NEW GENERATION THERMAL INSULATING COMPOSITE THIN FILM DEVELOPMENT

ATL Ltd.

Presenter:

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The Architecture of Heat

ATL

The Limits of Conventional Insulation

Geometry dependence restricts complex surface application.

High-density structures fail weight constraints in aerospace/industrial scaling.

Structural degradation and pore collapse occur at >1000°C extreme temperatures.

The Composite Thin-Film Paradigm

Ultra-Lightweight Porous Secondary Phase (Cryogel) + **Fibrous Matrix** (Cellulose/Viscose) = **Geometry-Independent Thermal Barrier**

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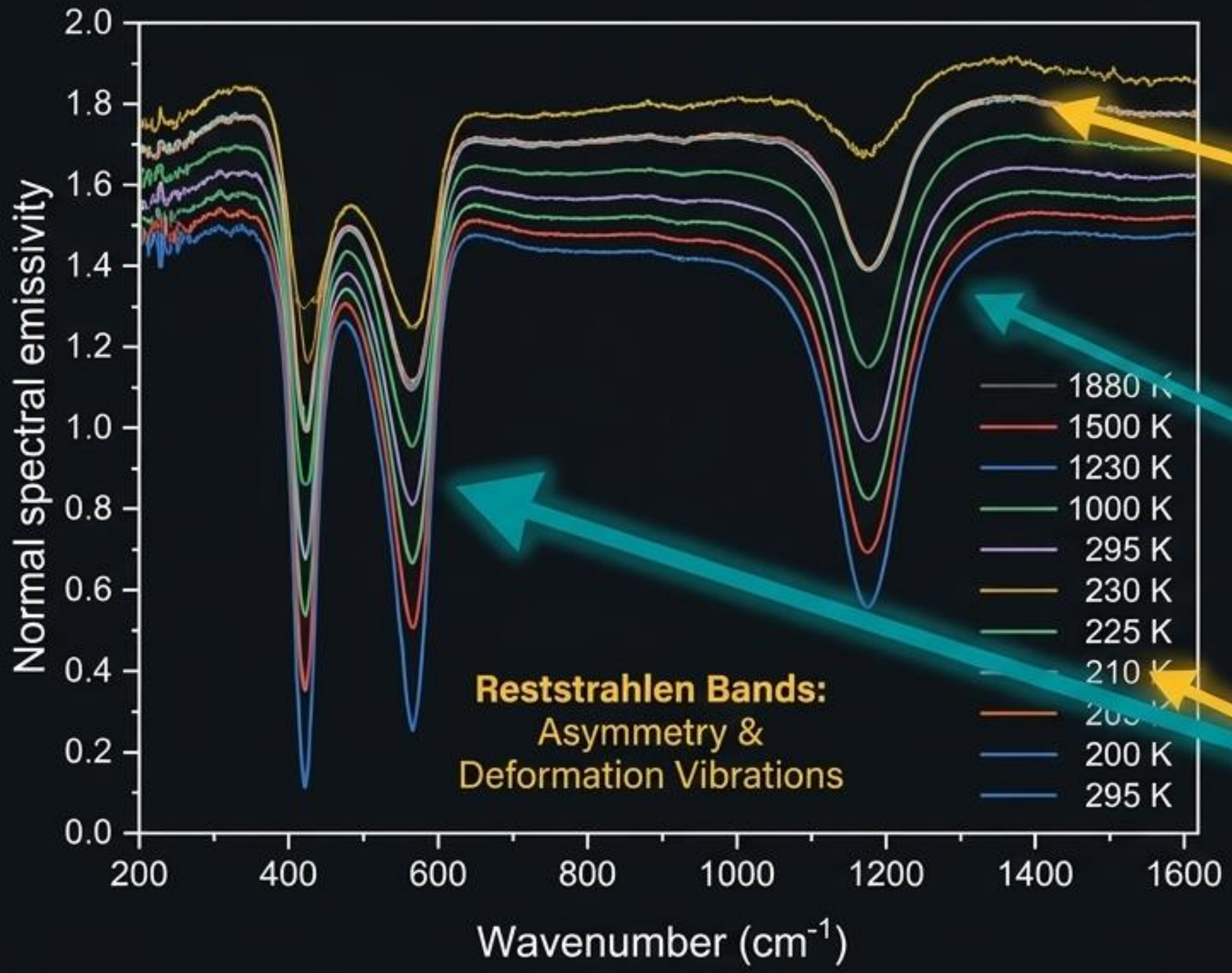

4. ábra. A SiO_2 minta spektrális emisszivitásának hőmérsékletfüggése

Key Physical Principles

Surface Roughness: Multiple scattering processes on complex granular geometries artificially increase emissivity.

The Porosity Shift: Porous materials shift the transmission edge compared to single crystals, disrupting radiative thermal transfer.

The Objective: Engineer a material with optimized nanoporosity to aggressively scatter near-infrared radiation.

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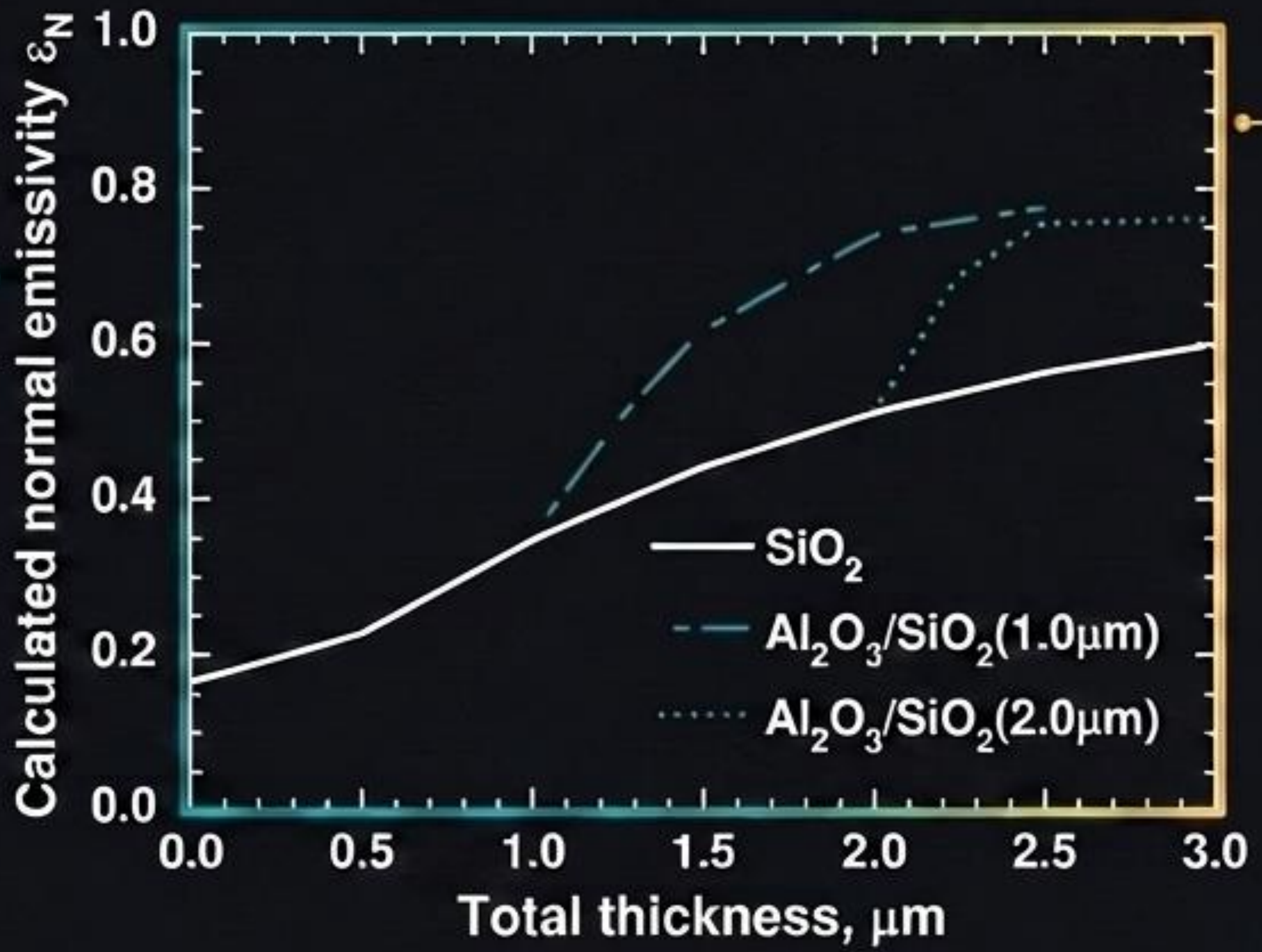
The Thickness Dependency

Emissivity directly scales with layer thickness when the film thickness is comparable to the wavelength of thermal radiation.



Design Implication

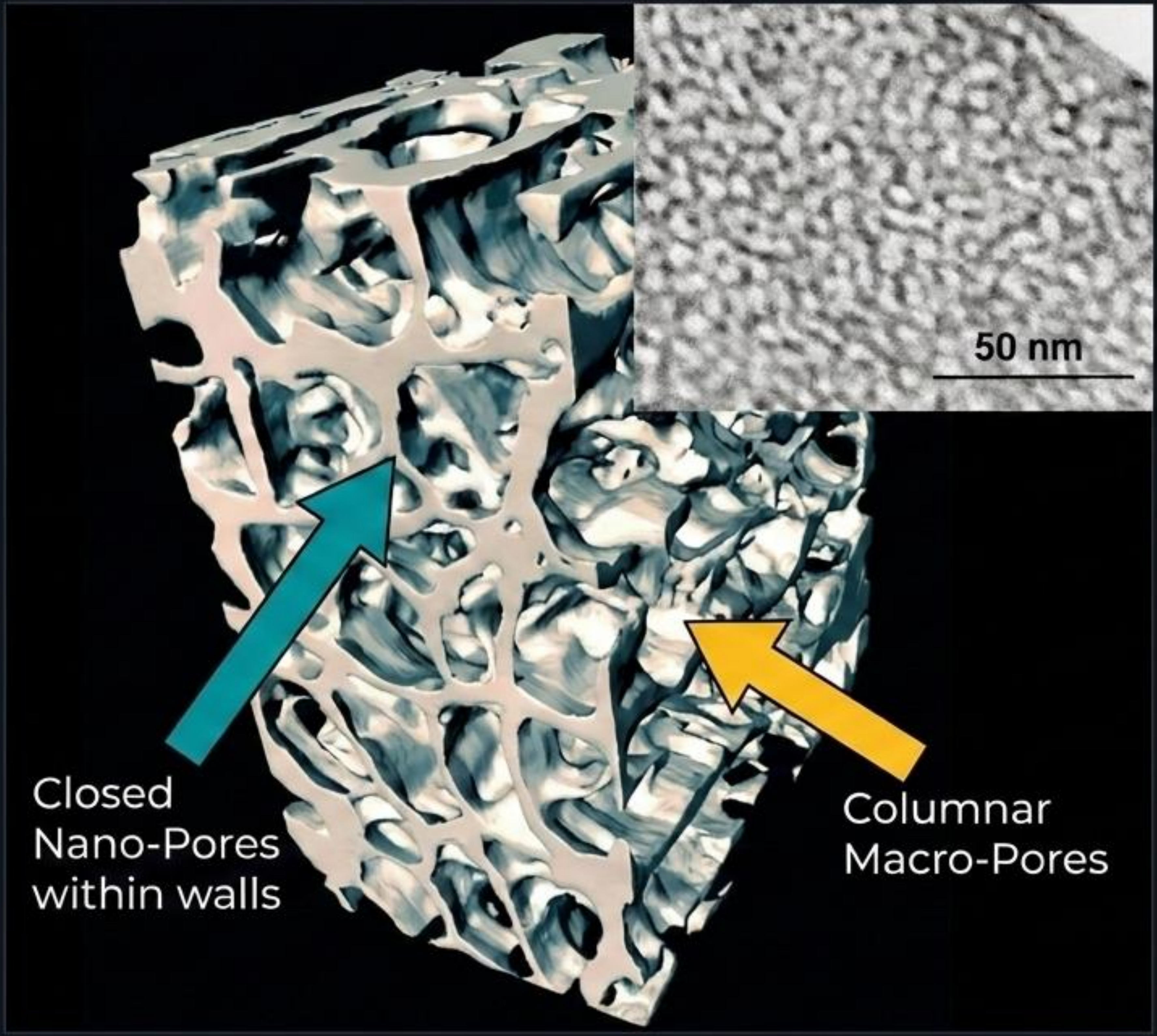
Validates that a geometry-independent thin-film coating can effectively manipulate high-temperature spectral radiant emittance if the internal micro-architecture is properly synthesized.

