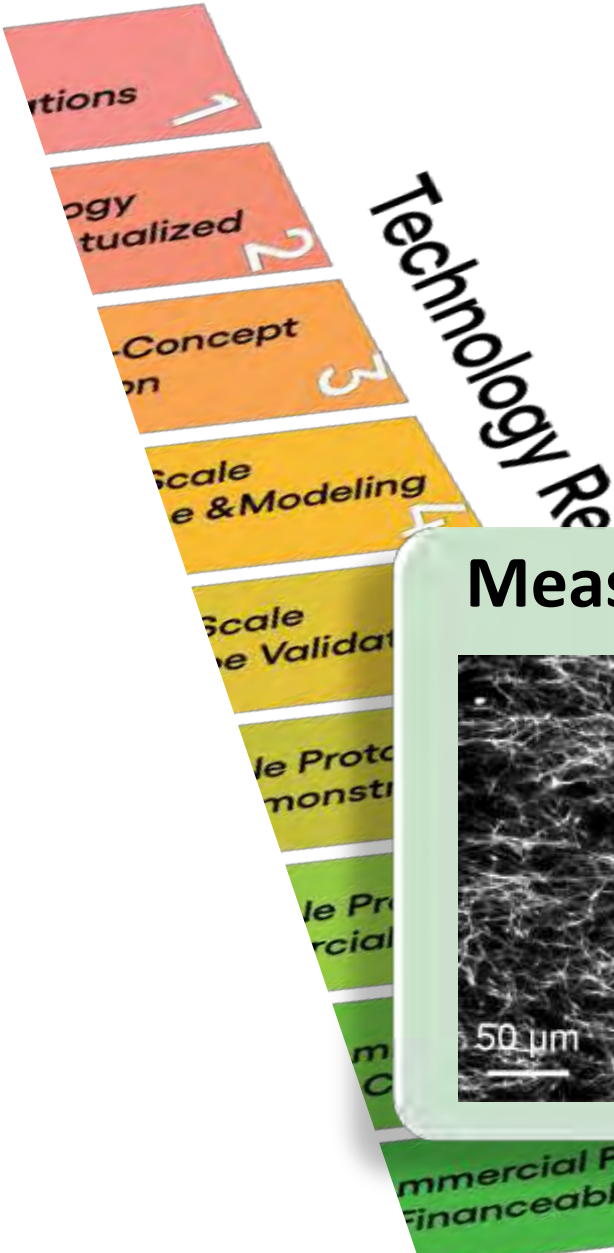
- Carson Meredith (GaTech)
- Greg Schueneman (FPL)



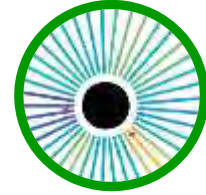
Multi-station coater

- Demo scalable coating
- chitin nanowhisker (ChNW)
- ChNW/CNC layers
- Heat sealing
- Increase WVTR
- Assess Biodegradability
- TEA/LCA

Methodology for Microscale Mechanical Characterization of CNF Hydrogels



Measurement



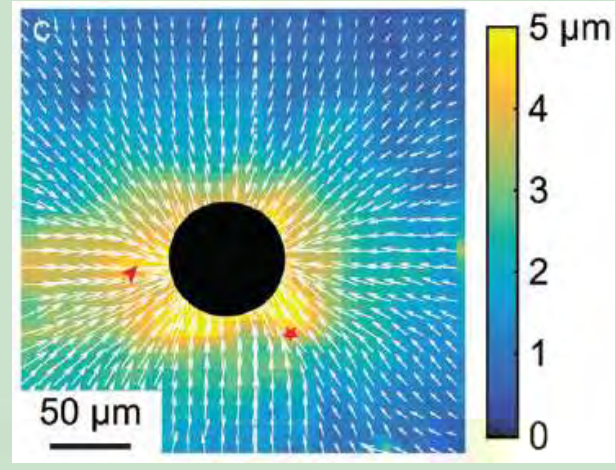
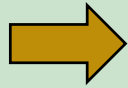
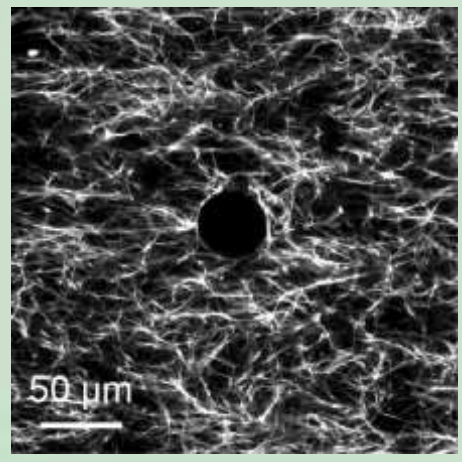
- Composites

