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Nanomechanics of Aerogels

Major Qualifying Project

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Abstract

The goal of this project is to study and analyze the nanomechanics of aerogels that are doped with materials like Carbon Nanotubes (CNT) and Graphene. Aerogels provide the framework for nanomaterial to exist in a three dimensional space. Doped Aerogels can potentially be used in applications like enhanced thermal insulation and effective light trapping due to its multi-functionality and superior mechanical properties. These hybrid aerogels are cheaper and easier to fabricate than pure carbon aerogels, which are known to exhibit catalytic properties. By analyzing the physical quantities in the aerogels such as molecular dynamics and mass, thermal fluctuations, electrical conductivity, we can explore new areas of material science with carbon nanotechnology [1].

Introduction

Nanomechanics is the science that deals with the fundamental mechanical properties like thermal, elastic and kinetic quantities of a physical system at a nanoscale. It helps in explaining some unexpected phenomenon's and pathological behavior compared to our existing understanding of the mechanics of macroscopic systems. The term aerogel is used to describe a sol-gel material in which the liquid component of the gel is replaced with gas. This results a solid nanostructure in tact without pore collapse in which 90~99% is air by volume. Aerogels can exhibit a wide range of density from 1 kg.m^{-3} (lower than the density of air) to 1000 kg.m^{-3} (above the solid density), which induces dramatic changes in the properties. Aerogels can exhibit versatile properties such as low thermal conductivity, dielectric constant, high specific surface area, adjustable density and refractive index. This is due to the high porosity as well as dual microscopic and macroscopic structural features. Aerogels are attractive materials, which can

find application in thermal insulations, chemical absorbents, sensors, catalytic carriers and space explorations [2].

Aerogel was first invented in 1931 by Kistler, and was named so (air +gel) because the liquid component inside the wet gel was replaced by air without damaging the solid microstructure. The fabrication of aerogels has not taken off to its full potential due to the high manufacturing costs and time consuming processes during synthesis. In 1968, Teichner's group worked on a significant innovation in which water-glass/water systems were replaced with organic solvents to prepare aerogels using Tetramethyorthosilicate. Russo found a method to introduce safer tetraethylorthosilicate in 1986, which led to the development of carbon dioxide (CO₂) supercritical fluid drying. More recently, unique aerogels such as carbon nanotube (CNT) aerogels, graphene aerogels, silicon aerogels and carbide aerogels have been developed and added to the aerogel community [2].

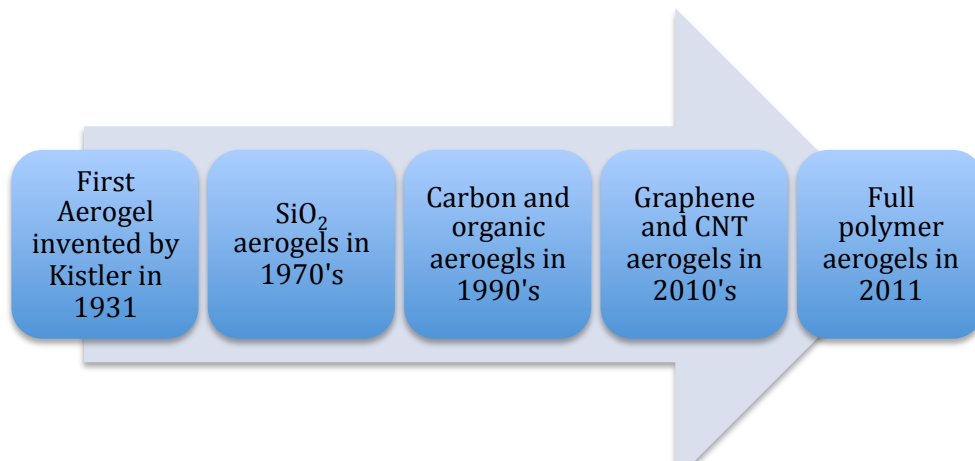


Figure 1: Timeline of the evolution of aerogels

Although Silica aerogels are the most widely studied class, their potential applications in industry, aerospace and daily life has been limited due to property analysis and hygroscopic nature. Hybrid aerogels with multiple components exhibit enhanced properties and multi-functions attracting more interest recently. In this paper, recent advances in the field of hybrid aerogels will be discussed, with particular emphasis on their fabrication methods, structures, properties and applications [3].