The Architecture of Heat

1. **Precursor Solution** — Low-energy, cost-effective inorganic Al-salts. No complexing agents required.
2. **Gelation** — Formation of the 3D continuous Hydrogel network.
3. **Rapid Freezing** — Liquid Nitrogen application (~100 °C sec-1). Creates lamellar architecture.
4. **Vacuum Sublimation** — Extraction of solvent crystals.
5. **Final Synthesis** — Stable Al2O3 Cryogel.

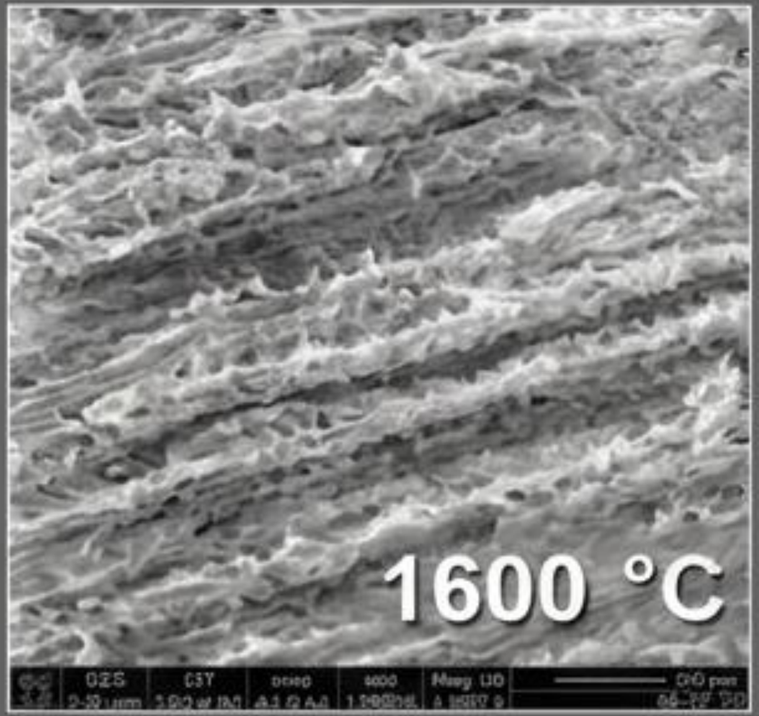
Note: ATL's method uses continuous 3D gels rather than dispersed particle systems, fundamentally altering extreme high-temperature thermal stability.

The Architecture of Heat



Cryogel Architecture

- Total Porosity: 56% - 73%
- Total Pore Volume: 3 - 4 cm3/g
- Formation Driver: Ultra-fast freezing kinetics dictate lamellar architecture.



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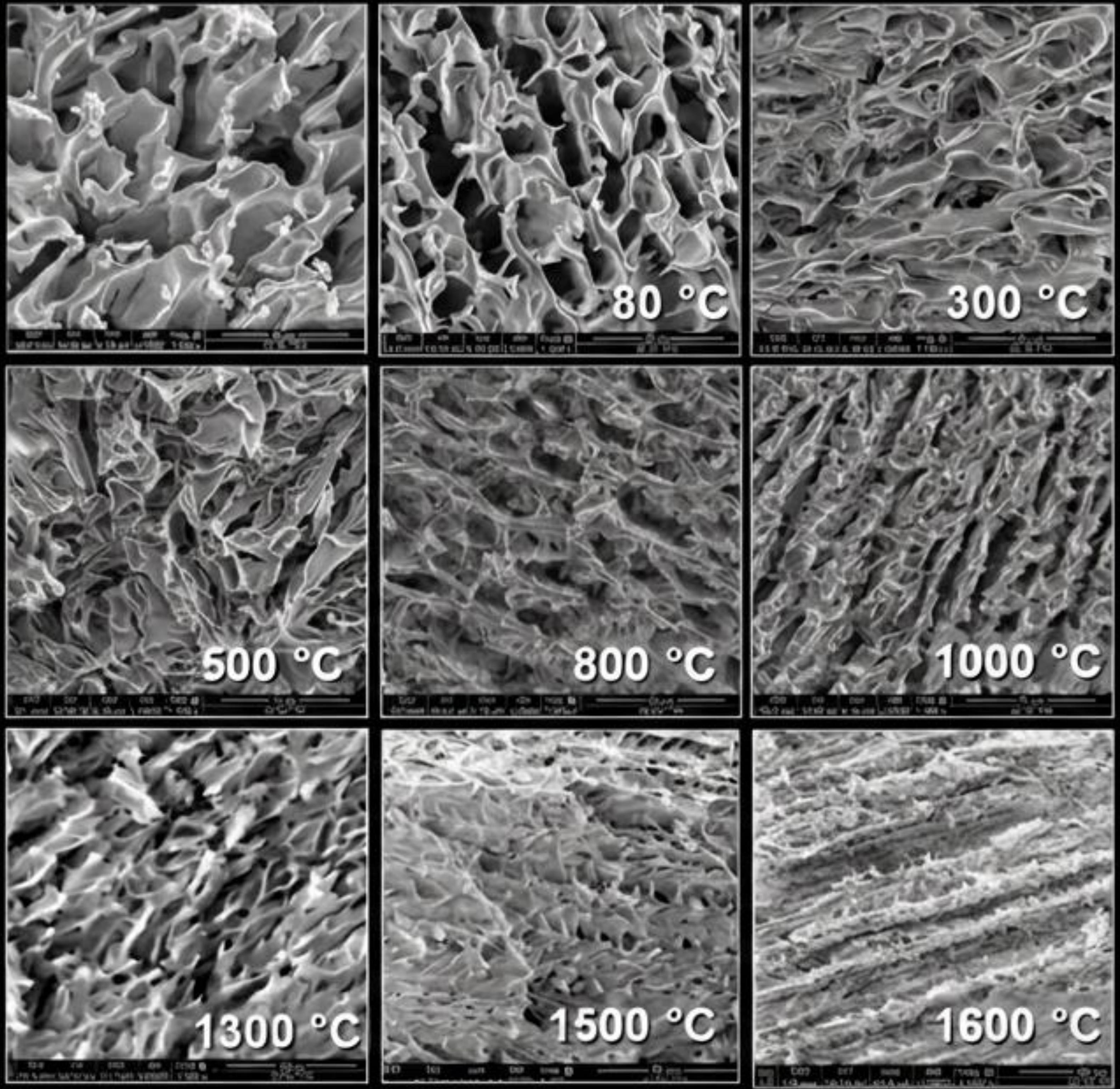
The Breakthrough in Energetic Gold

The Standard Limitation:
Conventional Al_2O_3 cryogels collapse completely at 1000°C due to the α-Al_2O_3 crystalline phase transition.

The ATL Innovation:
The ATL Innovation:
The proprietary 3D continuous gel network successfully preserves the hierarchical pore architecture past 1500°C.



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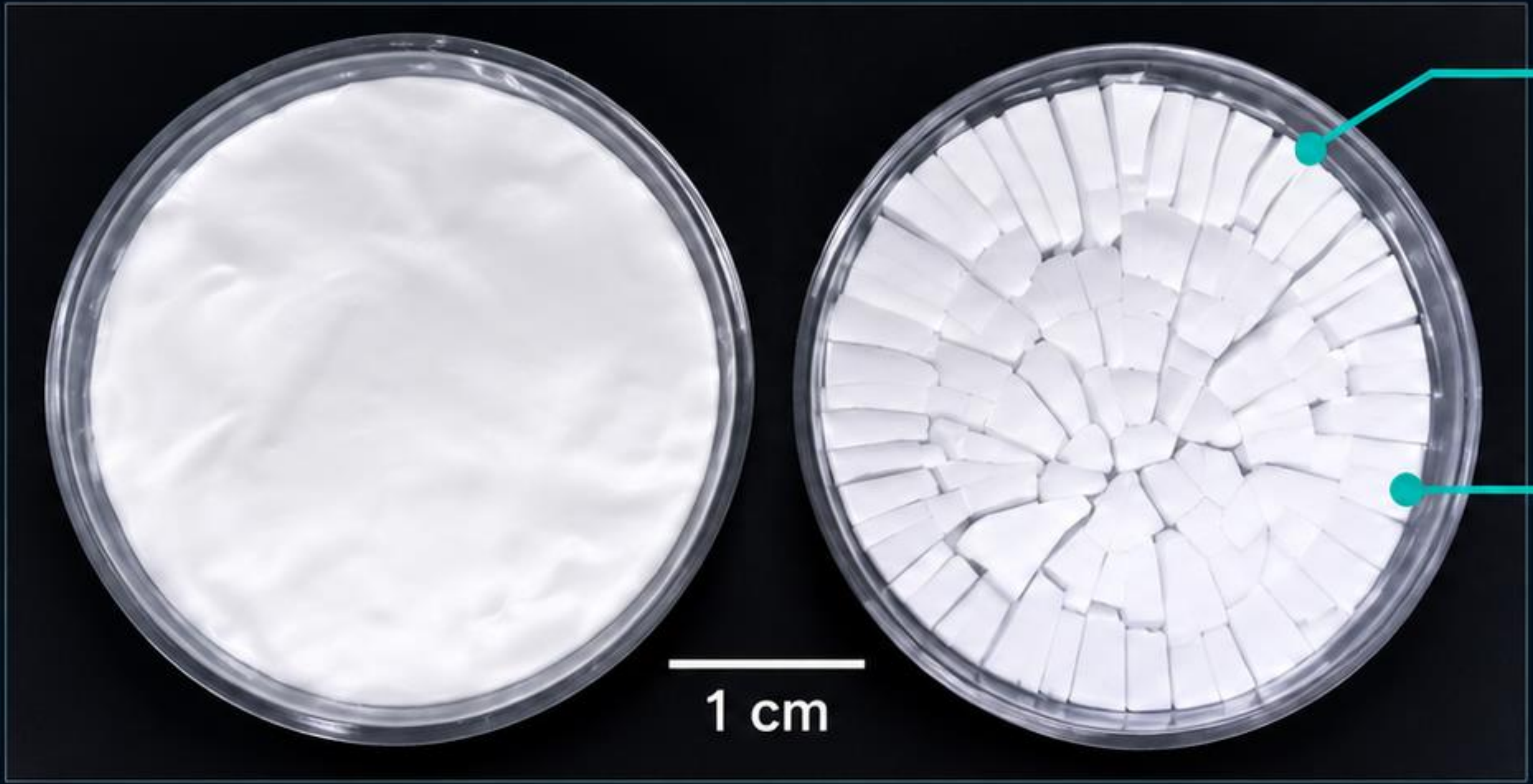

The Freeze-Drying Penalty

Ice crystal sublimation shatters the rigid 3D gel into <250 μm flakes.

The Matrix Solution

Integrating a continuous fibrous cellulose matrix prevents fragmentation.

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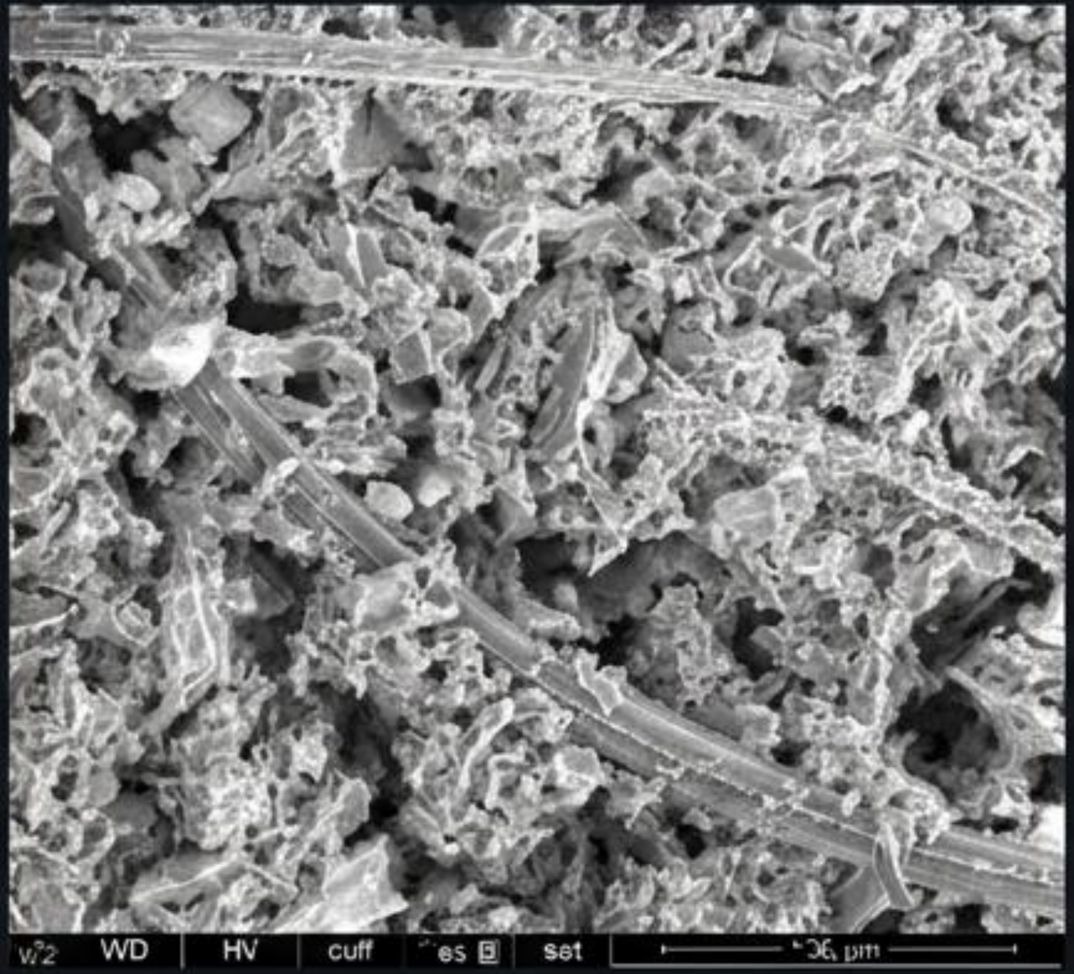


500x

5000x

Microstructural Integrity

- Separately lyophilized Al2O3 cryogel particles homogeneously distributed.
- Viscose/Cellulose fiber network provides continuous mechanical support prior to high-temperature calcination.
- Particle constraint optimized at < 250 μm for maximum integration.