Exploratory Project:

- Jacob Notbohm (US-Madison)
- John Considine (FPL)



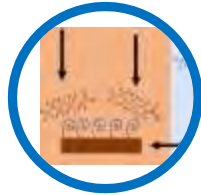
Measure Displacement



- Solves gap in capability
- Deformation
- Stiffness
- Strain localization
- Anisotropy

CNC-based Smart Advanced Filter Media for PFAS Removal from Contaminated Water

Membrane/Filter



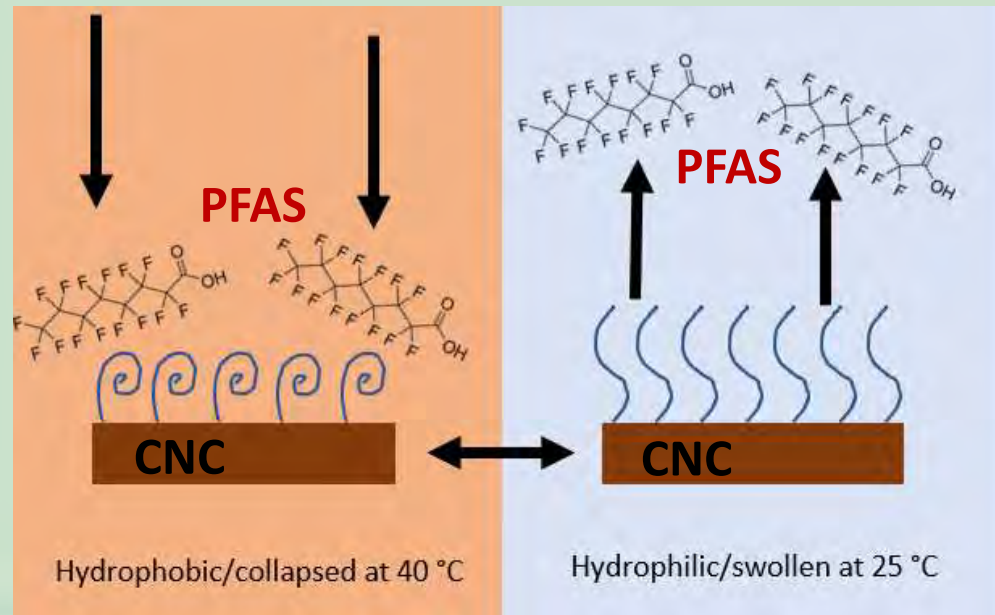
- Packaging
- TEA/LCA

Exploratory Project:

- Deepak Kumar (SUNY)
- Richard Bergman (FPL)

