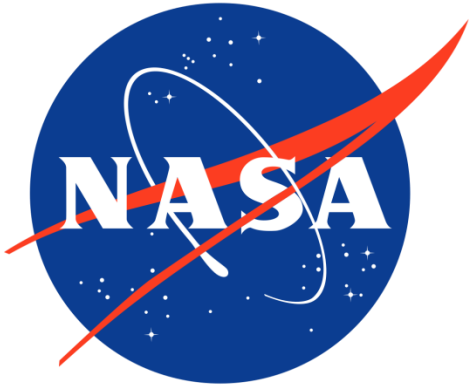
The 3D Printing of Polyimide Aerogels



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Jessica Cashman, NASA Glenn Research Center
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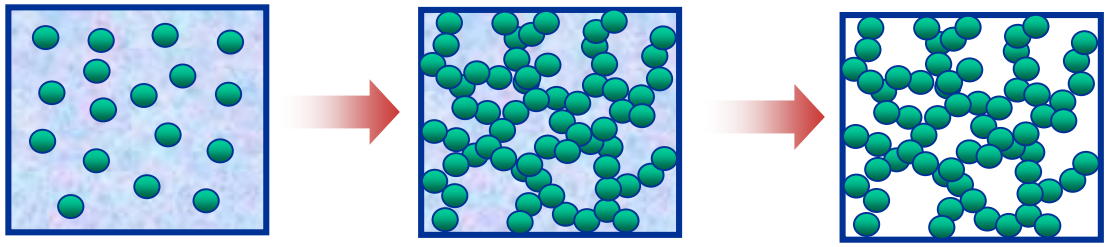
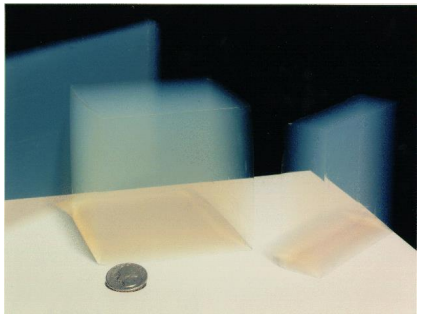
What are Aerogels?



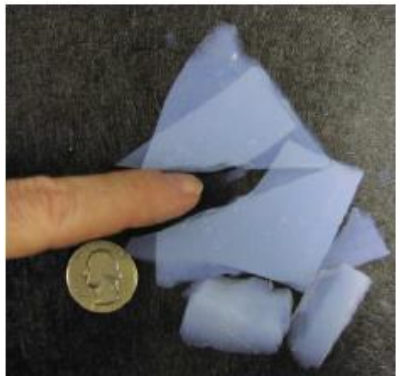
Sol Gel Aerogel



Typical monolithic silica aerogel

Silica Aerogels

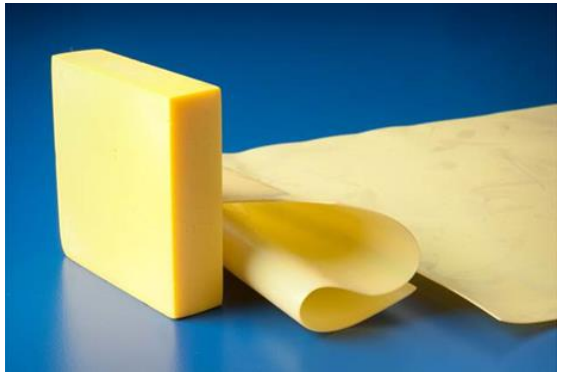
- Highly porous material made by removing liquid portion of a wet gel
- Low density (< 0.3 g/cm^3)
- High porosity (> 90 % air)
- High surface area (200 – 650 m^2/g) with extremely small pore size (10 – 40 nm)
- Very fragile – limited applications



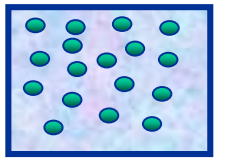
Cosmic dust collector Stardust Mission

Cross-linked Aromatic Polyimide Aerogels

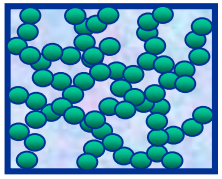
- Superior mechanical properties compared to silica aerogels while still maintaining good physical properties (density, porosity, surface area, etc.)
- Low thermal conductivity
- High temperature stability up to 400 ^{o}C (short term)
- Moisture resistance depending on backbone chemistry
- Durable and flexible
- Easy to manufacture into thin film