Interface Mechanics: Fiber-Matrix Bonding

Active Surface Hydroxyls

Naturally occurring groups on the Al-oxide-hydroxide hydrogel enable robust hydrogen bonding between particles and fibers.

Binder Optimization

16 m/m% Hydroxyethyl cellulose (HEC) proved superior to Aluminum acetate, eliminating poor adhesion and minimizing dust loss.

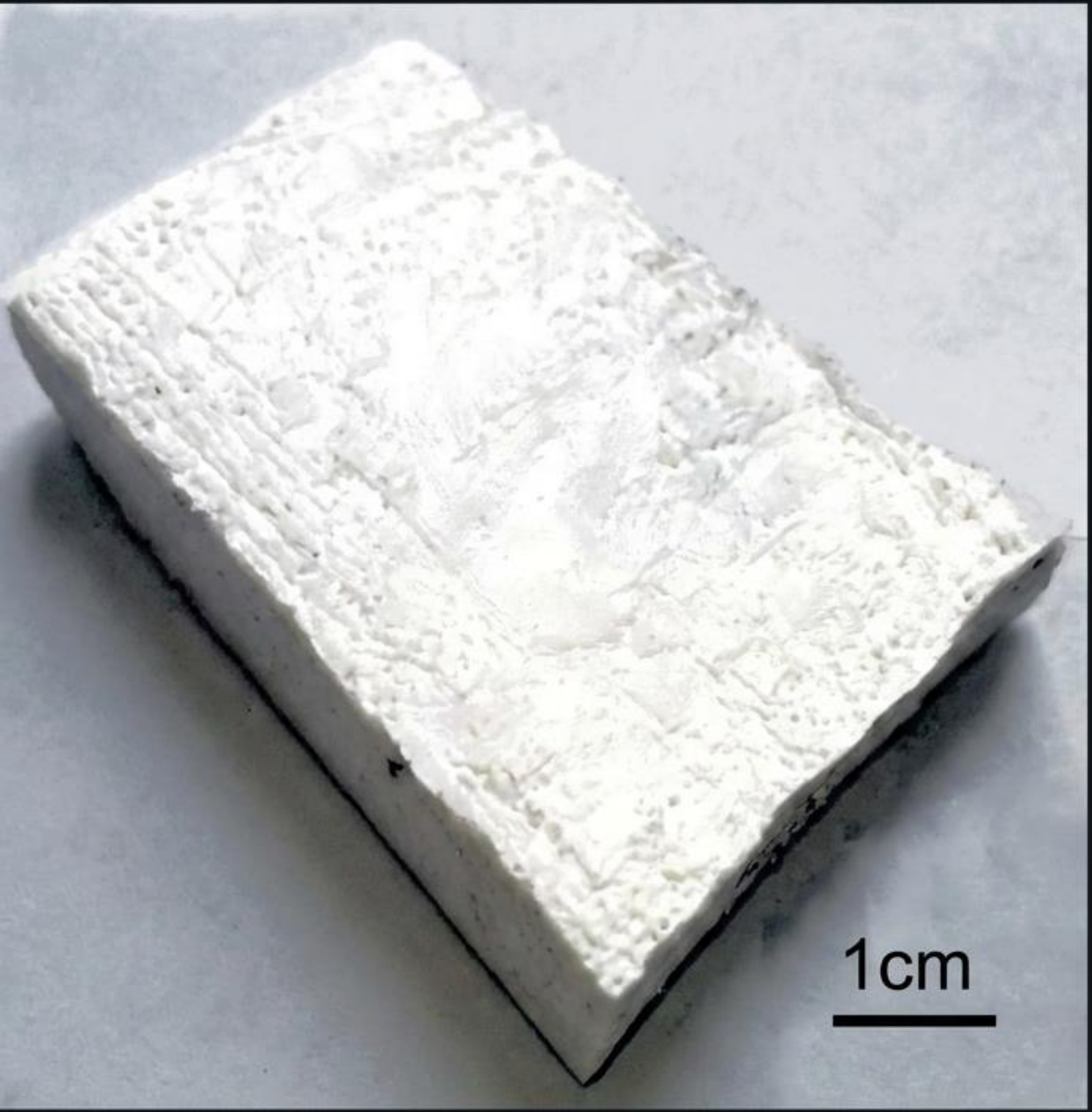
Mechanical Elasticity

The cellulose matrix reduces compression resistance while drastically increasing necessary tensile elasticity for flexible thin-film applications.

Macro-Scale Performance Analysis

Material System	Thermal Conductivity (W/mK)	Macro-Structure
ATL Al2O3-Cellulose Composite	0.040 – 0.045	Flexible Thin Film
Glass-Fiber Epoxy (Standard)	~0.10 – 0.50	Rigid
Commercial Rockwool (Benchmark)	~0.035 – 0.040	Bulky / Geometry-Dependent

ATL's composite achieves 60-80% better insulation than standard epoxy composites while enabling geometry-independent thin-film coating.



Prototype Realization

- ✓ Demonstrated layer homogeneity.
- ✓ Excellent substrate adhesion.
- ✓ High fracture-toughness and minimal dust loss.
- ✓ Operational limit with cellulose matrix intact: 250-350°C (with the alumina matrix surviving past 1500°C post-calcination).

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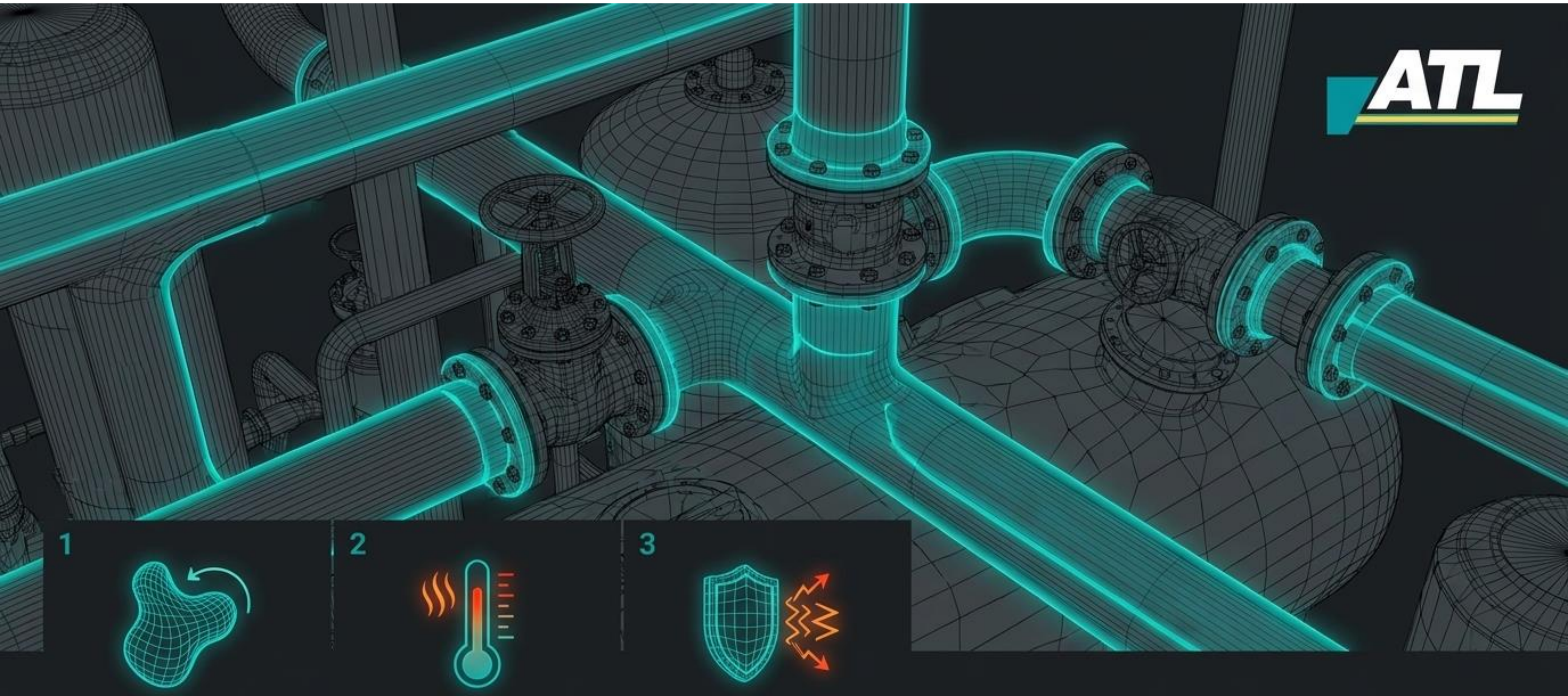
Synthesis Kinetics:
~100°C/s freezing dictates lamellar architecture.

Nano-Architecture:
56-73% hierarchical porosity (closed nano / columnar macro).

The Architecture of Heat Transfer Re-Engineered

Thermal Physics:
Manipulated transmission edges & extreme scattering yield 0.04 W/mK conductivity.

Macro-Engineering:
3D Gel network + HEC Cellulose integration enables 1500°C stability in a flexible thin film.



1

Geometry-Independent Application

Conformable to complex irregularities.

2

Extreme Thermal Stability

Structural integrity under high thermal loads.

3

Low Thermal Conductivity

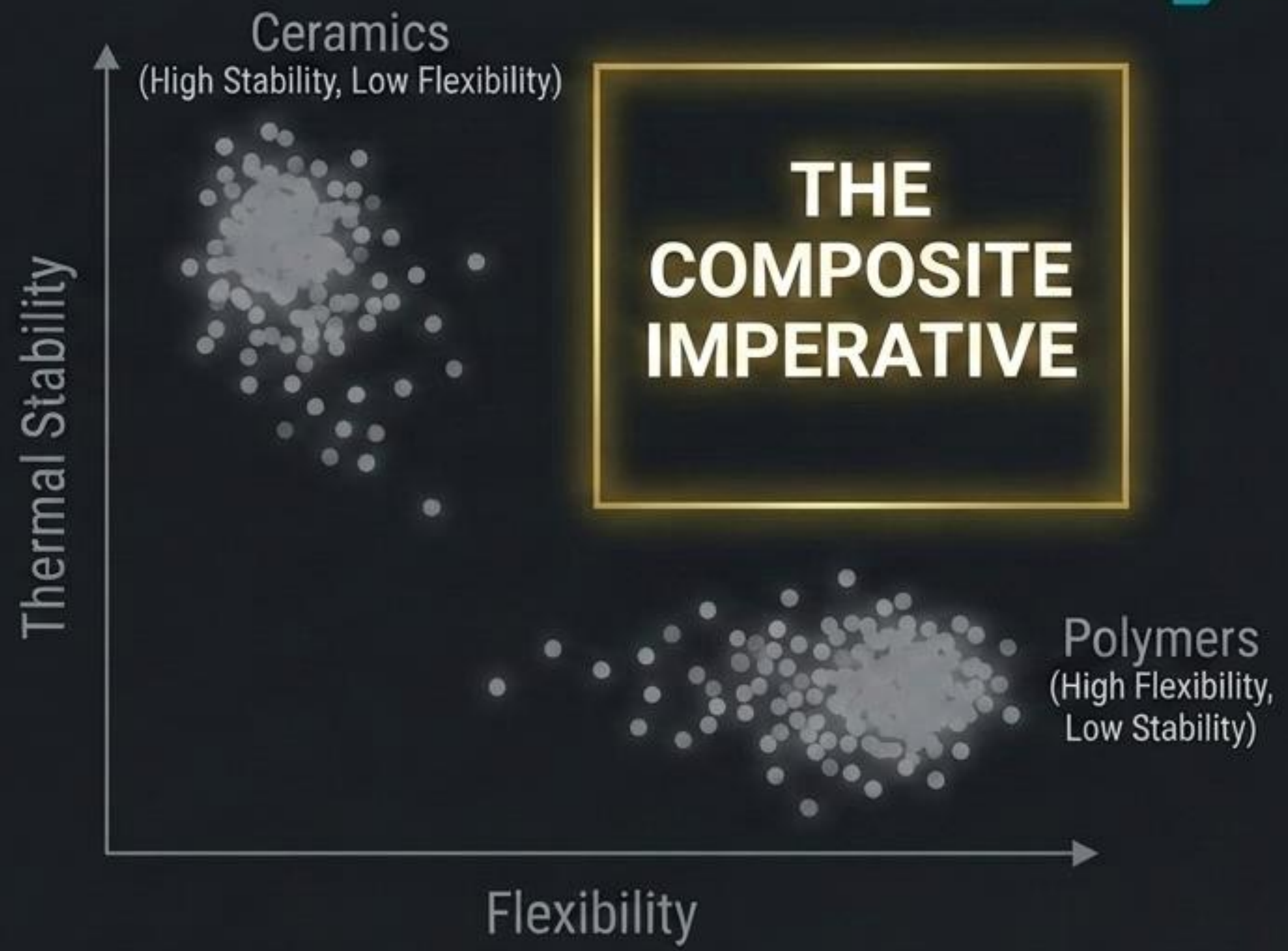
Maximized insulation in a thin profile.

