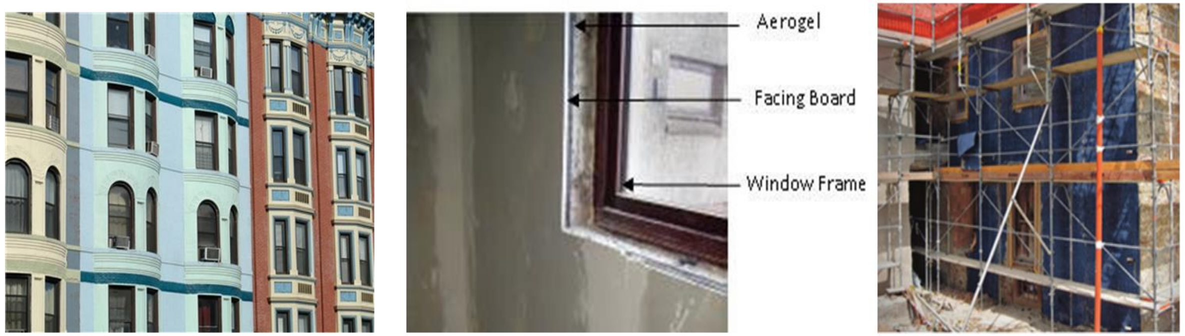
Innovative Aerogel Production for Low-cost and High-R Insulation



Heritage Building and House Structures Thermal Insulation with Aerogels (Retrofit)

Window Frames and Roofs Insulation with Aerogel Strips and Sheets

Space-age Building (Museum)

LNG Liquefaction & Regasification

Subsea (Pipe-in-Pipe)

Petrochemicals Manufacturing Plants

Performing Organization(s): Optowares, Inc.
PI Name and Title: Dr. Je Kyun Lee, Senior Staff Scientist
PI Tel and/or Email: 781) 243-3793, jlee@optowares.com
Presenter: Dr. Jung U. Yoon, Executive Vice President, 781) 427.7109, jyoon@optowares.com
WBS #, FOA Project # and/or any other Project #: Award Number: SBIR, DE-SC0021909 (Topic No: 13b)

Project Summary

Objective and outcome

- Develop ambient pressure (AP)-dried "pDCPD aerogel composites with low-cost (no greater than $1/square foot per inch or 3-5 times lower cost than silica aerogel composites) and high R-value/inch (≥ 8 or TC: ≤ 18 mW/m K),
- Good building insulation form factors with scalable manufacturing process

Team and Partners

Dr. Je Kyun Lee (PI)

Dr. Melissa Chen (Senior Material Scientist)

Mr. Andrew Leung (Principal Mechanical Engineer)

Dr. Jung U. Yoon (VP, Commercialization)

Technician (Supporting Experiment & Characterization)

We are closely working with our Technical and Business Assistance (TABA) partner, the LARTA Institute

Stats

Performance Period: 8 months f Phase II

DOE budget: $1,100,000 for Phase II,   Cost Share: None

Milestone 1: AP-dried pDCPD aerogels with higher R-value of ≥ 8 within the end of 20 months

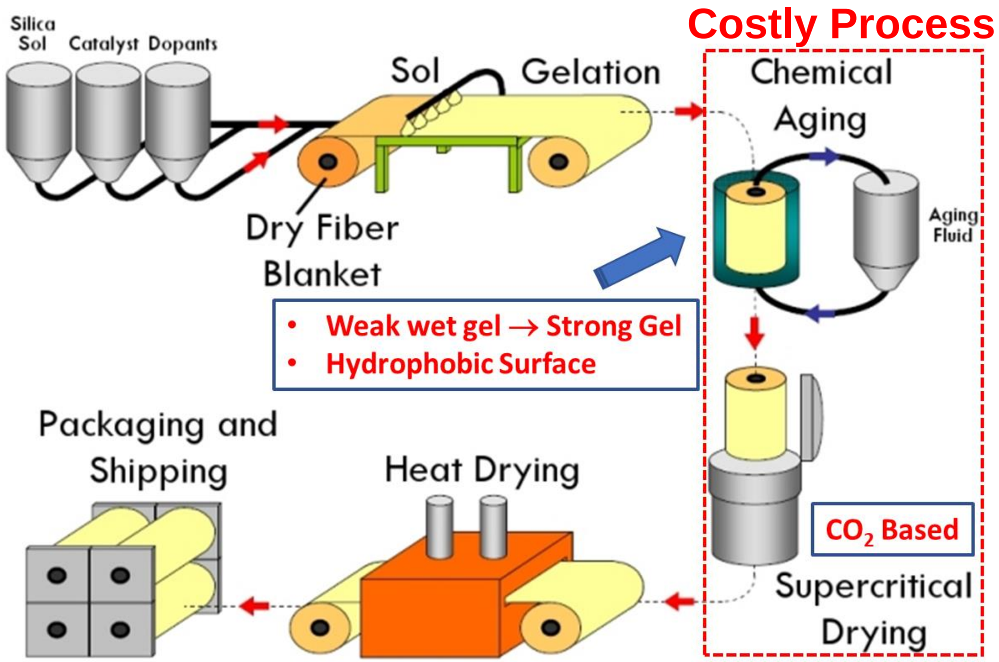
Milestone 2: Cost reduction to ≈ $ 0.6/ft^2 from currently $ 0.75/ft^2 by recycle catalyst within 24 months

Milestone 3: Scaled-up to 24 in x 48 in x 6-10 mm will be produced within the end of 24 months

Problem

Despite the exceptional insulating properties, aerogels are not widely used as demonstrated from small market size of $ 638 million in 2020*[1] compared to $ 23.4 billion in 2021 global insulation market due to its cost being two to five times more than the conventional insulations such as foamed plastics and fiberglass and rockwool.

- An aerogel is a solid with nanometer-size pores typically made from expensive material feedstocks (Silica) and complex and costly supercritical CO_2 drying or freeze-drying method.*[2]
- Costly supercritical-CO_2 or freeze-drying methods.*[3]
- Drying at ambient pressure (AP) can lead to Xerogel with the major mechanical and thermal damages.
- Expensive material feedstock.*[4]
- Silica aerogels are very brittle and dusty → higher cost, labor effort, and installation time*[4].