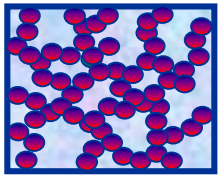
Reaction Scheme for Cross-linked PI Aerogel



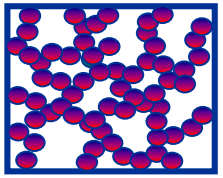
Monomers



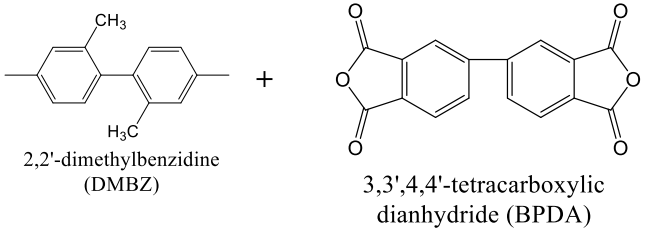
Polyamic Acid Solution



Polyimide Gel



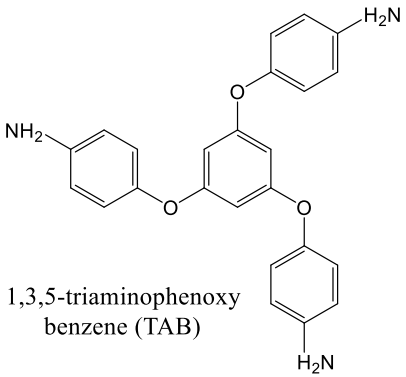
Polyimide Aerogel



2,2'-dimethylbenzidine (DMBZ)

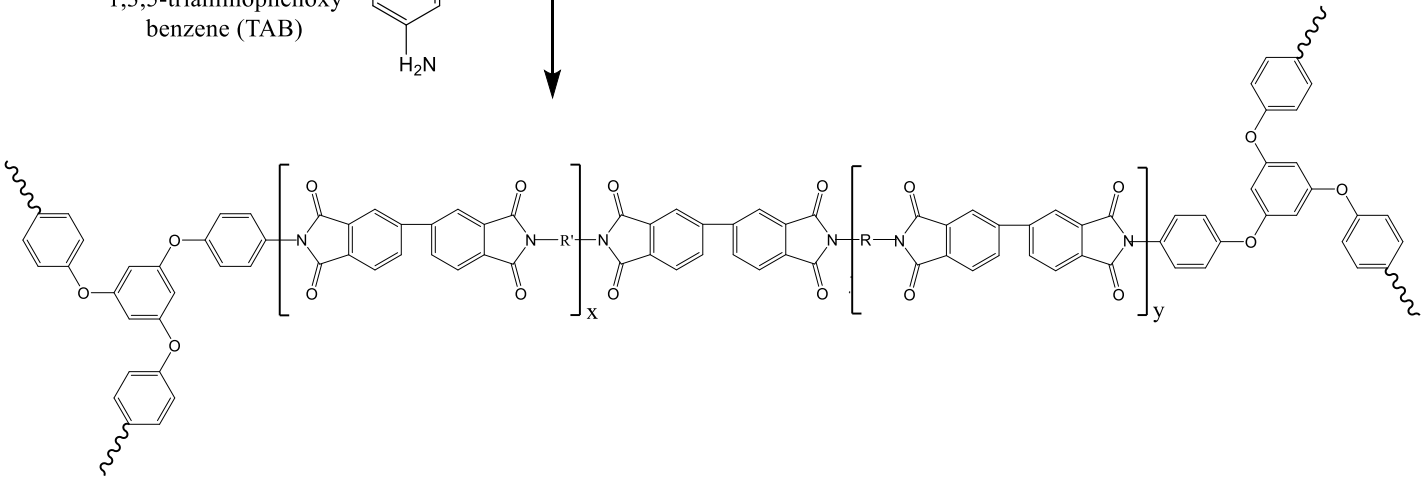
+

3,3',4,4'-tetracarboxylic dianhydride (BPDA)