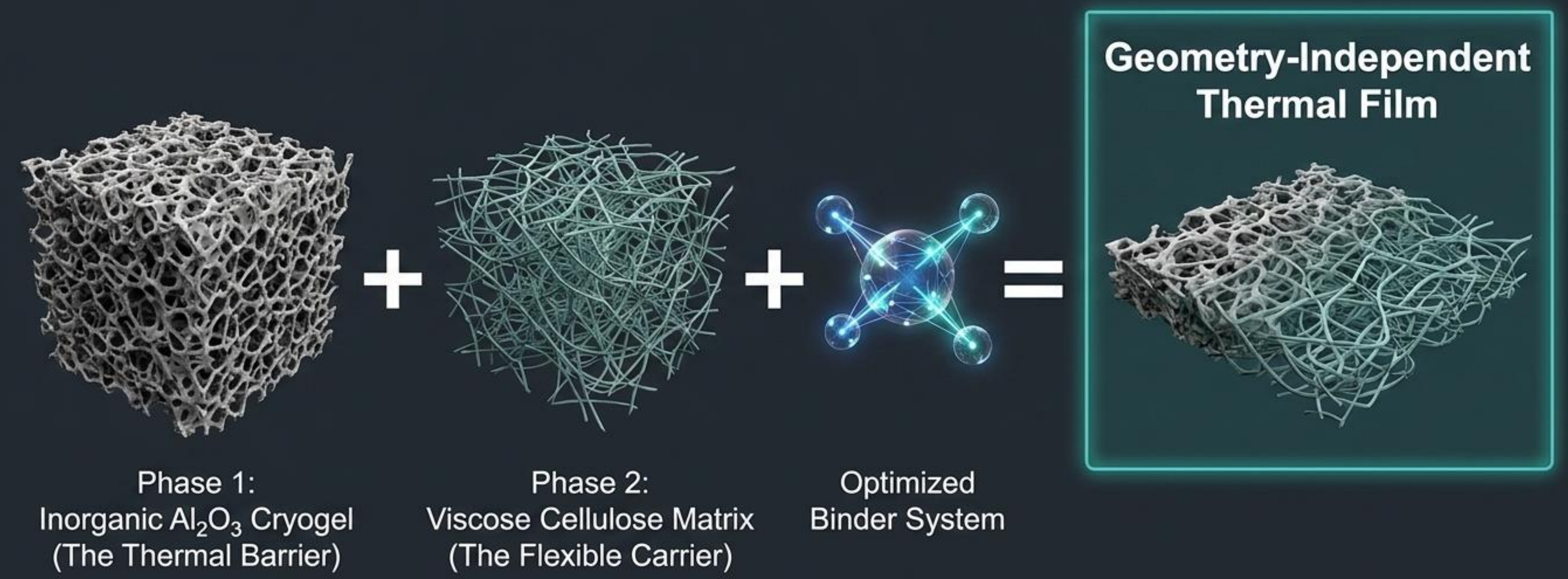
Rigid Ceramics

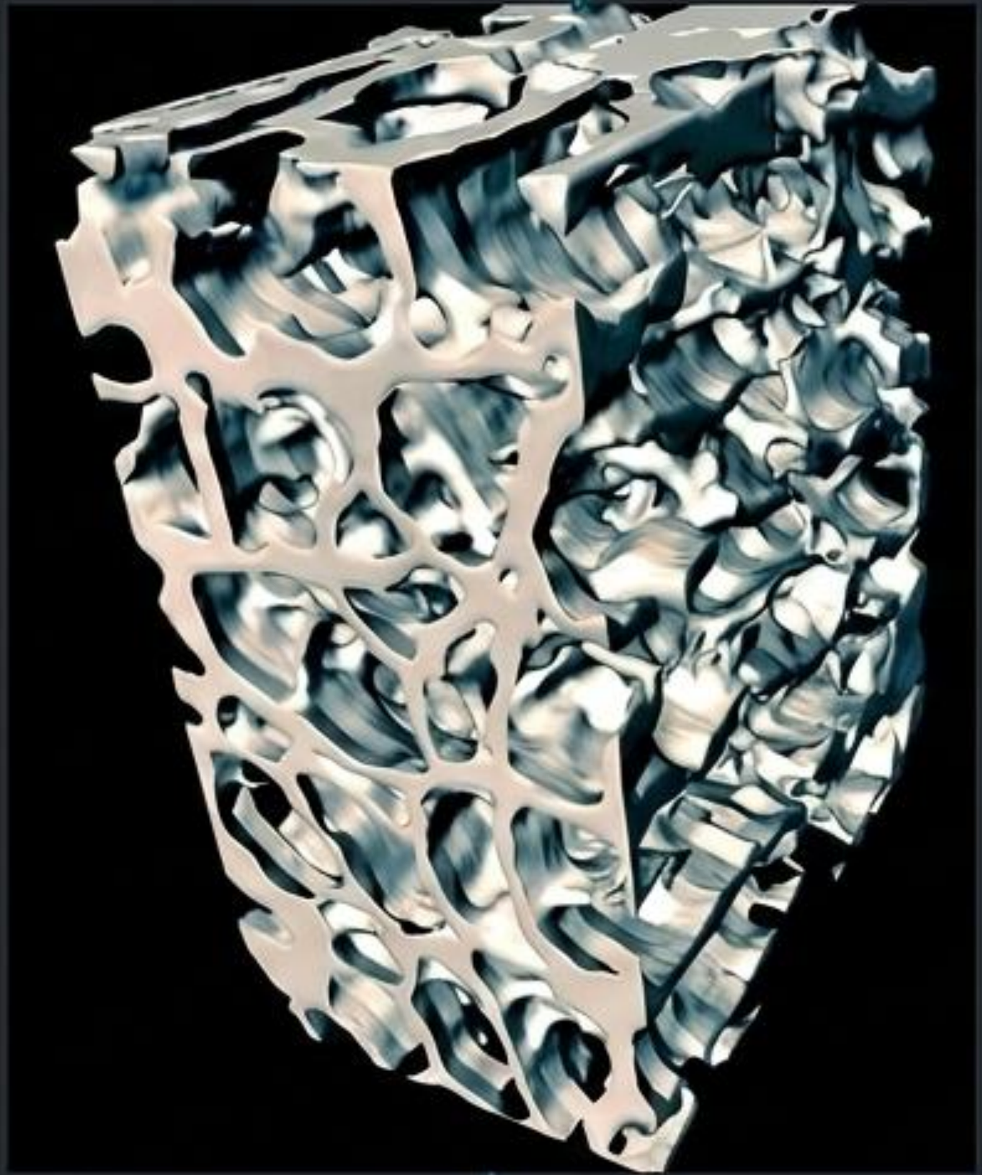
High thermal stability, but highly brittle.
Fails conformability.

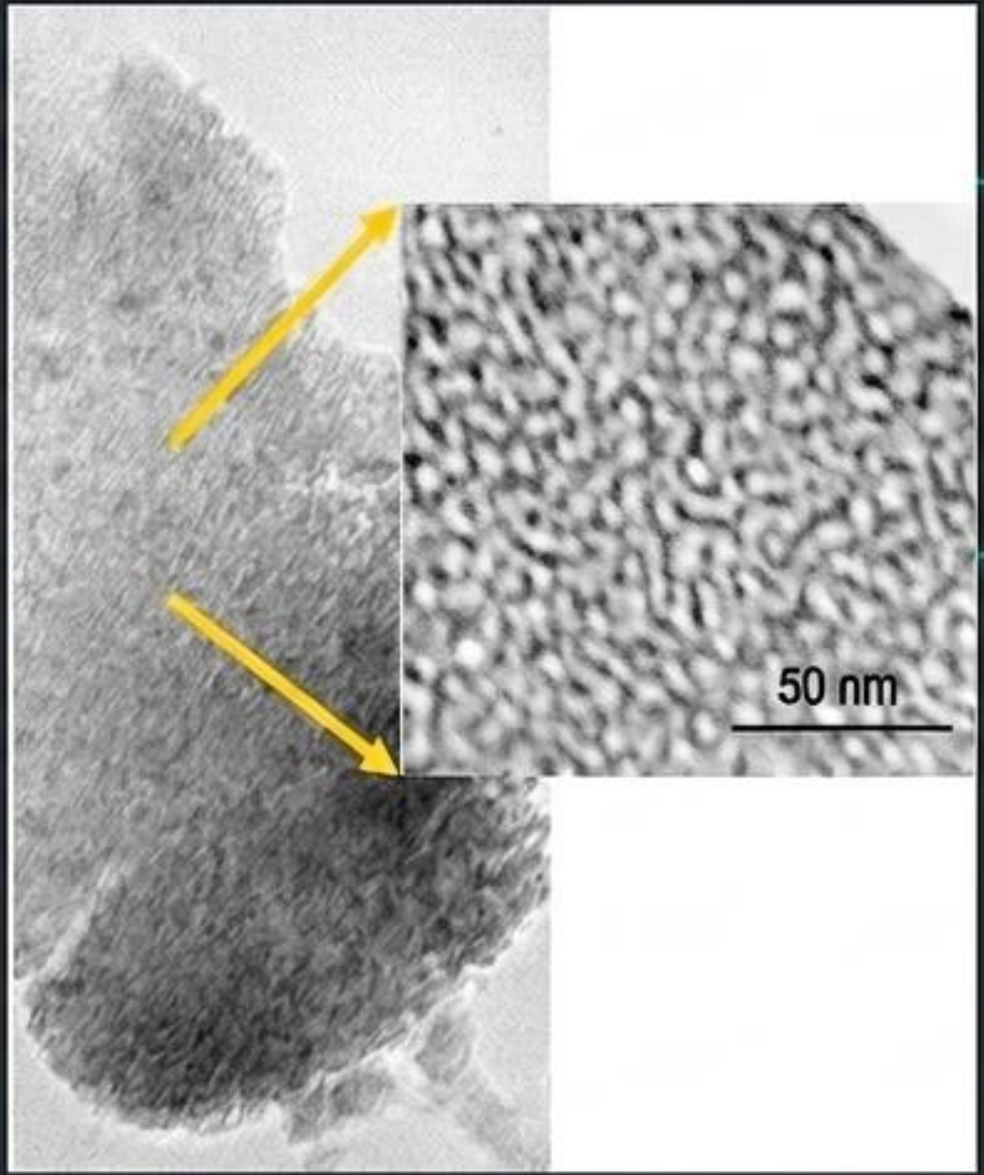
Standard Polymers

Highly flexible, but rapidly degrade under
thermal stress. Fails temperature constraints.




ATL
Geometry-Independent Thermal Film
Phase 1:
Inorganic Al_2O_3 Cryogel
(The Thermal Barrier)
Phase 2:
Viscose Cellulose Matrix
(The Flexible Carrier)
Optimized
Binder System





Total Porosity: 56–73% | Pore Volume: 3–4 cm^3/g

Hierarchical Structure: 2–20 µm open, anisotropic columnar macropores housing 7–9 nm closed nanopores.

Key Insight: The columnar arrangement directly replaces typical lamellar structures of standard inorganic oxides, enabling superior structural integrity.