(Diagrams of silica aerogel composite production)*[5]

Alignment and Impact

Optowares' ambient pressure (AP)-dried pDCPD aerogels will provide effective insulation materials with cost-competitiveness and highly energy-efficient manufacturing process

- Use economical atmospheric pressure drying with cost about five times as low as both freezing and CO_2-supercritical drying methods
- Utilize inexpensive and readily available percussor feedstocks at three times lower costs than silica aerogel percussor feedstocks.
- Use hydrophobic solvent as a reactive solvent and a hydrophilic solvent as a non-reactive solvent to make the process amenable for easy separation and recycling of solvents
- Lower cost will lead to greater adoption of aerogel for building insulation to reduce heating/cooling energy use
- Superior material properties (more toughness, durability, no dustiness, and good form factor) leads to lower labor effort and installation time
- Our new AP-dried pDCPD aerogel composites will support greenhouse gas emission reduction, government carbon targets, and increasing demand for energy efficiency requirement

Approach

Develop ambient pressure (AP) dried pDCPD aerogels composites using

- Polymerizing an inexpensive and readily available DCPD that costs 3-5 times less than the silica feedstocks- material cost of our new AP-dried pDCPD aerogel composite is estimated around \$ 0.75/ft^2 with potential reduction to \$ 0.6/ft^2 after recycling 30% catalyst
- Ambient pressure drying process - much less expensive and less energy-intensive than current aerogel manufacturing methods

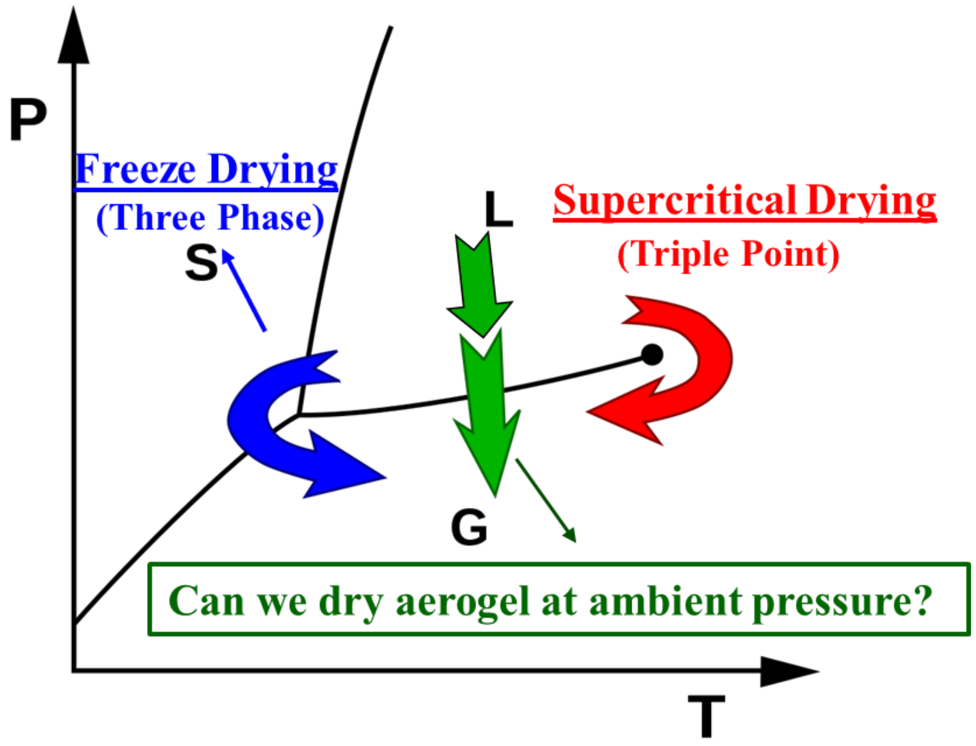
Achieve exceptional physical properties

- Low-cost (no greater than \$1/square foot per inch or 3-5 times lower cost than silica aerogel composites) and high R-value/inch (<=8 or TC: <= 18 mW/m K),
- Stiff, less dust, high durability, and good mechanical properties

No research team has demonstrated AP-dried pDCPD aerogel products yet.

Approach

Develop readily available, scalable, less energy-intensive manufacturing processes



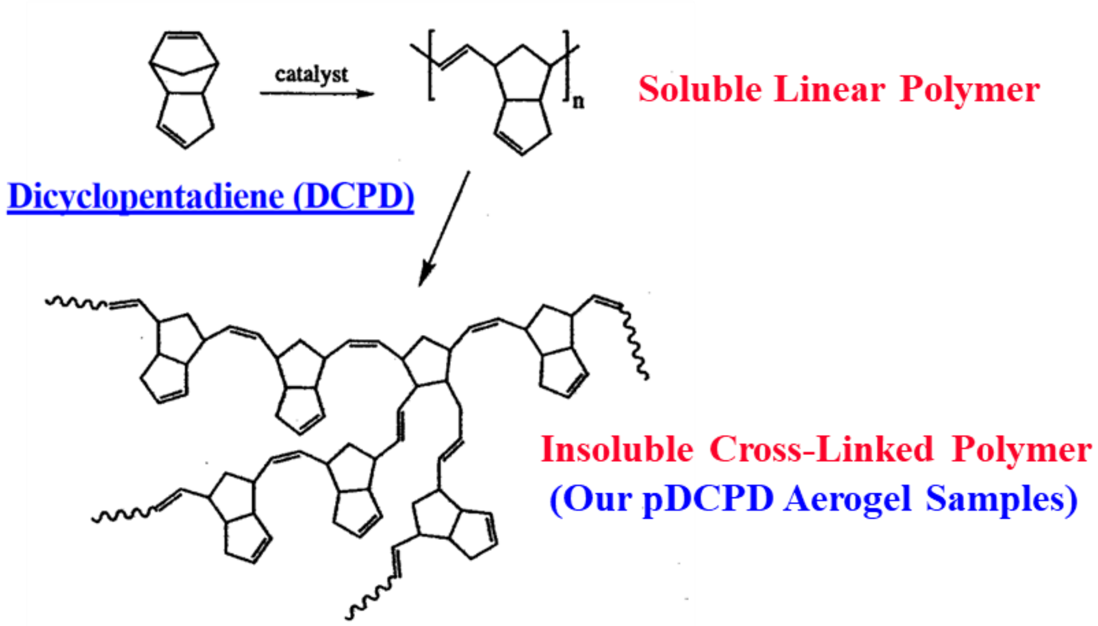
- AP-dried silica aerogel still require very expensive feedstock and are very brittle and dusty
- Develop ambient pressure drying process for aerogel that will preserve mechanical and thermal properties
- Ambient pressure process will be suitable for continuous and high-throughput manufacturing methods
- Process will be less costly without expensive equipment or high energy use required for supercritical drying and freeze-drying methods*3

If pDCPD aerogels can be made at ambient condition, there will be many applications.