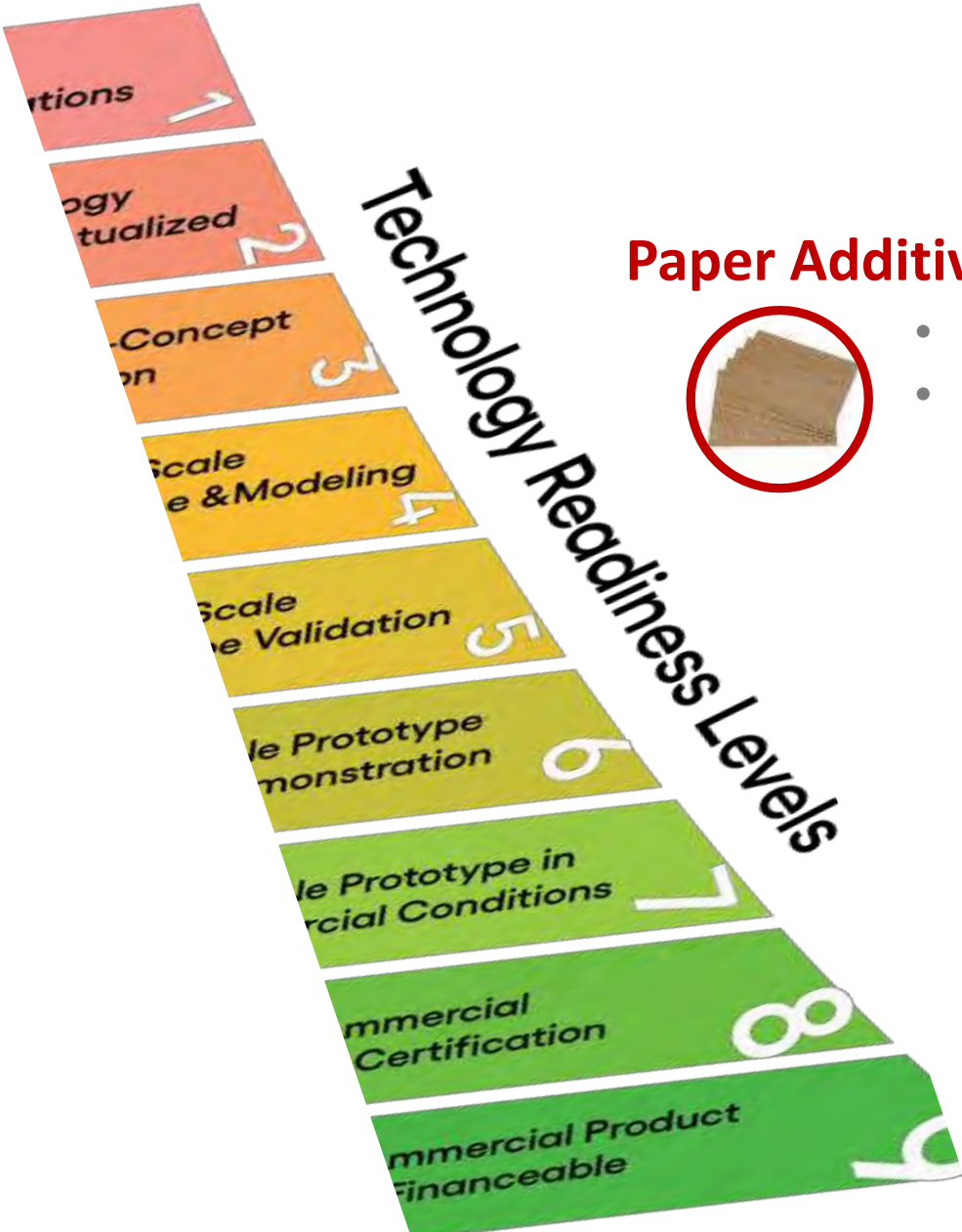


Technology Re



- Proof of concept
- Produce PNIPAM/PDA/CNC
- CNC-based filter media
- Test PFAS capture & release
- TEA/LCA