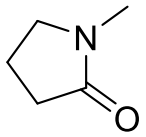


1,3,5-triaminophenoxy benzene (TAB)

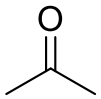
Acetic anhydride (AA)
Triethyl amine (TEA)



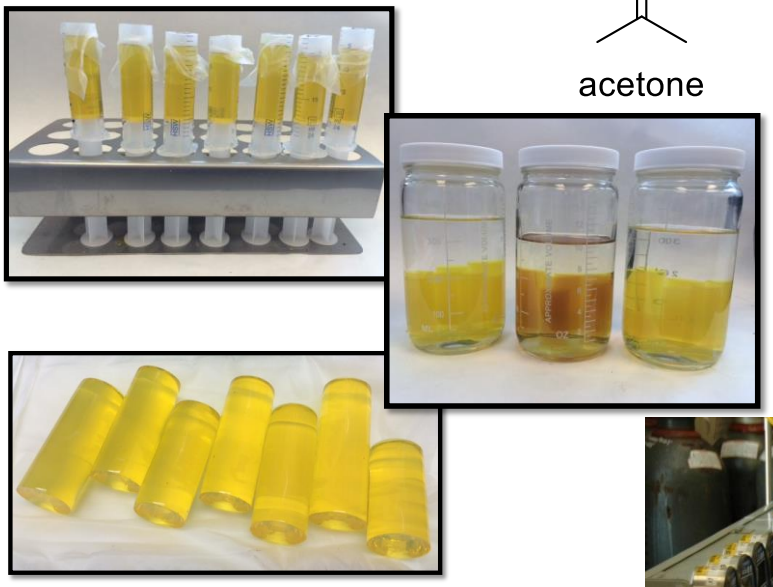
Supercritical Fluid Extraction

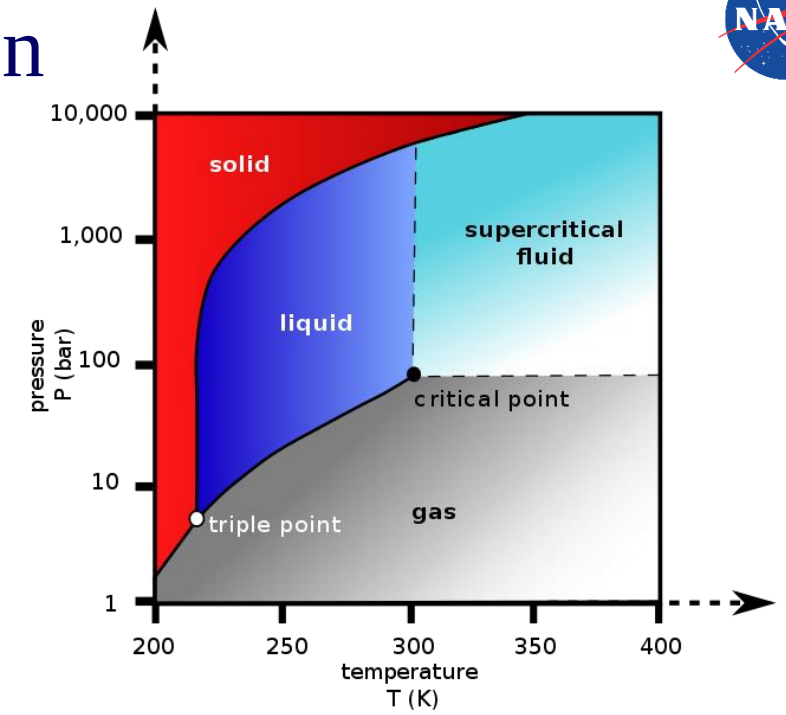


n-methylpyrrolidone (NMP)