Approach

Use low-cost feedstock to fabricate aerogel

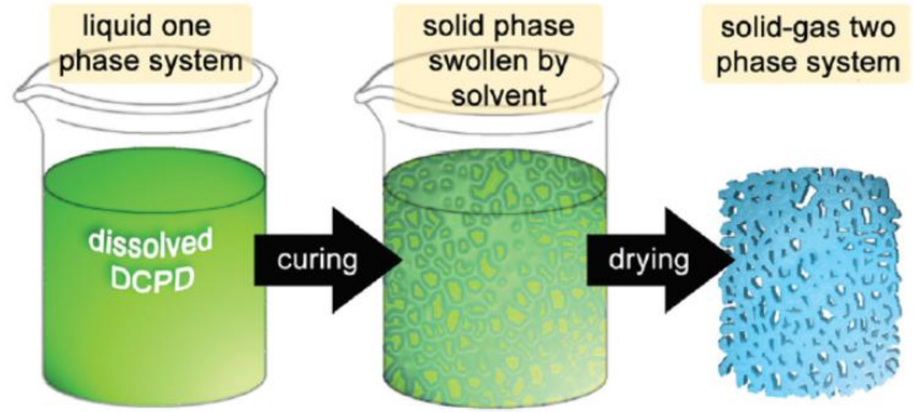
The cross-linked polydicyclopentadiene (pDCPD) can be easily formed by ring-opening metathesis polymerization (ROMP)



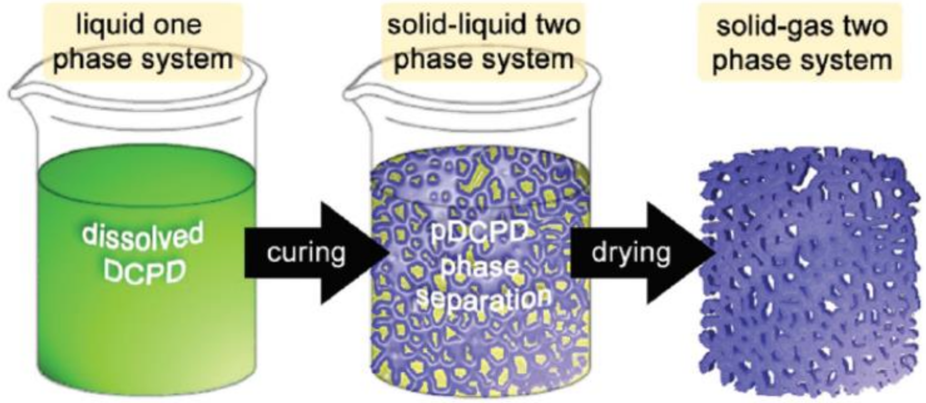
- pDCPD aerogel products via polymerizing an inexpensive and readily available DCPD that costs 3-5 times less than the silica feedstocks,
- Supercritical CO_2-dried pDCPD aerogels with densities of 0.112 - 0.138 g/cc previously showed high R-value of 6.9 - 9.4 (TC = 20.9 ~ 15.4 mW/m K)*[6] ,
- Very stiff, less dust, and good durability with extraordinary mechanical properties
- pDCPD aerogel can show the inherent hydrophobicity with great benefit over inorganic based aerogels that are needed for hydrophobic chemical treatment

Approach

Optowares developed the AP-Dried pDCPD Aerogels for The First Time by Using the Combined Principle of Spring-Back and CIPS during this DOE SBIR Phase I Program



"Spring-Back" (with Intrinsic Hydrophobicity)*7



"Chemically Induced Phase Separation (CIPS)*7

Modified Mechanisms

- ✓ No Silylation Reaction
  → Used Intrinsic Hydrophobicity of pDCPD
- ✓ Used Gelation at near RT
- ✓ Conducted Solvent Exchange at near RT
- ✓ Similar Solvent System As Used in CIPS Method
- ✓ Conducted Drying at Atmospheric Condition from Refrigerator T. (Not Freezing Temp.) to RT

Approach

Commercialization Plan

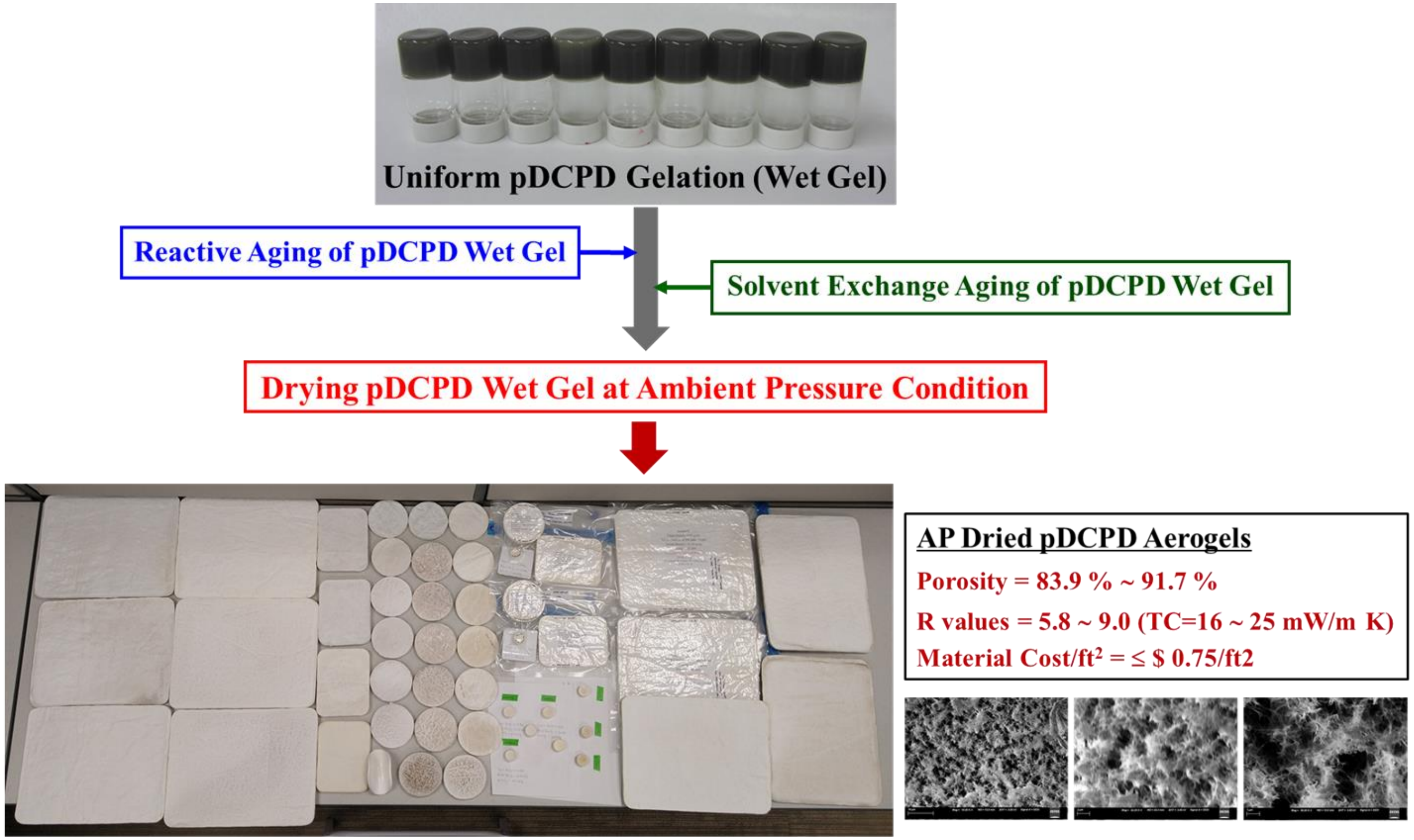
- **Potential applications (Global aerogel market - $1,045 million by 2025)*[1]**
  - Replace the current 1) Silica Aerogel and 2) Conventional Inorganic and Organic Thermal Insulation Materials such as Rockwool, Glass Fiber, spray PU and Expanded PS Foams, and to develop 3) New Applications (Window, Barriers for Lithium-ion Batteries…)