Lignin Coated (LC-) MFCs for recycled packaging paper



Paper Additive



- Packaging
- TEA/LCA

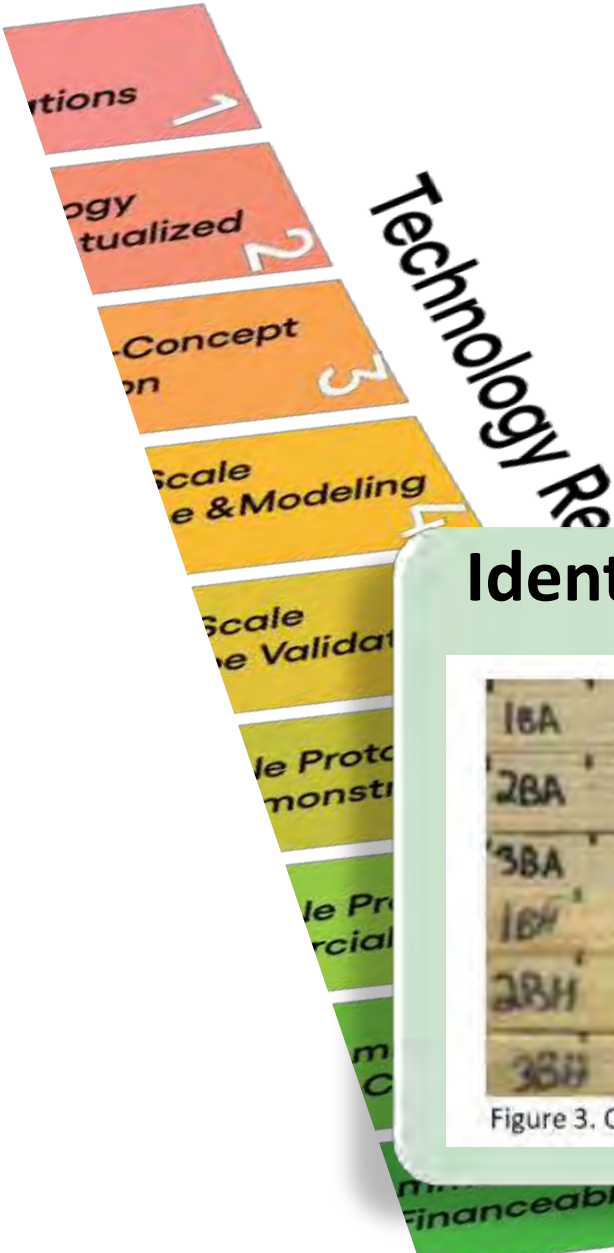
Exploratory Project:

- G.M. Scott (SUNY)
- Biljana Bujanovic (FPL)



- Produce LC-MFC
- LC-MFC as additive
- Linerboard
- Assess mechanical props
- Assess water resistance
- Assess Biodegradability
- Hand Sheets
- Scale up to Pilot line
- TEA/LCA

Enhancing Wood Veneer by Impregnating with CNCs



CNC-Veneer



- Construction

Exploratory Project:

- Dilpreet Bajwa (MSU)
- Nicole Stark (FPL)