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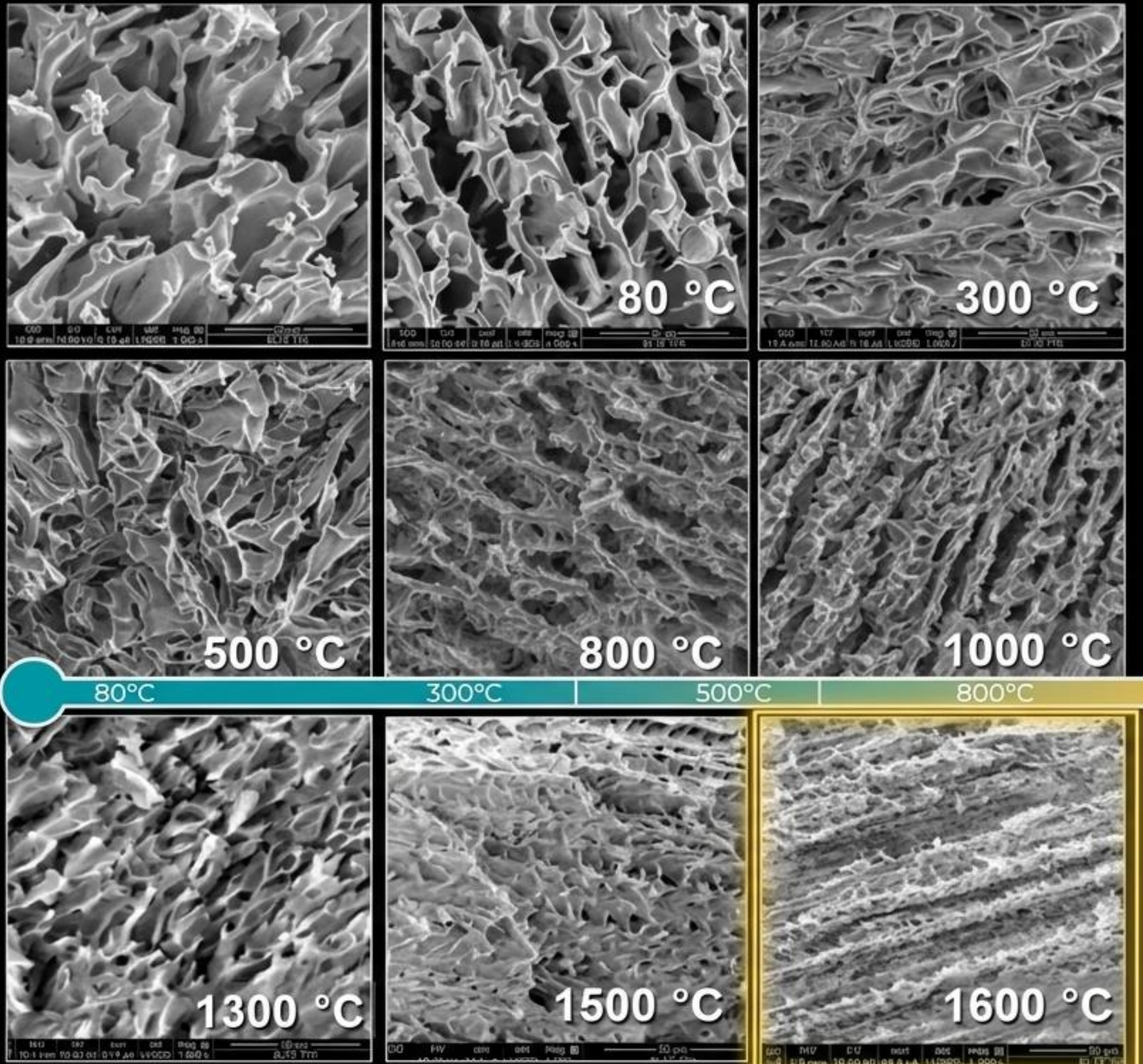
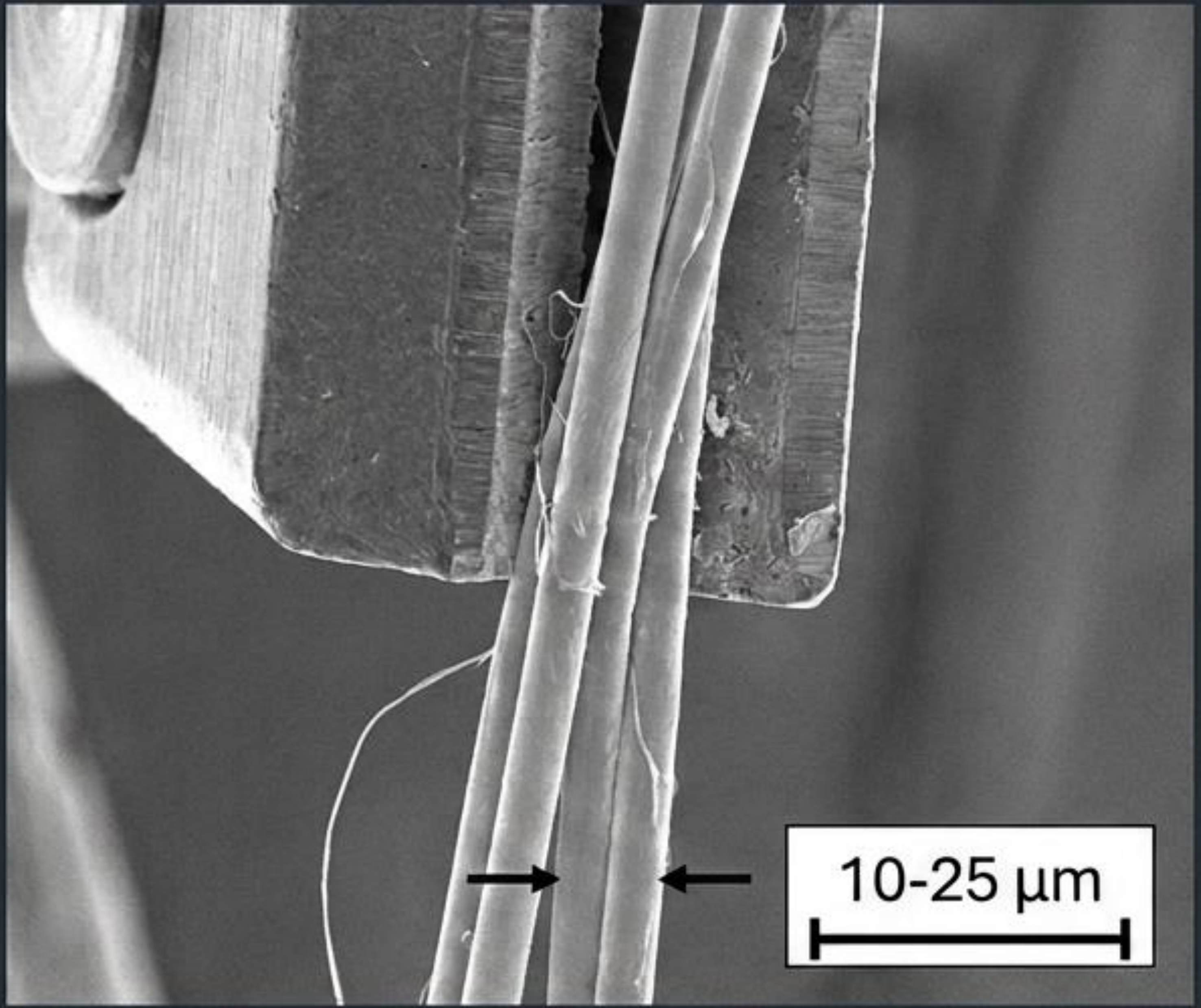
Unprecedented 1500°C+ stability achieved via novel sol-gel 3D continuous network.





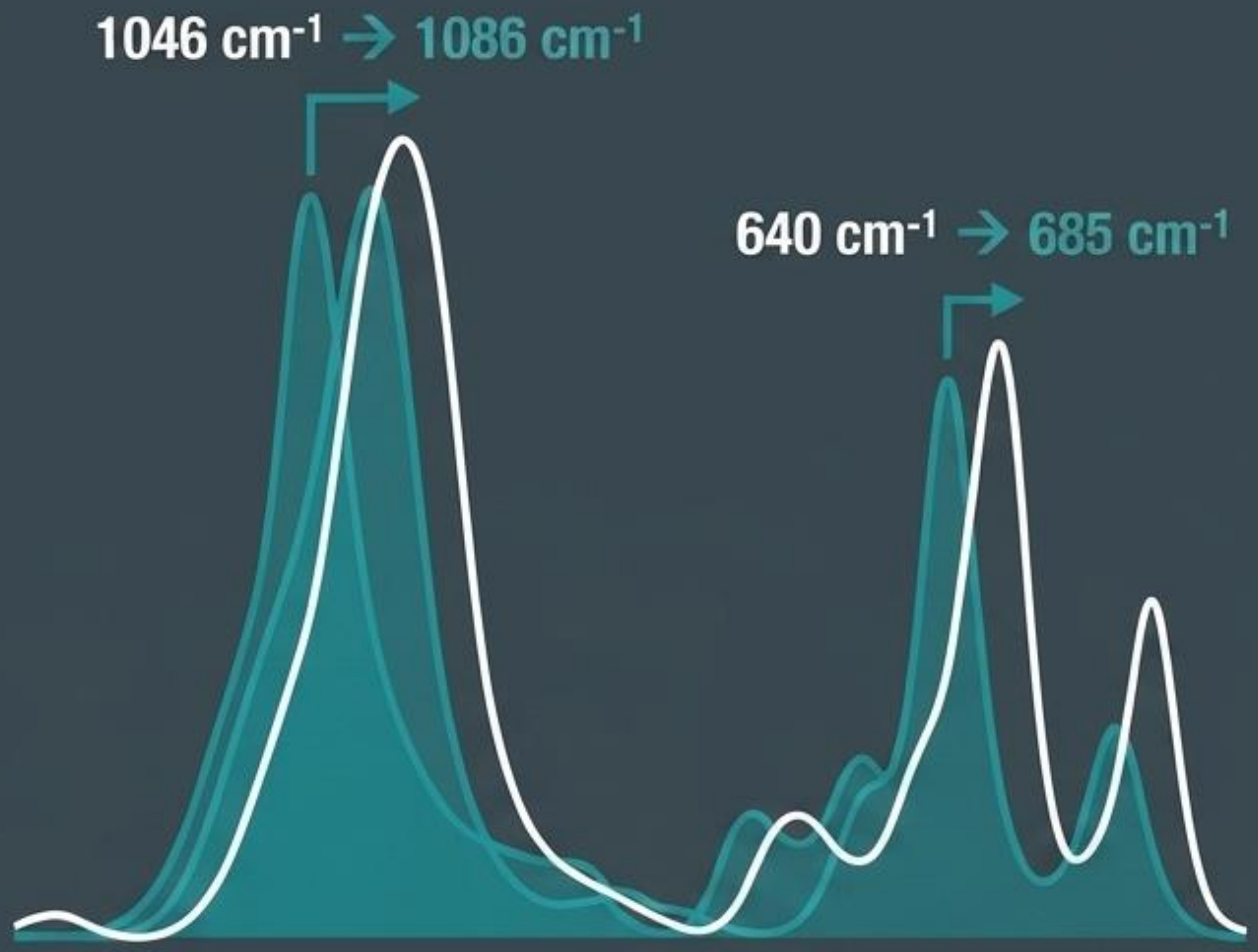
Cost & Sourcing: High scalability, renewable origin.

Thermal Profile: Inherently low thermal conductivity.