- **Explore licensing or manufacturing options to bring pDCPD aerogel to market**

Pros of Licensing	Cons (Licensing)
Lower risk – licensee will know the market/industry Income from royalties (typically 2-10%?) Cost effective route to get product to market – simpler and less expensive Optowares can concentrate on innovating	Lower profit Loss of control over manufacturing, distribution, sales, branding, marketing, and payout A company may license the product but never put it into production
Pros (Manufacturing)	**Cons (Manufacturing)**
Higher potential profits than licensing May make it to market faster than licensing Maintain more control over product and marketing	More risk Costly to set up a manufacturing line – may need to find investor(s) Requires business expertise

Progress and Future Work

Preparation Process, Samples, and Important Properties of The First AP-Dried pDCPD Aerogel Composites Developed during This DOE SBIR Program[*8,*9]



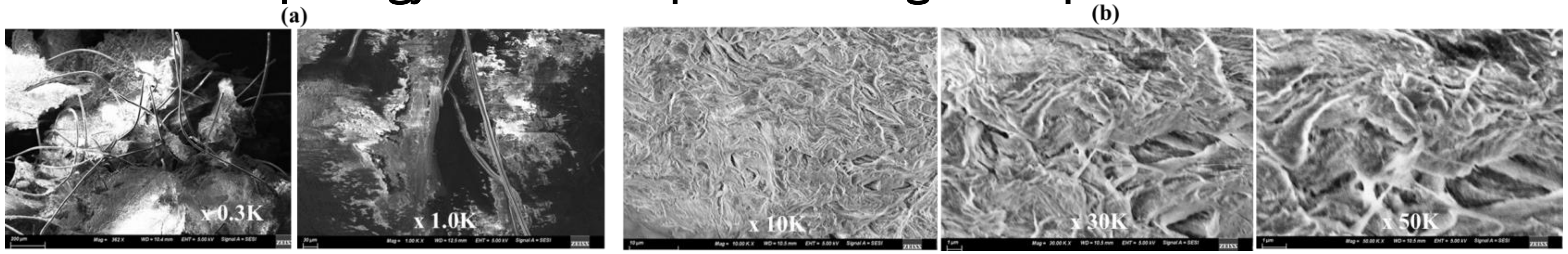
Ambient Pressure Dried pDCPD Aerogel Monoliths and Composites

- <u>First-ever demonstration</u> of ambient pressure dried aerogel fabrication using pDCPD wet gel
- Good intrinsic hydrophobicity, stiffness, and low dustiness