acetone

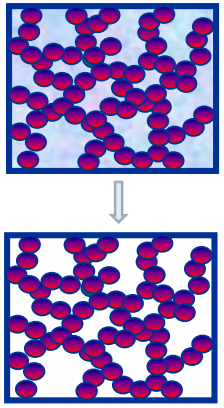




Final stage of aerogel production

O=C=O

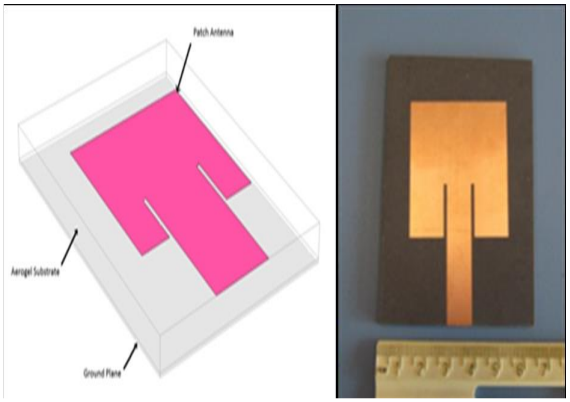
CO_2, (l)



Polyimide Gel

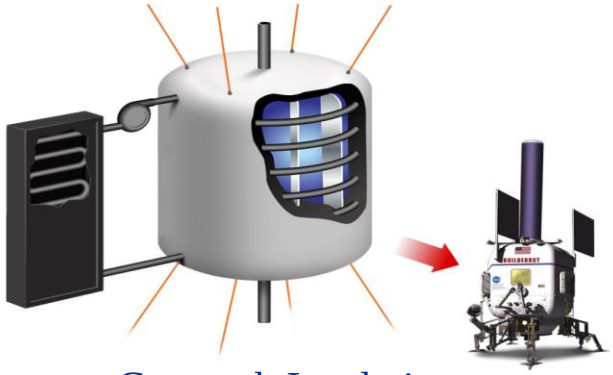
Polyimide Aerogel

Potential Applications



Antenna Substrate

Sandwich Structure



Cryotank Insulation

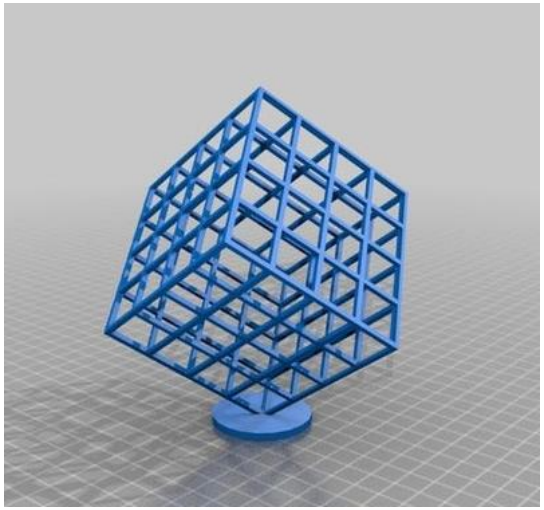
Heat Shielding

Insulation for EVA Suits

Ultra-lightweight, Multifunctional Structures for Habitats and Rovers

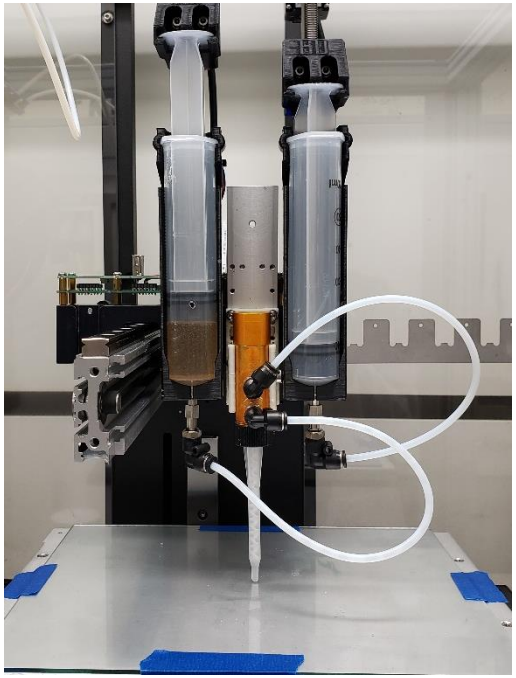
Purpose of this Research

- Novel concept for additive manufacturing
- Currently molds are needed to shape aerogels, which limits it's complexity
- Increasing the architectural complexity of aerogels increases its versatility
- Expand polyimide aerogels in its uses for every day life as well as its uses as a material for extreme environments