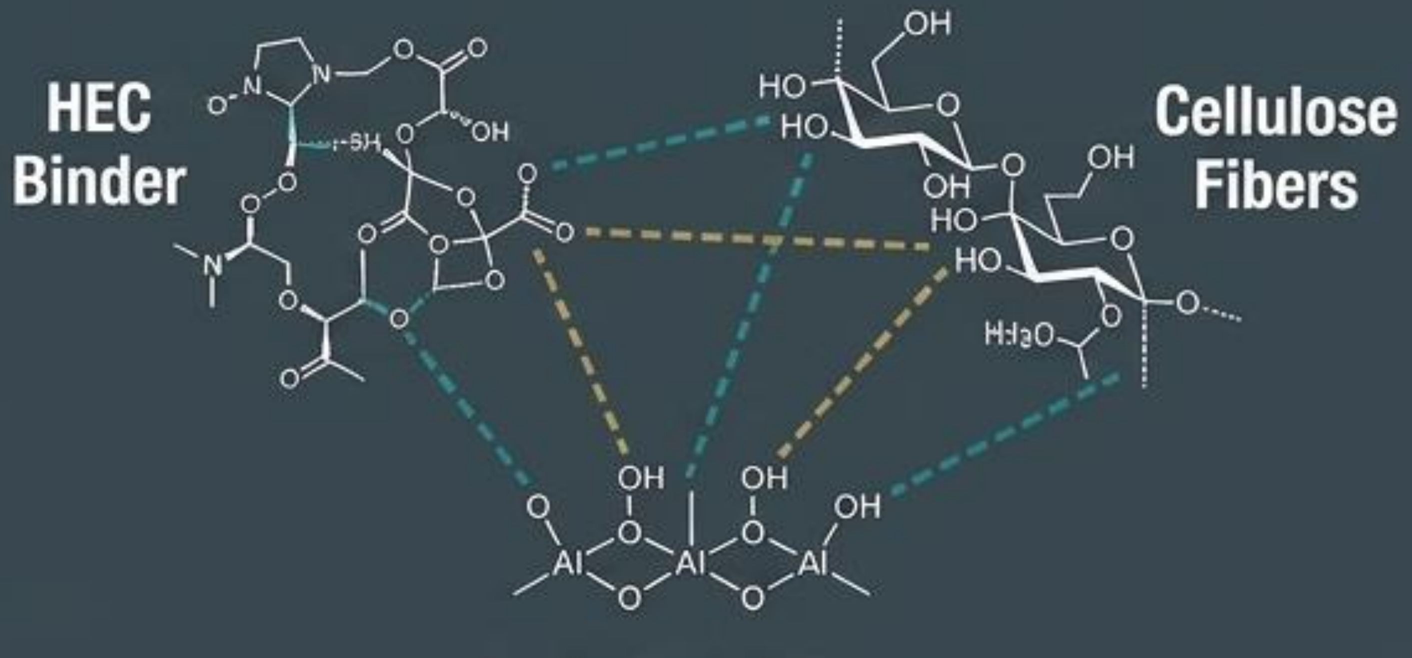
Chemical Utility: Abundant free hydroxyl (-OH) groups allowing for robust secondary bonding without primary chemical reactions.

Aluminum-acetate	16 m/m% HEC Optimization Hydroxyethyl-cellulose (HEC) + 5% Glycerin
• Poor adhesion to substrate.	• Homogeneous dispersion.
• Brittle structure.	• Flexible film.
• High dusting loss.	• Strong secondary bonds.
Fails as a binder	**Optimized System**

Key Insight: Glycerin acts as the critical plasticizer to overcome the inherent rigidity of polymer cellulose systems.



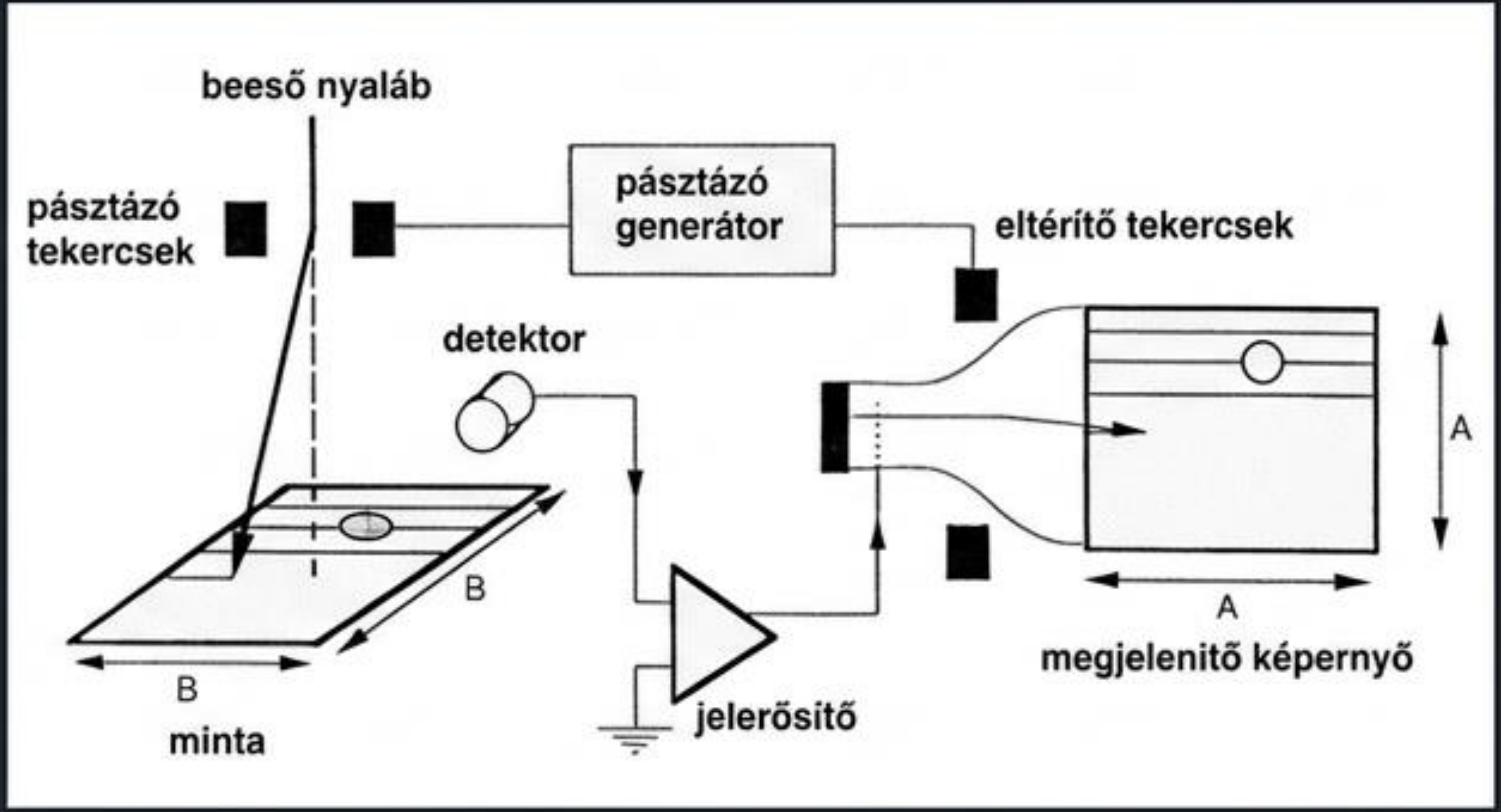
IR Spectroscopy



Al_2O_3 Hydroxyl Groups

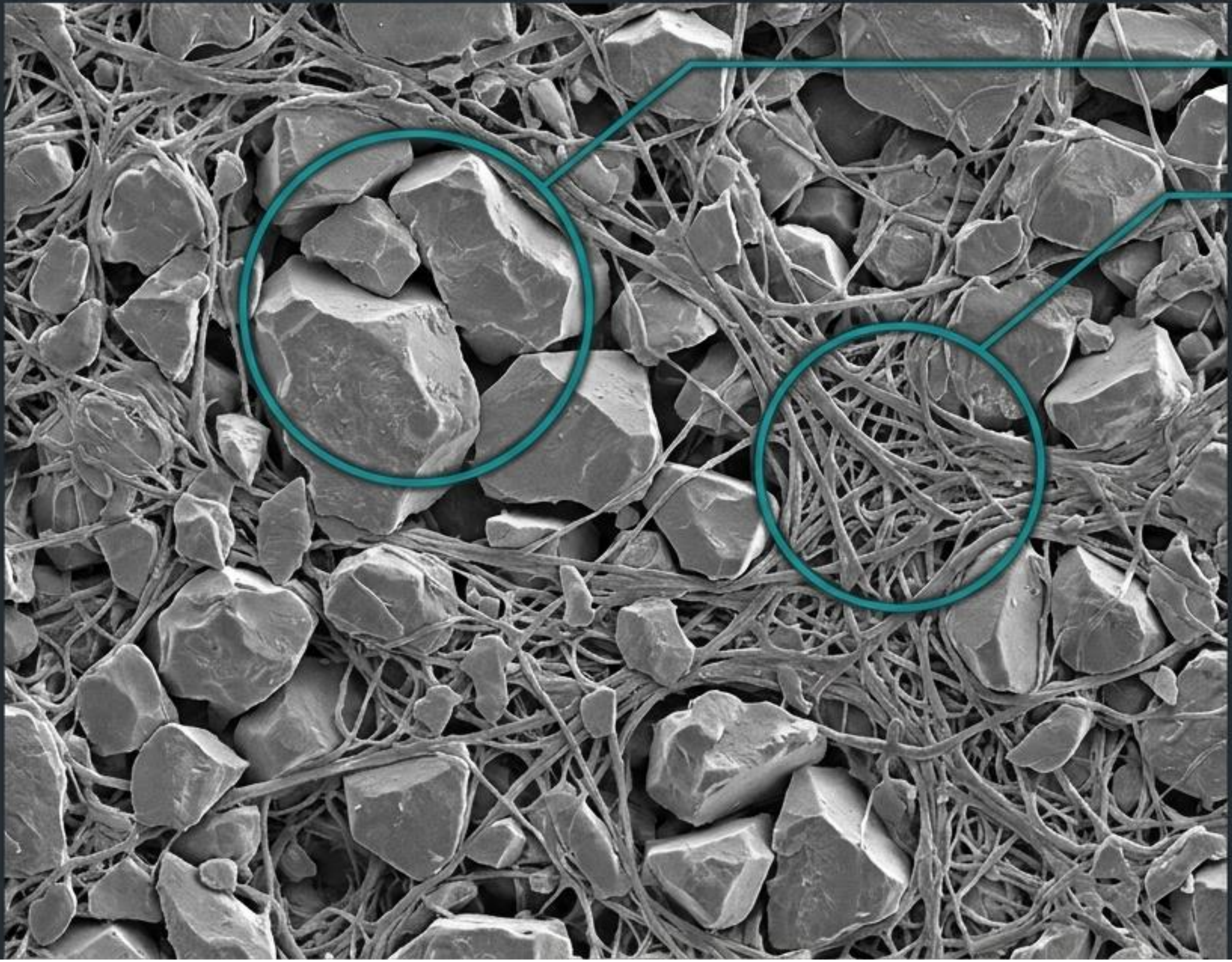
Strong secondary cohesion established. Zero primary chemical bonds required.

1. Insulating ceramics charge under standard electron beams.



2. Applying a conductive metal coating destroys the visibility of the crucial 5-20 nm nanopores.

3. Solution: Environmental SEM (ESEM) compensates for charging, preserving the native nanostructure for accurate evaluation.



1. Firmly embedded Al_2O_3 cryogel particles (< 250 µm).
2. Continuous, intact viscose fiber matrix binding the particles.

Homogeneous dispersion. Zero phase separation.

Deployment Reality: The process is directly analogous to traditional industrial paint application, eliminating the need for custom-machined insulation geometries.

The Thermal Transport Challenge in Thin Films

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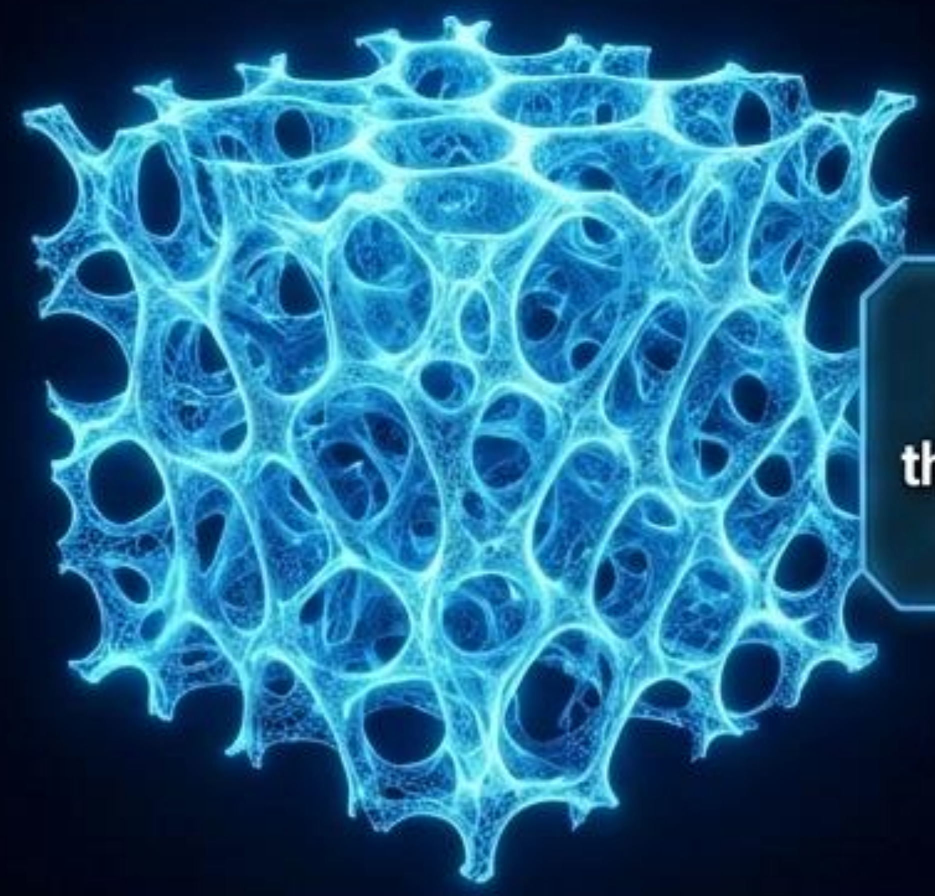
The Brittleness Paradox of Cryogels

MAXIMUM POROSITY



Maximum Porosity. Conduction requires mass. Optimal insulation requires engineered void (porosity >70%).