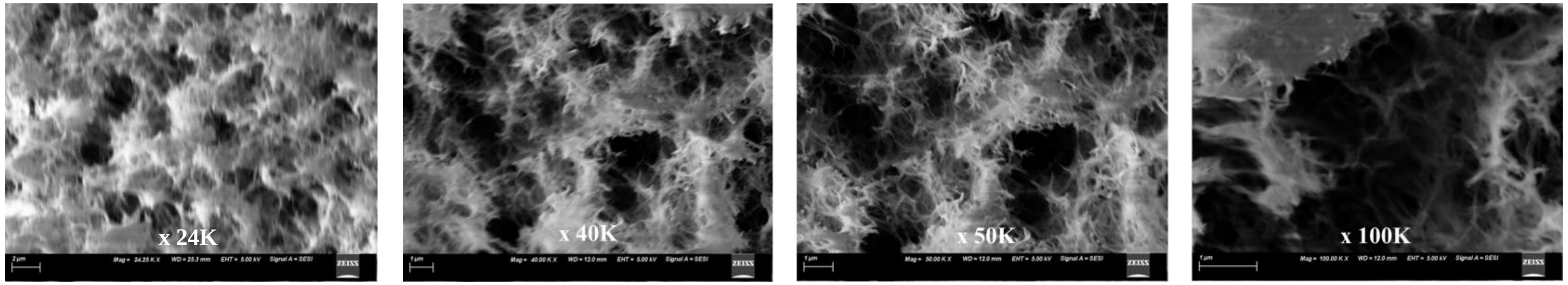
Progress and Future Work

Pore Morphologies, Sizes, and Volumes of AP-Dried pDCPD Aerogel Products

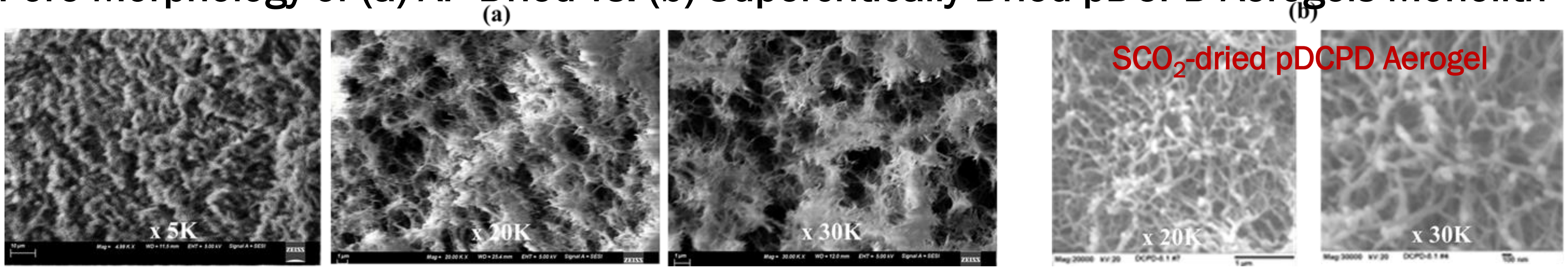
Morphology of AP-Dried pDCPD Aerogel Composites

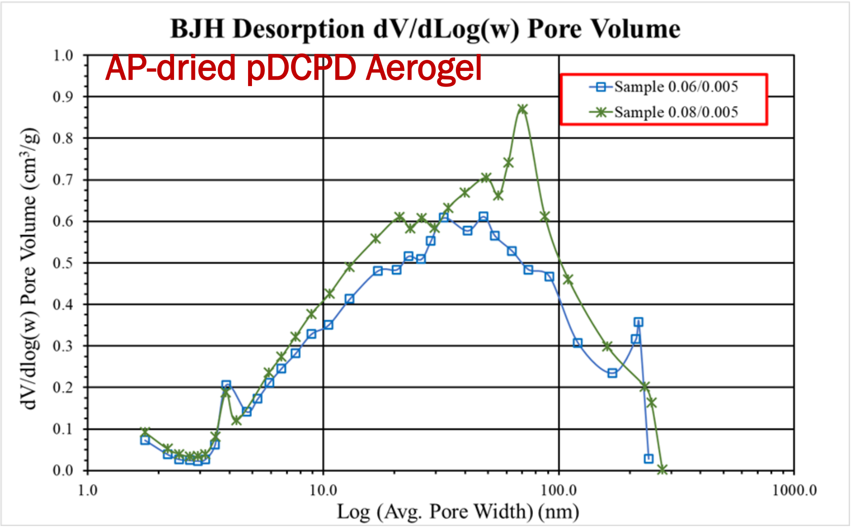


Pore morphology of fracture surface of AP-dried pDCPD aerogel monolith

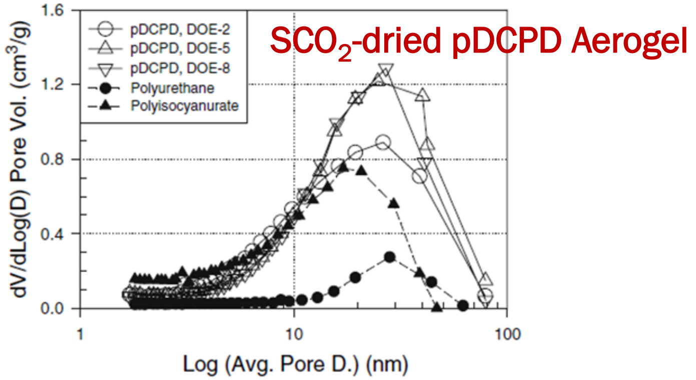


Pore Morphology of (a) AP-Dried vs. (b) Supercritically-Dried pDCPD Aerogels Monolith