Identify mechanism for enhance properties

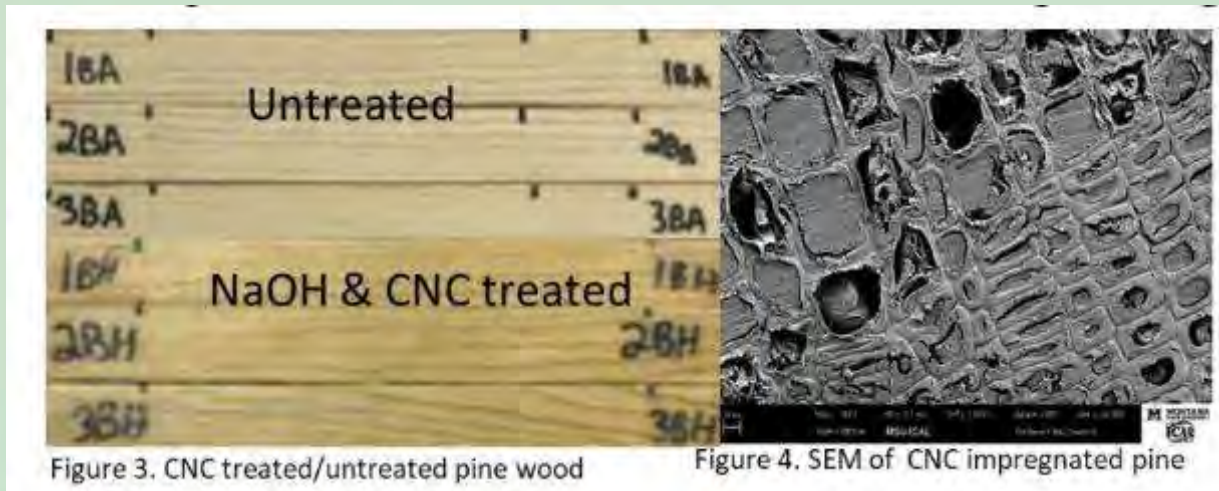
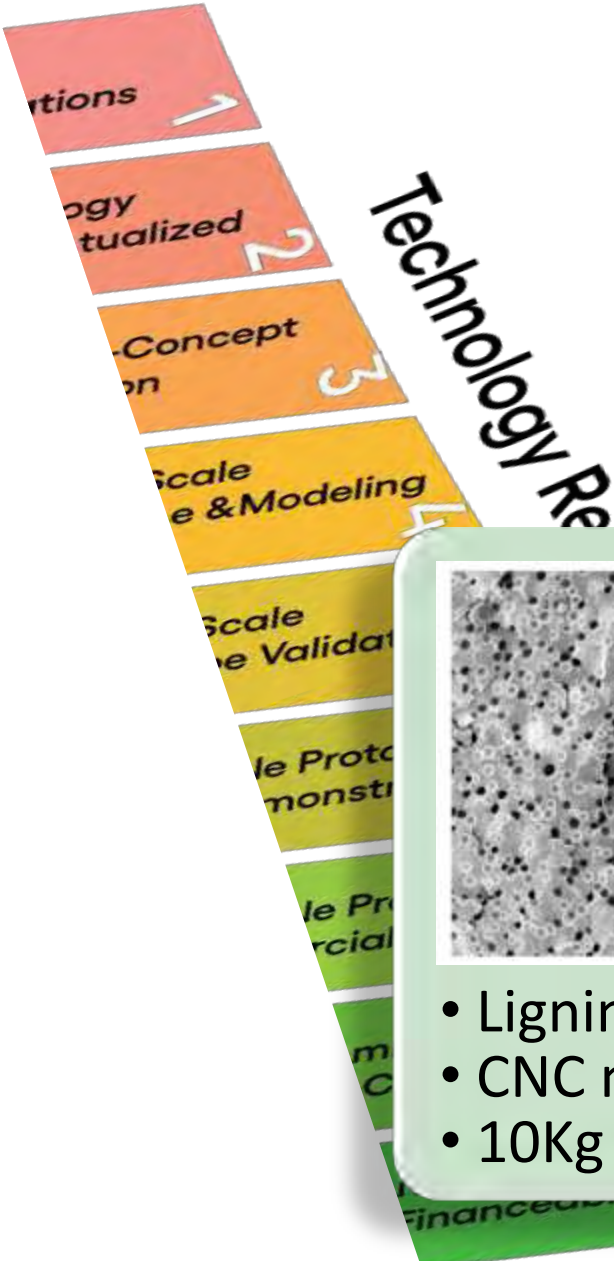


Figure 3. CNC treated/untreated pine wood

Figure 4. SEM of CNC impregnated pine

- Wood flooring application
- Early wood veneers
- Maple, SYP
- Infuse with CNCs
- Evaluate location of CNCs
- Mech & durability testing
- Nanoindentation

CNC-PU Foams at industrial scale



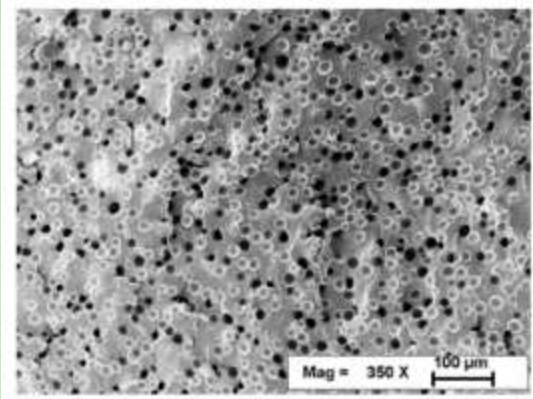
Foams



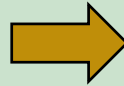
- Construction
- Packaging
- TEA/LCA

Development Project:

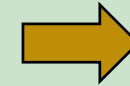
- Srikanth Pilla (Uni Delaware)
- Biljana Bujanovic (FPL)



- Lignin based foams
- CNC nucleation agent
- 10Kg master batch

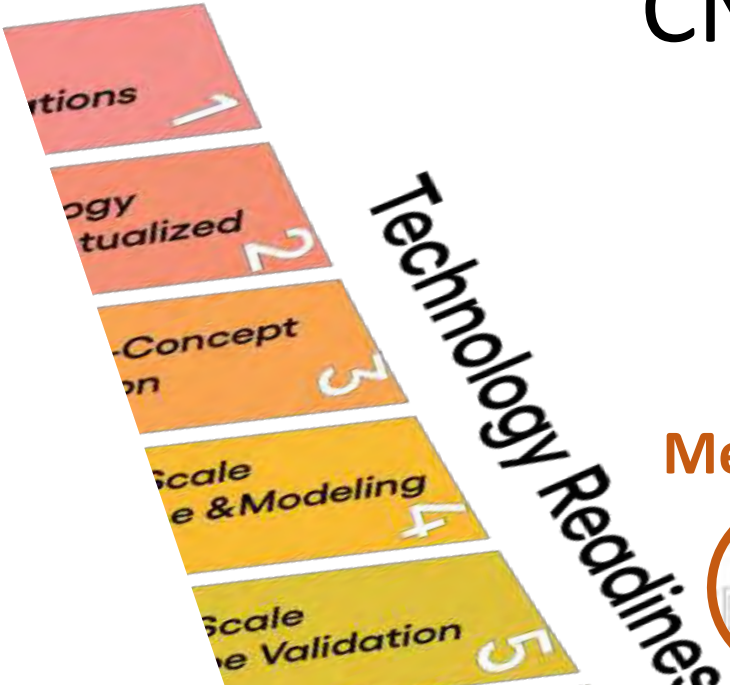


- Rigid Foam
- Flexible Foam





- 1ft x 1ft panels
- Prototype container
- TEA/LCA

Development of a rapid and robust instrument for CNC size distribution measurement

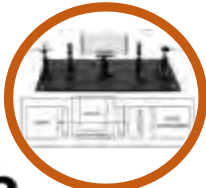


Development Project:

- Victor Breedveld (GaTech)
- Robert Moon (FPL)

Georgia Tech  

Measurement



- Production
- TEA/LCA

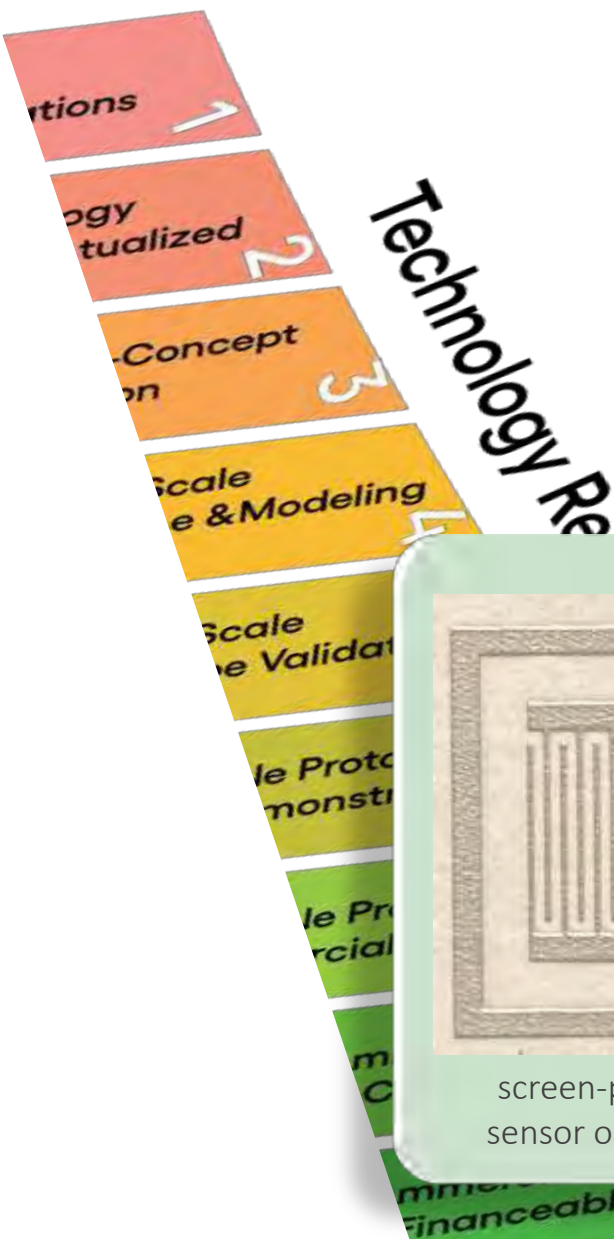
Zero-Angle Depolarized Scattering (ZADS)