Vacuum freeze-drying (lyophilization) **creates perfect thermal insulators that inherently shatter into 1-2 mm granules.**

MECHANICAL FAILURE

Mechanical Failure. Extreme porosity in inorganic oxides (Silica/Alumina) destroys structural integrity, preventing thin-film applications.

The Objective: Engineer a system that maintains 3D hierarchical porosity while achieving 2D macroscopic flexibility and adhesion.

Structural Innovation: Hierarchical Porous Composites

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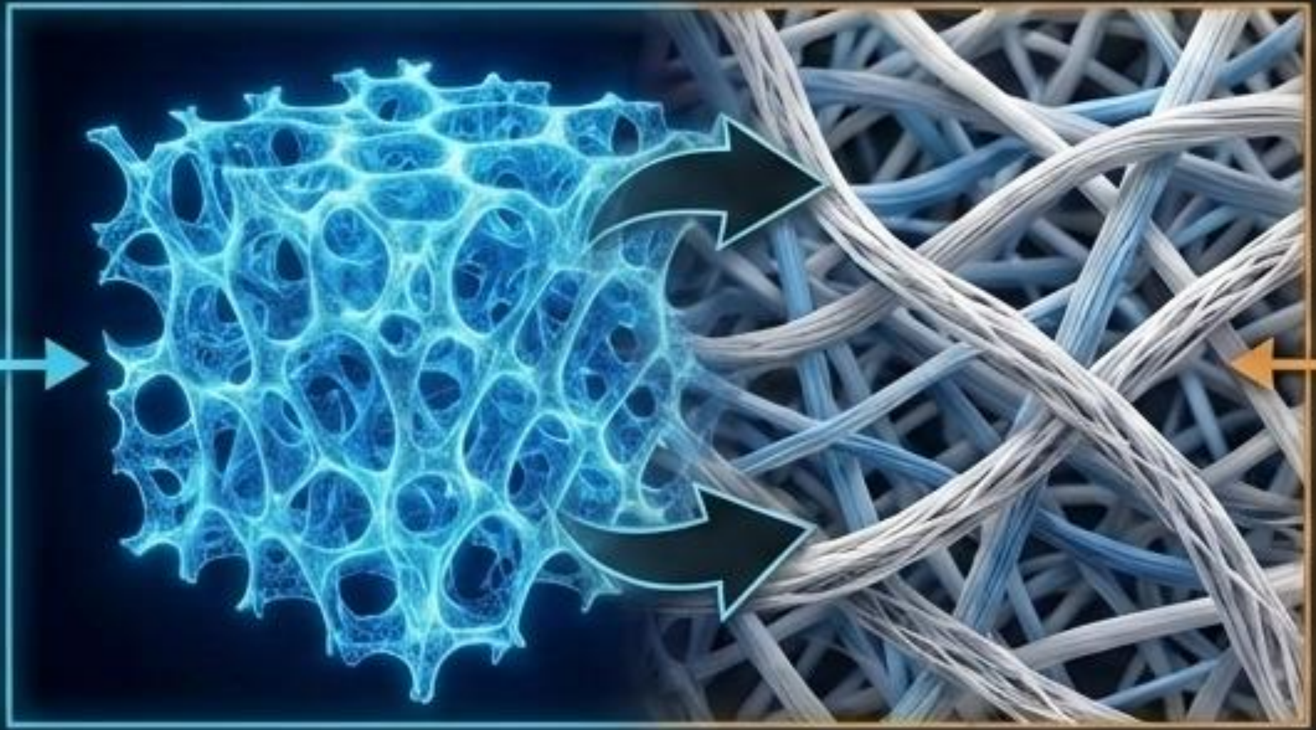
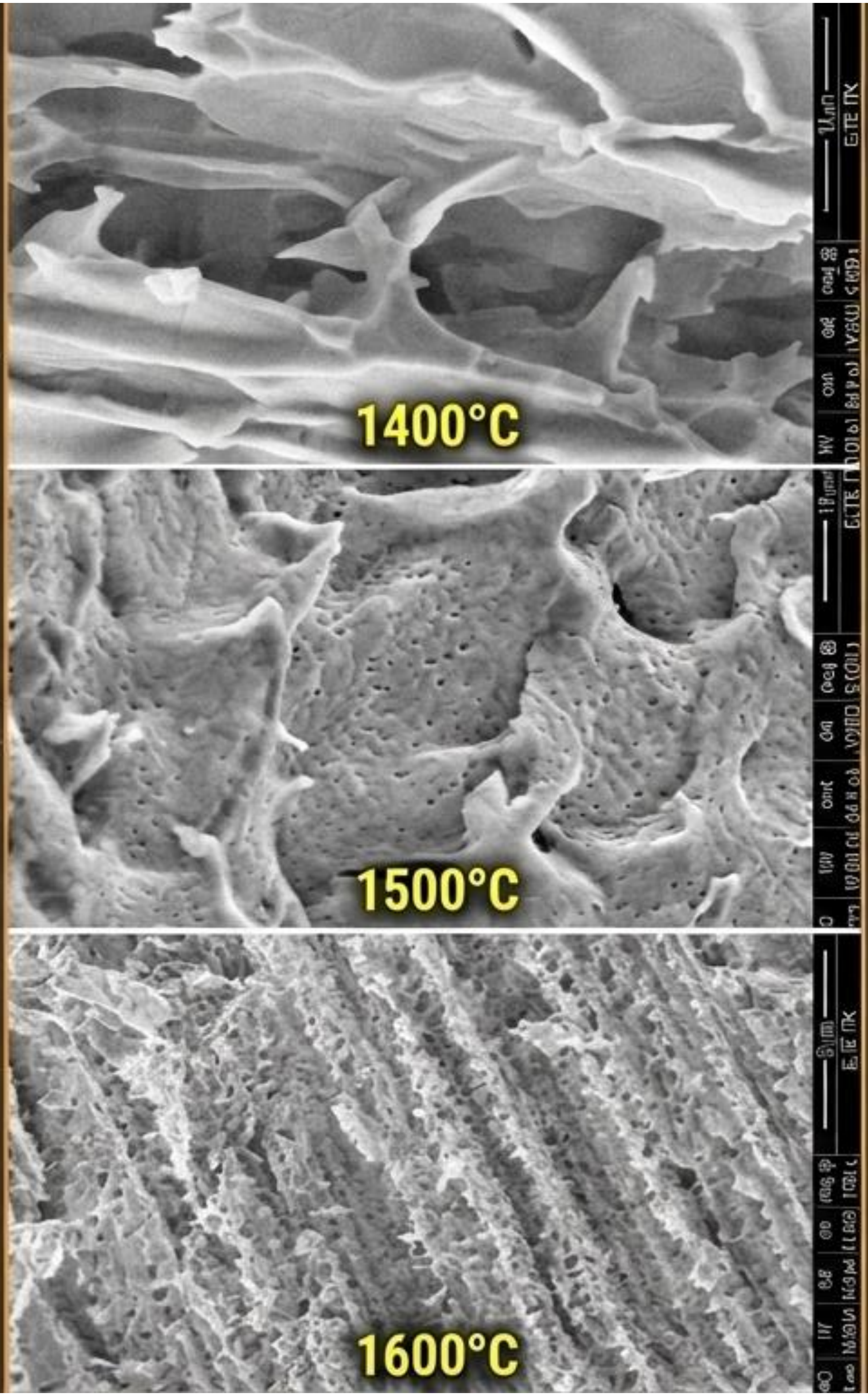
Suspending highly porous 3D cryogels within a continuous fibrous matrix.

Layer 1: 3D Inorganic Cryogels
(trapping air in meso/nanopores)



Layer 2: 1D/2D Fibrous Matrix
(providing tensile strength and flexibility)

Track A: Silica-Cellulose Systems	Track B: Pure Alumina Systems
• **Target:** < 350°C applications • **Matrix:** Woven Cellulose / Viscose • **Advantage:** Cost-effective scaling via waterglass precursor	• **Target:** 1000°C - 1600°C extreme environments • **Matrix:** Porous Al_2O_3 electrospun fibers • **Advantage:** Absolute thermal degradation resistance



Alumina Cryogels: Engineering for Extreme Temperatures

Thermally stable up to 1600°C

- **Total Porosity: 71 - 72%**
- **Macropore Diameter: 7 - 11 μm**
  (Blocks convective heat transfer)
- **Nanopore Diameter: 8 - 11 nm**
  (Traps air in pore walls, minimizing conduction)
- **Density: 0.17 - 0.18 g/cm³**

Scientific Insight

- Rapid freezing (~100°C sec^{-1}) combined with lyophilization forces solvent crystallization, creating a highly organized hierarchical pore network resilient to extreme thermal stress.

Synthesis of Porous Alumina Fibers

Transforming structural reinforcements into active thermal barriers.

Electrospinning / Rotary Spinning → Al-nitrate / Al-chloride Precursor Optimization → Controlled Step-heating

The Breakthrough:

Conventional Al_2O_3 fibers act as thermal bridges. By utilizing a sol-gel method with Aluminum nitrate and controlled heating, we successfully spun porous fibers (10-30 μm diameter) with ~40 nm internal pores.

Heating Profile Impact

Heating Profile	Porosity (%)	Pore Size (nm)
Slow & Stepped	40-45% Porosity	5-8 nm pores
Medium (300°C/h)	65-72% Porosity	70-150 nm pores
Rapid Flash (1500°C/h)	Up to 80% Porosity	Macroporous

Result: A fully continuous 3D structure that maintains mechanical flexibility while actively resisting heat flow.

Silica (SiO_2) Cryogels: Cost-Effective Micro-Structures

Scalable sol-gel synthesis for < 350°C applications.

Precursor Optimization

Traditional Method

TEOS (Tetraethyl orthosilicate).

Expensive, slow gelation (50-70 hours).

ATL Innovation

Waterglass (Na_2SiO_3 aqueous solution).

Drastically reduced cost, water-soluble, utilizing HNO_3 acidic catalysis.

Key Outcome: A highly stable, low-cost thermal barrier with performance comparable to premium precursors.

Structural Optimization

- Eliminated HCl catalysis to prevent NaCl crystal formation.
- Achieved homogenous mesoporous structures.

Porosity: ~60 - 75%

Pore Size: 20 - 200 nm

Overcoming Brittleness: Cellulose Matrix Integration

The Integration Challenge: Lyophilized SiO_2 fractures into 1-2 mm granules.
To form a thin film, these must be bound into a continuous phase.

Silica Cryogel

Superior insulation
(λ = 0.029 - 0.04 W/mK)
+ Extreme Brittleness.

Cellulose Fiber

Mechanical flexibility
+ Moderate insulation
(~300°C limit).

Why Cellulose?

- Low thermal conductivity.
- High mechanical strength and elasticity.
- Cost-effective, renewable biological polymer (Viscose).
- Free hydroxyl groups allow for secondary bonding.

The Composite

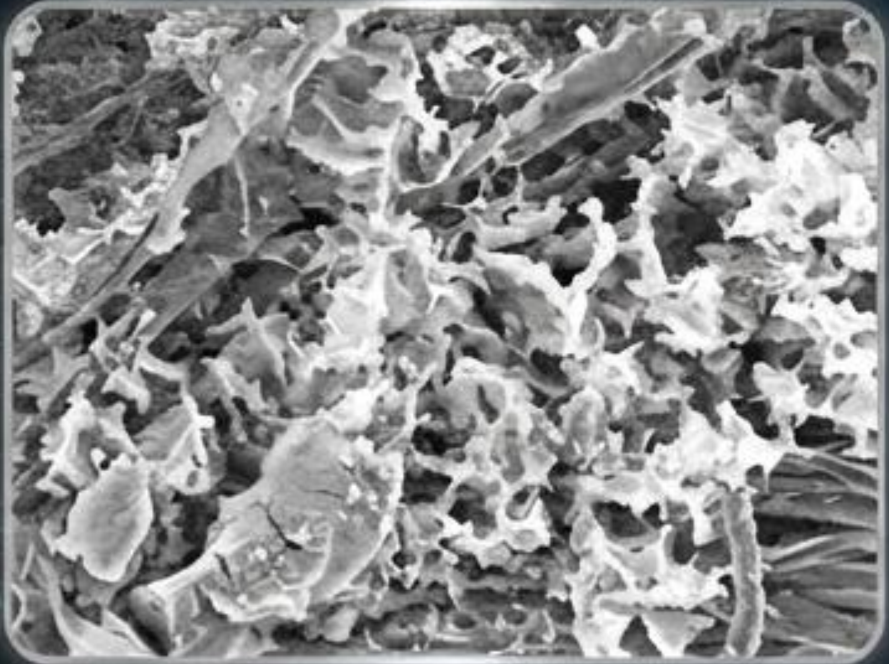
Flexible, highly insulative thin film.