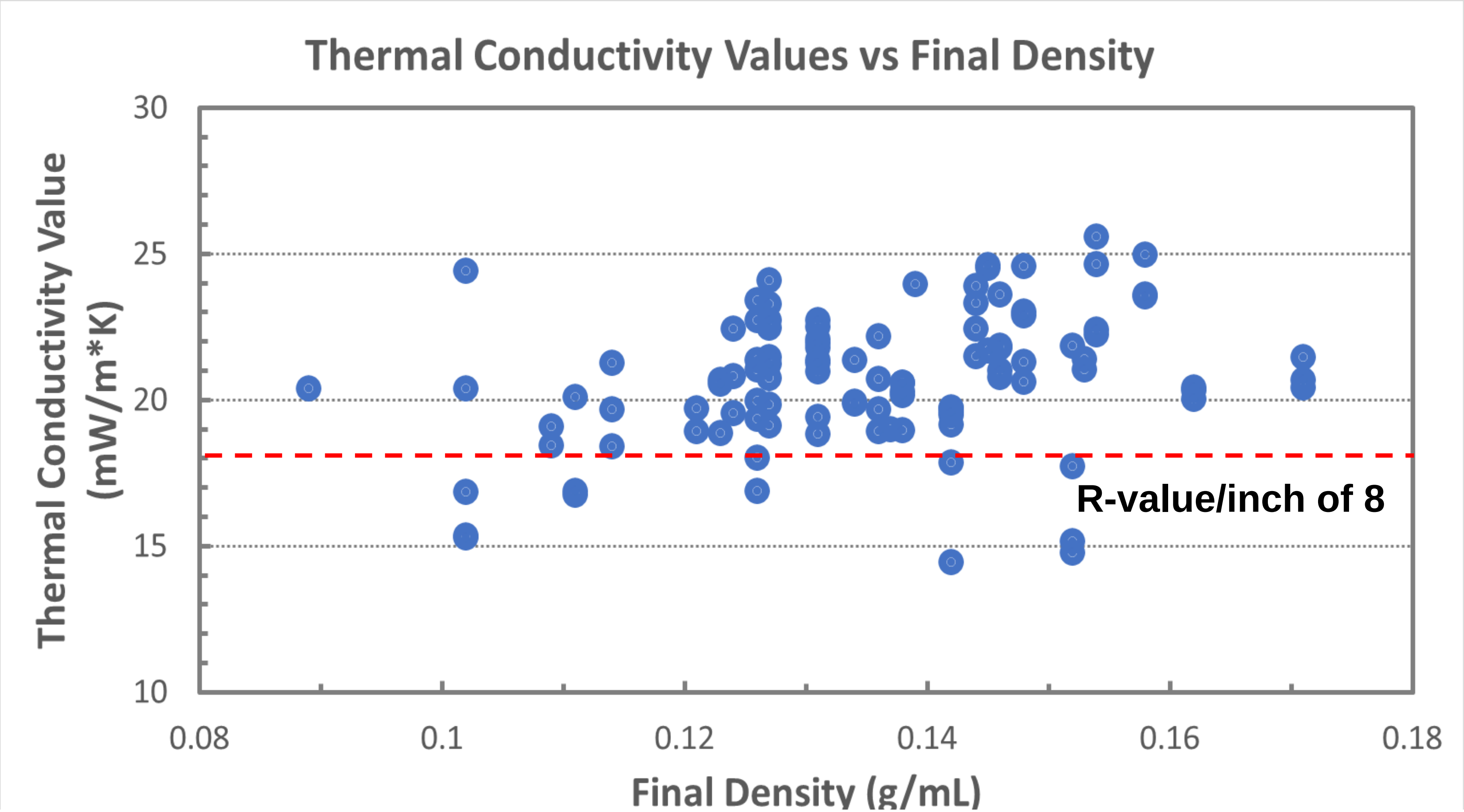


VS.



Progress and Future Work

Improvement of R-value (TC-value)

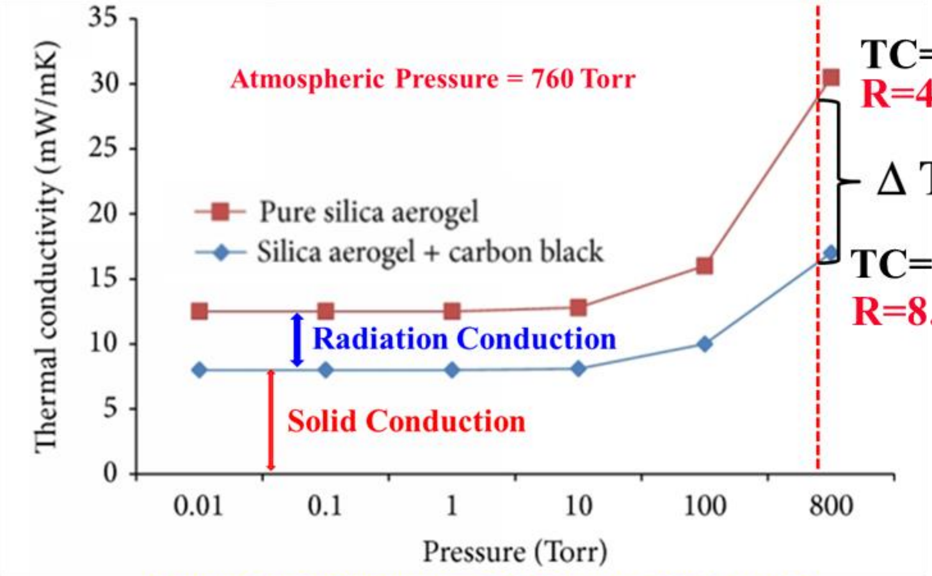


Average TC value = 21-24 mW/ m K
Or
R-value/inch = 6.0-6.9

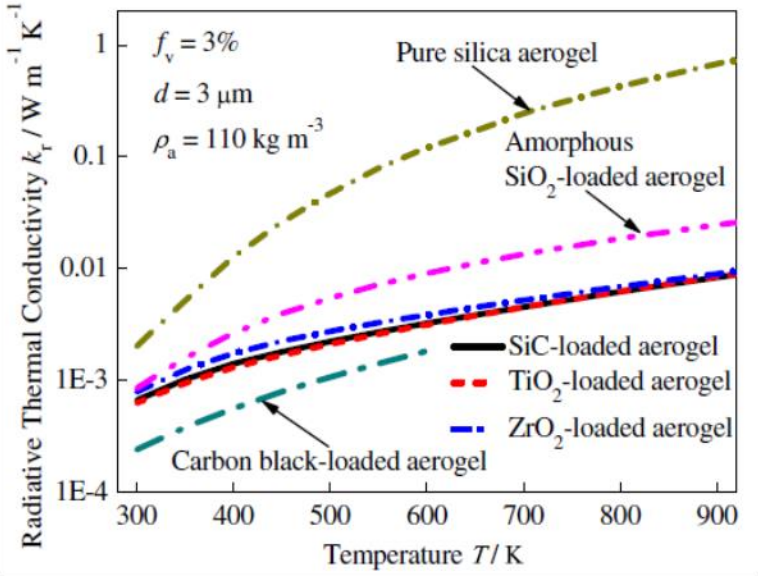
These are higher TC and lower R-values than our target values (R-value ≥ 8 or TC: ≤ 18 mW/m K)

Progress and Future Work

Improvement of R-Value (TC value) to meet target R-value/inch of 8 or TC value of 18 mW/m K) by Incorporating IR Opacifiers*[10],*[11]