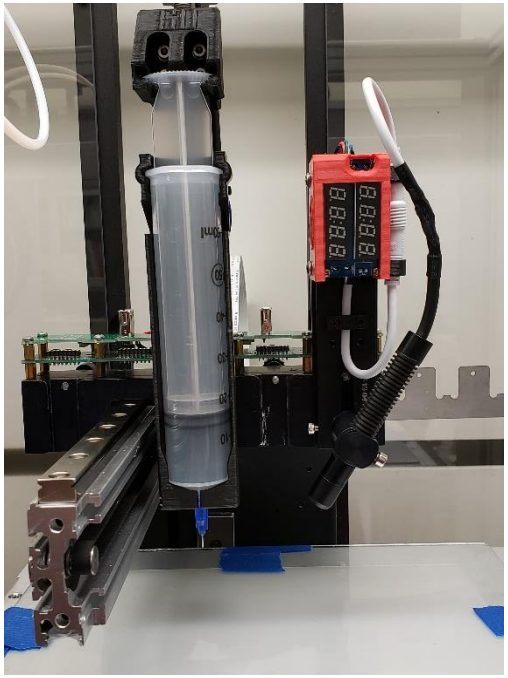


Three Approaches

Direct Printing



Mixing Tip



UV Curing

Common Variables

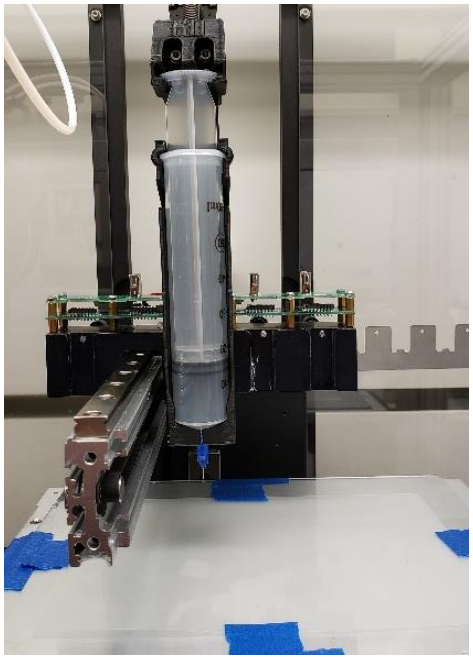
Chemical formulation:

- Weight percentage
- Gel time

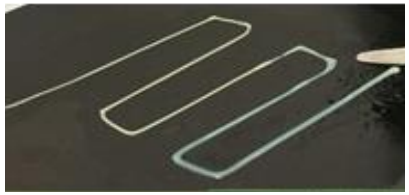
Printer:

- Print speed
- Overlap percentage
- Layer height
- Poly(amic) acid/TEA ratio
- Tip diameter

Direct Printing



Design



Actual

Process

- Multiple vials of sol
- Mixing in TEA one at a time
- Printing until solidified
- Switch out sol