The Formulation: Dispersing 500°C heat-treated cryogel granules into a chemically treated cellulose fiber matrix.

Matrix Synergy: Woven Textiles vs. Short Fibers

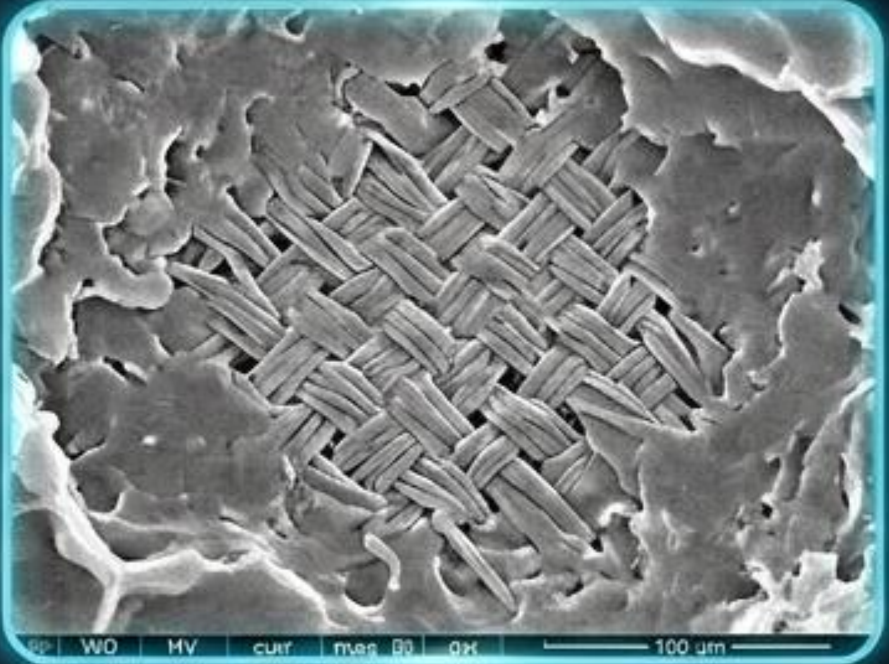
Engineering the matrix prior to lyophilization.

Method A: Short Viscose Fibers



- Added post-lyophilization. Creates good homogeneity and easily follows irregular structured surfaces.
- Prone to minor cracking; requires Hydroxyethyl cellulose (HEC) to eliminate dusting (< 0.03%).

Method B: 100% Woven Cellulose Textile (The Breakthrough)



- Textile added to the acidic silica gel prior to lyophilization (utilizing 2M NaOH/Urea treatment).
- Result: Completely eliminates cracking, forming a continuous monolithic system.
- Porosity Boost: Increases total pore volume of the silica cryogel from 60-63% up to 70-75%, maximizing thermal resistance.

Metrological Challenges in Nanostructured Thin Films

The Problem: Standard building-material thermal tests are fundamentally incompatible with advanced nanocomposite thin films.

Sources of Error in Standard Testing

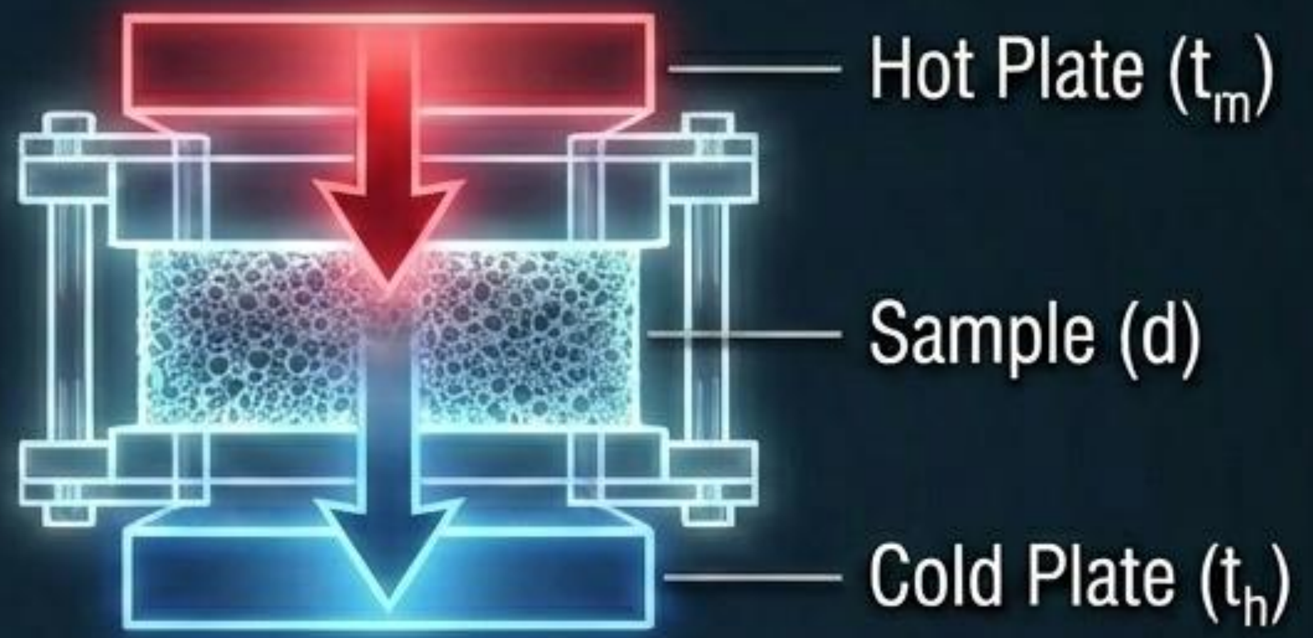
- **Thickness limits**: Standard tests cannot accurately resolve films < 5 mm.
- **Surface roughness & Nanoporosity**: Causes immense contact resistance errors.
- **Substrate interference**: Inability to separate film performance from substrate thermal mass.

The Analytical Mandate

- Requires a method capable of analyzing stationary thermal states.
- Needs extreme precision (targeting $\lambda < 0.5$ W/mK).
- Must evaluate parallel surface planes without destructive compression.

Proprietary Steady-State Thermal Metrology

Custom Fourier-law stationary testing apparatus.



$$q \cdot d = \lambda \cdot (t_m - t_h)$$

Methodology

- Determines exact heat flux density (q) passing through the sample.
- Captures true temperature gradients ($t_m - t_h$) independent of contact resistance anomalies.
- Accounts for precise sample thickness (d).

Capabilities

Portable, high-precision, explicitly optimized for nanostructured thin films and capable of mapping complex substrate-film boundary dynamics.

Thermal Validation: SiO_2-Cellulose Performance

Matching commercial bulk insulators in a thin-film geometry.

Insight: The woven textile integration drastically reduces thermal bridging, pushing the composite's performance into the elite tier of stationary insulating materials.

Extreme Environment Validation: Pure Alumina Composites

Complete thermal stability > 1000°C.

0.040 W/(m·K)

Thermal Conductivity

1500°C - 1600°C

Continuous Thermal Limit

The Ultimate Composite:
Al_2O_3 Cryogel + Porous Al_2O_3 Fiber Matrix.

Scientific Context: Published thermal conductivity data for monolithic Al_2O_3 cryogels typically range from 0.10 - 0.50 W/(mK). The ATL composite achieves an unprecedented 0.040 W/(mK) through the synergistic integration of hierarchically porous components, eliminating solid conduction pathways.

Mechanical Resilience & Substrate Adhesion

Engineered for geometric flexibility and robust real-world application.

Substrate Adhesion (Pull-Off Force)		
Glass	**Polystyrene**	**Polypropylene**
12,206 ± 250 (Excellent bonding)	13,416 ± 300 (Optimal bonding)	8,429 ± 100 (Moderate bonding)

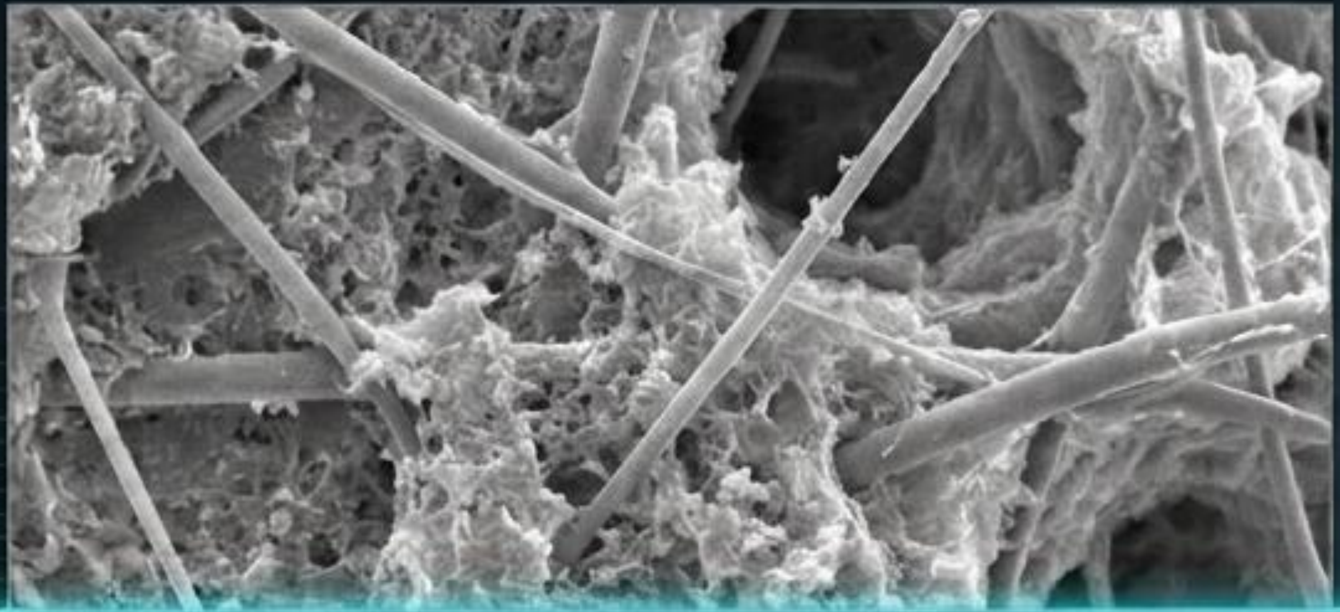
Dynamic Condensation Recovery: Films exposed to 80% relative humidity temporarily lose adhesion and soften. When returned to 40-45% RH, the matrix fully dries within 24 hours, completely restoring adhesion.

Porosity Stability: Zero degradation of the nanostructure occurs during extreme moisture cycling.

Industrial Benchmarking: The High-Temperature Frontier

Comparing ATL Al_2O_3 composites against aerospace-grade aerogels.

NASA Benchmark



NASA / Aspen Spaceloft

- Composition: Silica aerogel + organic polymer fibers.
- Thermal Conductivity: 0.015 - 0.023 W/(m·K)
- Operational Limit: Max 200°C (organic matrix degrades).

ATL Benchmark

ATL Al_2O_3 Composite

- Composition: Al_2O_3 cryogel + porous Al_2O_3 fibers.
- Thermal Conductivity: 0.040 W/(m·K)
- Operational Limit: Up to 1600°C.

The Conclusion: ATL provides near-aerogel thermal performance in extreme-temperature environments where traditional aerogels incinerate. Ideal for hydrogen propulsion and aerospace shielding.

The Next Generation of Thermal Management

Structural Mastery

- Successfully resolved the cryogel brittleness paradox.
- Achieved 75-80% functional porosity integrated within a flexible, continuous matrix.

Scale & Cost Efficiency (< 350°C)

- Replaced expensive TEOS with waterglass for scalable SiO_2 composites.
- Achieved elite 0.042 W/mK performance matching commercial Rockwool in a geometry-agnostic thin film.

Extreme Environment Supremacy (> 1000°C)

- Pioneered fully porous Al_2O_3 electrospun fibers.
- Delivered 0.040 W/mK insulation at 1600°C, unlocking new paradigms for aerospace, heavy industry, and energy systems.

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Questions & Discussion