Atmospheric Pressure = 760 Torr or &760 mmHg

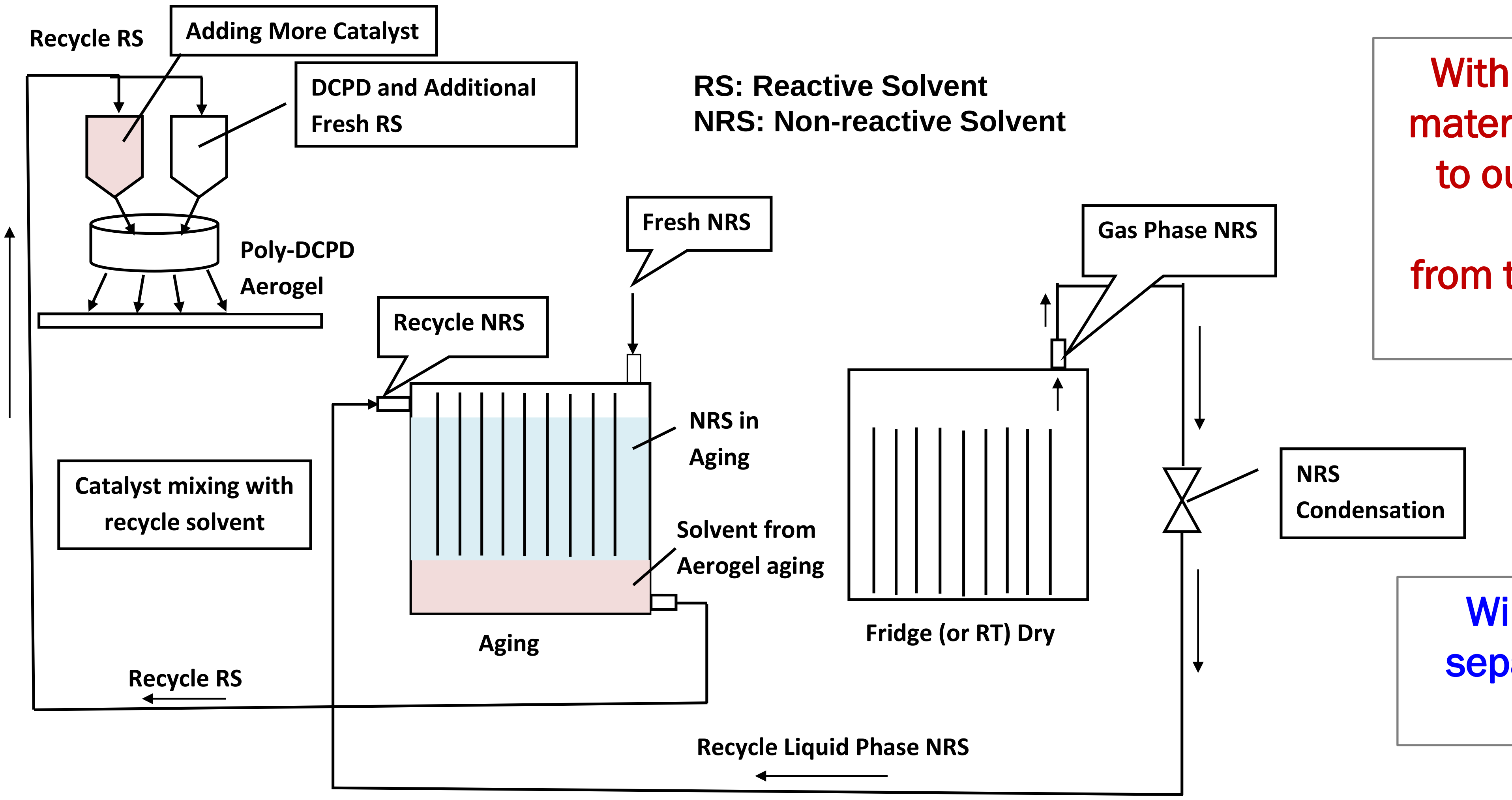


- ✓ TC values of carbon-black opacifier-added silica aerogel are lower (higher R-value) than those of pure silica aerogel.

Possible choice of IR Opacifiers: Carbon black, carbon fiber, single wall and multiwall carbon nanotubes, alumina fiber, asbestos fiber, zirconia fiber, alumina, clay, mica, silicas, calcium carbonate, titanium dioxide, talc, zinc oxide, barium sulfates, and iron oxide, …etc.

Progress and Future Work

Develop Solvent/Catalyst Recycling Process for pDCPD Aerogel



RS: Reactive Solvent
NRS: Non-reactive Solvent

With 30% recycling, the material cost will be down to our Target Cost: ≈ $ 0.6/ft2 from the current cost of $ 0.75/ft2.

Will be compared with separation method after Distillation!

Progress and Future Work

- **Scale up the manufacturing process**
  - Fabricate AP-Dried pDCPD Aerogel Composites with 24 in x 48 in with 6 mm or 10 mm thickness
- **Engage potential customers**
  - Test out potential customer applications

Letter of Collaboration **March 28th, 2022**

SBIR Program Manager
Department of Energy

Dear DOE SBIR Program Manager,

The purpose of this letter is to provide Ford Motor Company's support of the proposal in response to DOE's SBIR Funding Opportunity Topic # DOE 13b. Optowares' proposal is entitled "Innovative Aerogel Production for Low-cost and High-R Insulation.

The Ford Motor Company (www.ford.com) is an American multinational automaker and the most trusted mobility company, headquartered in Dearborn, Michigan, a suburb of Detroit. Founded by Henry Ford and incorporated on June 16, 1903. Ford is interested in Optowares' DOE SBIR Phase I research to develop innovative, polymer-based aerogel material with high-R values and at 3-5 times cost reduction compared to current silica-based aerogel materials as thermal barrier for battery in electric vehicles.

If your proposal is selected for award, it is Ford's intent to support Optowares' Phase II effort. Ford proposes to provide resources to support the overall goals and objectives identified in the Project Description. Ford will provide guidance and support through technical discussions with Optowares' team regarding properties of aerogel materials for electric vehicle applications, so that Optowares' team use our guidance when fine-tuning their aerogel material recipe.

We look forward to working with you on this interesting and exciting project. Any rights or obligations of Ford, if any, regarding this potential project is subject to the successful negotiation and execution of definitive agreements.

Mr. Lee,

My name is . I am in San Antonio, Texas. I would like to be an OEM maker of ice/drink coolers utilizing aerogel as the primary insulating component. I'm finding the costs associated with acquiring traditionally manufactured aerogel to be prohibitive for my project. In looking at aerogels and their future, I came across your name and article on the US department of Energy website. I am very interested to hear how your project is going or how it went and whether there is a company commercially producing aerogels utilizing your ambient pressure (less expensive) technology.

Thanks for any information you can allow.