Lab-Scale: ZADS today

Prototype I & II Concept: ZADS 2023-2025 (two copies: RBI @ Georgia Tech and FPL)

- More accurate than DLS
- Robust, simple to run
- Prototype I & II
- Validate at FPL
- TEA/LCA
- \$15,000 Price

Nanocellulose infiltrated paper substrates (NCIP) for printed sensors and electronics



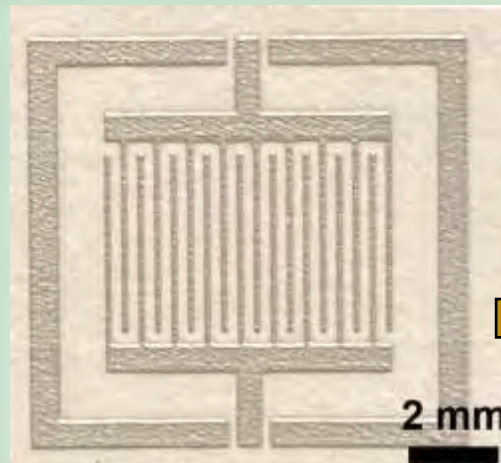
Electronics



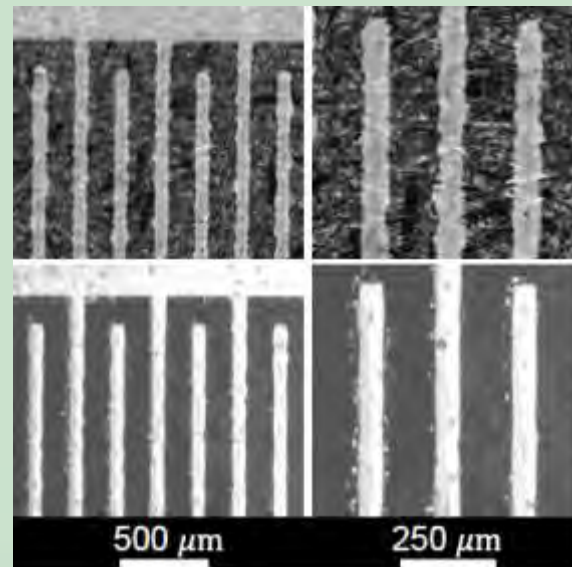
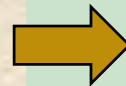
- Packaging
- Moisture sensor

Development Project:

- Kevin Turner (UPenn)
- John Considine (FPL)



screen-printed capacitive sensor on a NCIP substrate



cardstock

NCIP

- Scale up substrate fab
- Moisture sensors

Scaling up CNC-Agroprotein Nanocomposite Coating for Perishable Fruits



Coating/Films



- Packaging
- TEA/LCA

Development Project:

- Maksud Rahman (Rice)
- Nicole Stark (FPL)



RICE



Providing a little extra peel

MOISTURE STAYS INSIDE

AIR STAYS OUTSIDE



- Soy – Protein
- Corn – Protein
- Optimize formulation
- Scale-up dip coating
- TEA/LCA