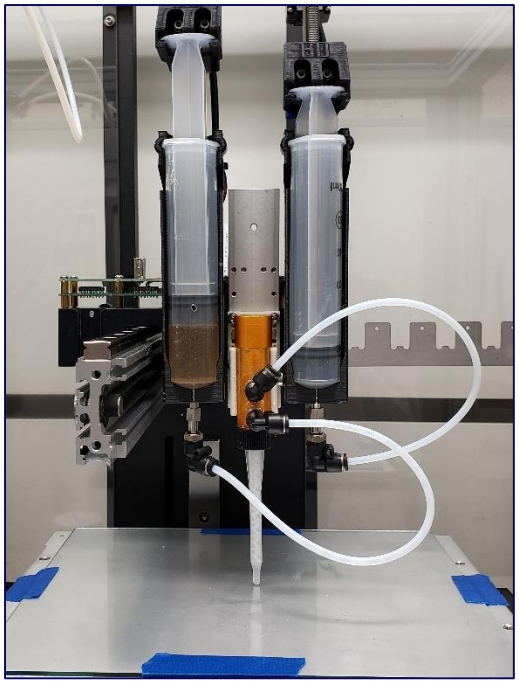
Pros

- Predictable
- Can print up to 10 minutes without gelling

Cons

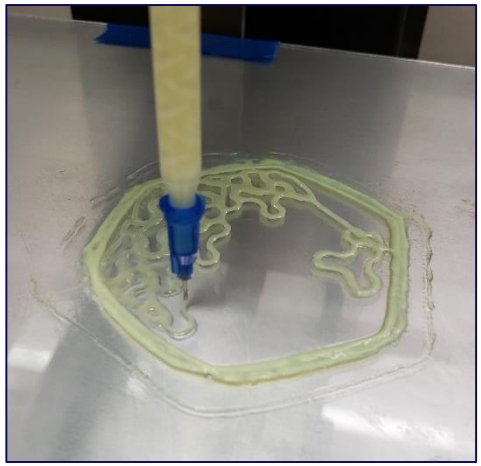
- Long process
- Switching syringes
- Not sustainable for long print jobs
- Sol will gel in syringe

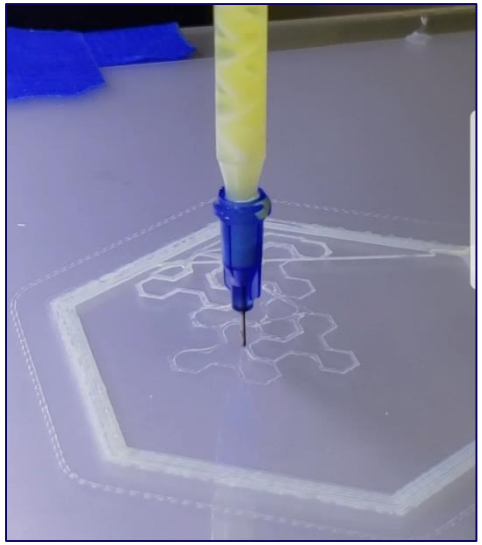
Mixing Tip



Process

- Poly(amic) acid in one extruder, TEA in another
- Connected to a mixing tip
- Solidifies soon after extruding

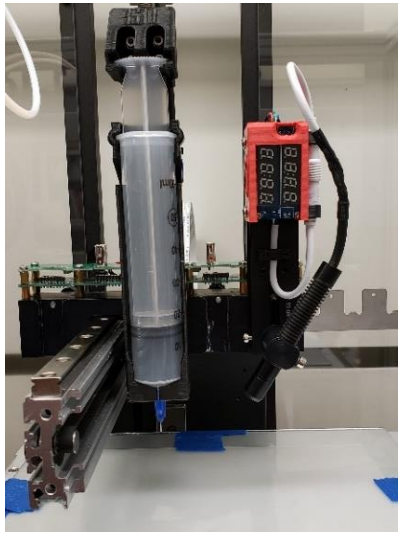


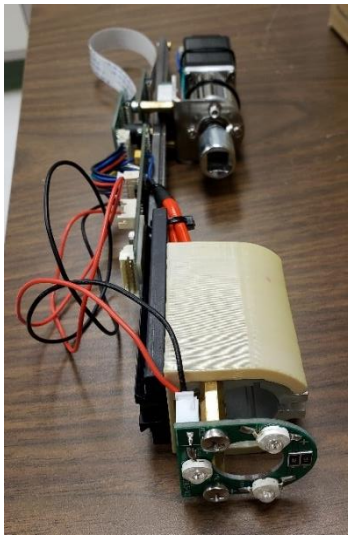


Overcoming:

- Gelling in mixing tip
- Pressure buildup

UV Curing





- UV curable polyimide
- Varying parameters
  - Intensity
  - Speed
- Two ways to UV cure
  - Pen: wavelength - 405 nm, high intensity
  - LED Array: wavelength - 405nm, low intensity

  Then post cure in UV curing chamber

Conclusions and On-going Work

The process for 3D printing polyimide aerogels can lead to applications in aeronautics, industry, and space. As it becomes a more defined process, more intricate parts can be produced.

Continuation:

- Improve printing parameters and formulations
- Improve repeatability
- Demonstrate feasibility of 3D printing UV curable polymers
- Characterize the 3D printed samples

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