Boston Architects & Builders,

Building Shelter

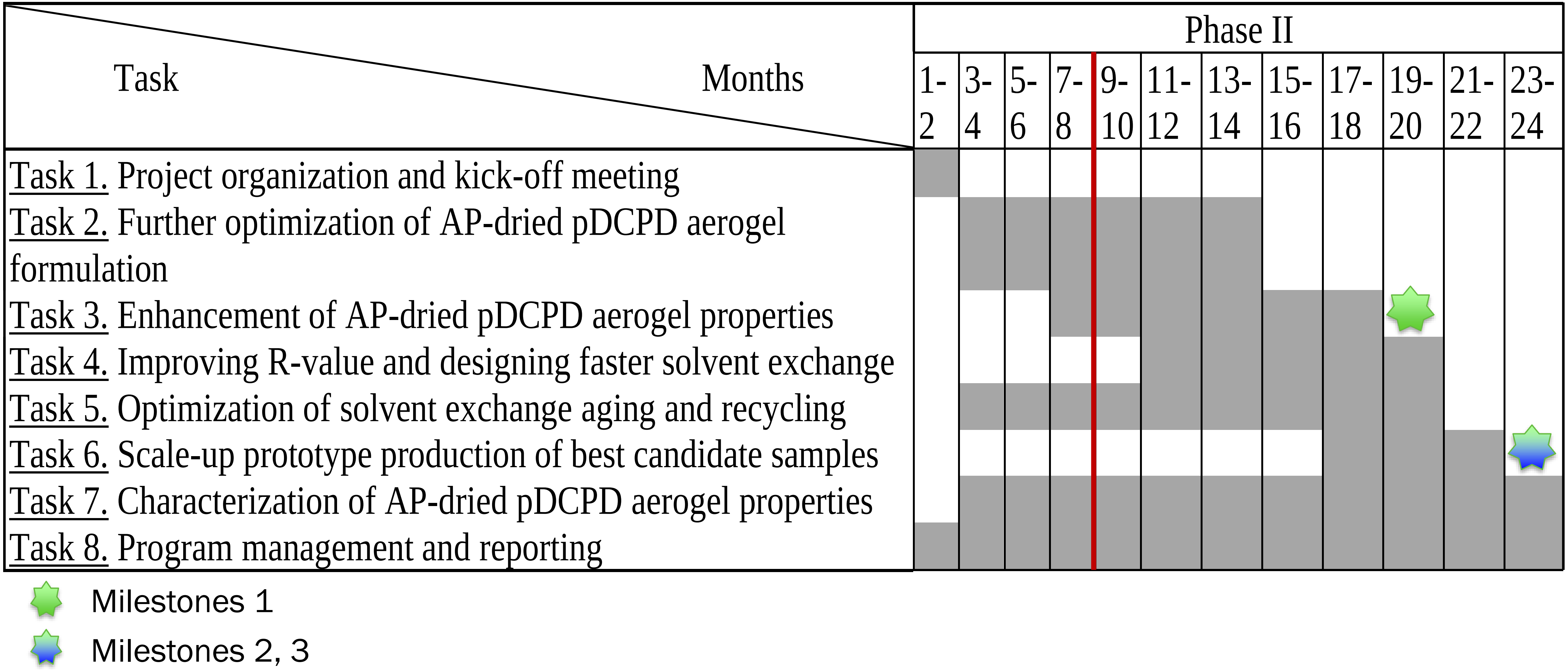
Thank You

Performing Organization(s): Optowares, Inc.
PI Name and Title: Dr. Je Kyun Lee, Senior Staff Scientist
PI Tel and/or Email: 781) 243-3793, jlee@optowares.com
Presenter: Dr. Jung U. Yoon, Executive Vice President, 781) 427.7109, jyoon@optowares.com
WBS #, FOA Project # and/or any other Project #: Award Number: SBIR, DE-SC0021909 (Topic No: 13b)

REFERENCE SLIDES

1. https://www.psmarketresearch.com/market-analysis/silica-aerogel-market
2. S.S. Kistler, “Coherent Expanded Aerogels and Jellies”, Nature, 127, 741 (1931).
3. S. Czionka et al., “Freeze-drying method as a new approach to the synthesis of polyurea aerogels from isocyanate and water”, J. Sol-Gel Sci. Tech., 87, 685 (2018). and https://en.wikipedia.org/wiki/Supercritical_drying
4. S Mumme, DOE 21-Solicitation, 13b, “Advanced Building Construction Technologies; Low-cost, High-R Insulation” (2020).
5. http://www.aerogel.org/?p=1058
6. J.K. Lee & G. Gould, “Polydicyclopentadiene Based Aerogel: A New Insulation Material”, J. Sol-Gel Sci. Tech., 44, 29 (2007).
7. S. Kovacis & C. Slugovc, “Ring-opening Metathesis Polymerisation derived pDCPD based materials”, Mater. Chem. Front., 4, 2235 (2020).
8. JK Lee, Y. Tang, A Leung “Ambient Pressure Dried Microporous Polydicyclopentadiene Based Aerogel Monoliths and Composites” Provisional Patent Application No.: 63454983 (2023).
9. JK Lee, Y. Tang, A Leung, “Method And Apparatus For Manufacturing Ambient Pressure Dried Microporous Polydicyclopentadiene Aerogel Sheets” Provisional Patent Application No.: 63454988 (2023).
10. T. Rettlebach, et al., “Thermal conductivity of silica aerogel powders at temperatures from 10 to 275K,” J Non Cryst Solids, 186, 278 (1995).
11. JJ Zhao et al., “Optical and radiative properties of infrared opacifier particles loaded in silica aerogels for high temperature thermal insulation”, Int. J. Therm. Sci., 70, 54 (2013).

Project Execution



Team

Dr. Je Kyun Lee, who received his Ph.D. in Polymer Engineering from the University of Akron and next, conducted his postdoctoral at MIT, is serve as a PI with over 30 years' experience knowledgeable in all phases of R&D including aerogel related experience of over 14 years. He has 24 publications including aerogel-relevant 8 papers and 177 patents (36 US, 47 PCT, and 94 Korea) including aerogel-relevant 165 patents granted and pending. He was the first inventor & reporter for pDCPD aerogels and polyurea aerogels which were filed and published, He also filed provisional patent for AP-dried pDCPD aerogel funded by DOE SBIR Phase I program. Also, he successfully commercialize aerogel business in two companies: Aspen Aerogel as a team member and LG Chem as a project leader for aerogel R&D and construction of pilot plant.

Dr. Melissa Chen, who received her Ph.D. in Physical Chemistry at Boston University and is working as a senior scientist, worked as a key staff for developing formulation and process during this DOE Phase I program. She was one of key inventors for our new AP-dried pDCPD aerogel products. She will continue to work as a leading scientist to optimize formulation and design methods and apparatus for reactive aging, solvent exchange, and drying at ambient pressure.

Mr. Andrew Leung, who received his B.S. in Mechanical Engineering from the University of Massachusetts at Amherst and is working as a principal production engineer, worked as a key staff for developing formulation and process during Phase I program. He will work to design apparatus for measuring R-value, solvent recovery, catalyst reuse, and drying.

Dr. Jung U. Yoon, who received his Ph.D. in Materials Science and Engineering at MIT and was a technical staff at MIT Lincoln Laboratory for over ten years, is an executive VP and a visiting scholar at UMass at Amherst, has 20 years of engineering experience in product development including 10 years managing an engineering team. He will characterize our AP-dried pDCPD aerogels and lead the commercialization efforts closely working with our Technical and Business Assistance (TABA) partner, the LARTA Institute.