2024 P³Nano RFP – Timeline

Draft

Anticipated 2024 Funding:	\$1.75 million
<i>Official</i> - RFP:	18-Sept 2023
Submission Deadline:	13-Nov 2023
• US Endowment Proposal Portal	
Notification of funded projects:	18-Dec 2023
<i>Official</i> - Announcement:	Jan 2023
USFS Contracting:	April 2023

Funding Program Objectives

Draft

Advance Commercialization of Wood Nanomaterials:

- Cellulose, lignin
- Increase technology to a higher TRLs

Build/Strengthen Collaborations with FPL:

- FPL scientists have significant role in projects (Co-PI) **
- Link with TEA/LCA team at FPL & UW-Madison
- Access to FPL resources & expertise

Create a Sustainable Future:

- Reduce dependence on greenhouse gas intensive materials
- Circular Economy: recycle, reuse, biodegradability

FPL Emerging Emphasis Areas



Green Building/Construction

- **Cellulose Nanomaterials** in Cement and Asphalt
- Cross Laminated Timber Tall Buildings
- Lignin and Carbon Foam Structural Insulation Panels



Renewable Packaging

- Paper and Board
- Films/Barriers
- Replacement for Single Use Plastic
- Enabled by **Cellulose Nanomaterials**



Wood to Value

- Biochar
- Biorefinery
 - **Cellulose Nanomaterials**, Lignin, Hemicellulose



Sustainability & Forest Carbon

Project Category

Draft

Exploratory Projects:

\$100K (~1-2 year project)

- TRLs 2 to 3
- fundamental science, engineering, validation
- explore new phenomena, mechanisms, solutions
- Should link how this will solve barriers to commercialization

Development Projects:

up to **\$500K** (~2-3 year project)

- TRLs 3 to 7
- Scaling up: Larger volumes, industrial systems
- Focused on addressing technical barriers to commercialization

Submission Requirements

Draft

Project Teams:

- FPL scientist PI or Co-PI
- Lead institution (where most work & spending completed)
- Industrial collaborator is encouraged
- TEA/LCA encouraged

Lead Institution:

- Must provide minimum 20% cost share
- USDA-FS funding rules apply (Land-grant, indirect, tuition, etc.)

Submission:

- Completed by FPL scientist
- Proposal document
- Budget document (Lead institution approved budget)

Proposal Guidelines

Draft

Proposal Document:

- 1-page project snapshot
- 5-page project narrative
- 1-page budget snapshot
- X-pages PI & Co-PI bios
- X-pages for letters of support from collaborators



Proposal Guidelines

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1-Page Project Snapshot:

1. Title
2. Project category: Explore or Develop
3. Funding requested & cost share
4. Principal Investigator(s)
5. Collaborator(s)
6. Role of each Team Member
7. Project Abstract:
 - Highlight the concept or innovation
 - Highlight current TRL
 - Highlight how project will increase TRL

5-Page Project Narrative:

8. General Description of Need
9. Key Questions Being Addressed
10. Relevant Preliminary Work by PIs
11. Research/Development Approach
12. Work Plan & Deliverables (Task list)
13. Timeline & Milestones

1-Page Budget Narrative:

14. Budget summary list each year
 - Itemize: (salary, benefits, tuition, supplies, equipment, subcontracts, travel, cost share, other)
 - Total funds requested & cost share
 - Justification summary for each item.

Travel Guidelines

Draft

P³Nano Grantee Meeting:

- PIs required to attend
- Give update & network
- Budget for $X+1$ times to attend. X = years of project

Conferences:

- Tech Transfer – presenting project results
- 1 conference per year.

Research Lab Visits:

- As needed for project success

Proposal Guidelines

Draft

Proposal Document:

- 1-page project snapshot
- 5-page project narrative
- 1-page budget snapshot
- X-pages PI & Co-PI bios
- X-pages for letters of support from collaborators

Budget Document:

- Must provide minimum 20% cost share
- **USDA-FS funding rules apply** (Land-grant, indirect, tuition, etc.)
- Itemize: (salary, benefits, tuition, supplies, equipment, subcontracts, travel, cost share, other)