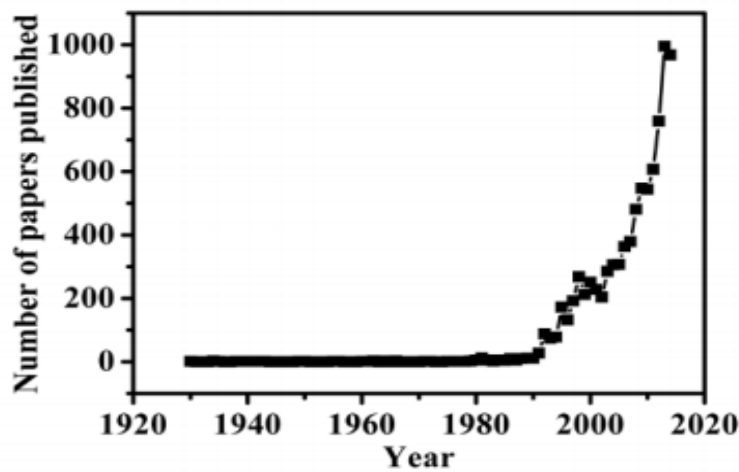


Figure 2: Number of papers published yearly through the last decade on aerogels

Theory

Silica aerogels contain more than 90% air and less than 10% solid silica. This is mixed with a cross-link network structure with desirable properties such as low thermal conductivity, low bulk density and high specific surface area. Such properties makes silica aerogels doped with carbon-based nanomaterials the ideal candidate for various applications like filters for pollutants, thermal insulators and drug delivery. High manufacturing costs, time and mechanical properties severely impede scaling up and exploration in various applications.

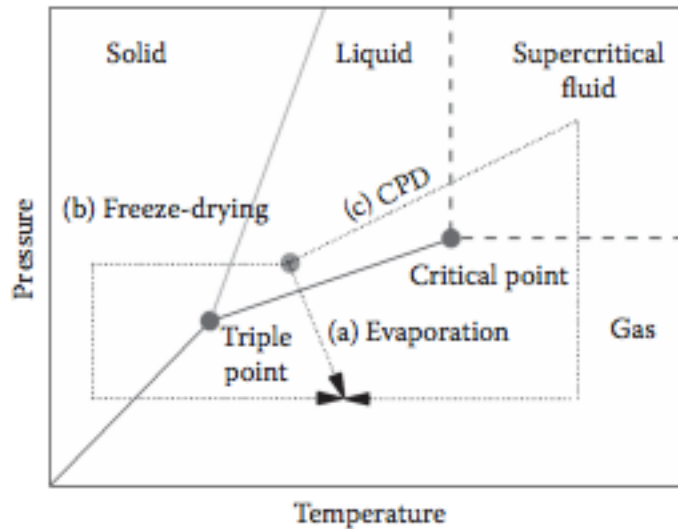


Figure 3: Principles of three drying methods (evaporation, freeze-drying, and CPD)

There are three possible methods of liquid removal from the hydrogel structure during fabrication: (1) evaporation, (2) Critical Point Drying (CPD), and (3) freeze-drying. The CPD or freeze-drying methods are the preferred options to remove the pore liquid effectively and still preserve the gel networks of the aerogel. Selection between the two drying methods strongly depends on the cost requirements of the fabricated aerogel [3].

Critical Point Drying Method (CPD)

The CPD method, also known as the supercritical drying method, has been widely used in industry and research to preserve the porous structures in the production of aerogels. During the CPD process, the transformation of liquid to gas takes place through the supercritical region instead of crossing the liquid-gas boundary. The liquid and gas phases mix within the

supercritical region to form a supercritical fluid. Surface tension, which causes the gel structure to collapse, is avoided by using this method.

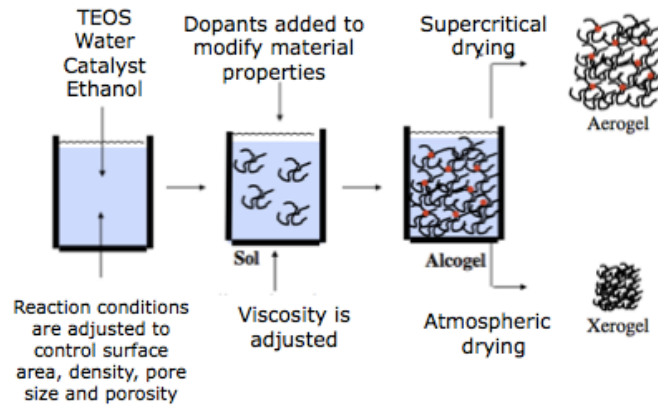


Figure 4: Aerogel production flow using Supercritical Drying Method

Freeze-Drying Method

The Freeze-Drying technique is another common method to eliminate the liquid from hydrogels. In this method, the hydrogels are first frozen and then placed in vacuum. By freezing, the hydrogel transforms to ice and is then heated while still under vacuum below the triple point of water at 0.006 atm. By doing this, the ice directly sublimates into vapor and the aerogel is formed. More microcrystals can be formed simultaneously from a quick freeze thereby helping in building better gel structures [4].

Freeze-Drying is cheaper but more time consuming than CPD. However, CPD method exhibits larger surface areas and better multifunctional properties over those obtained from the Freeze-Drying method [4].

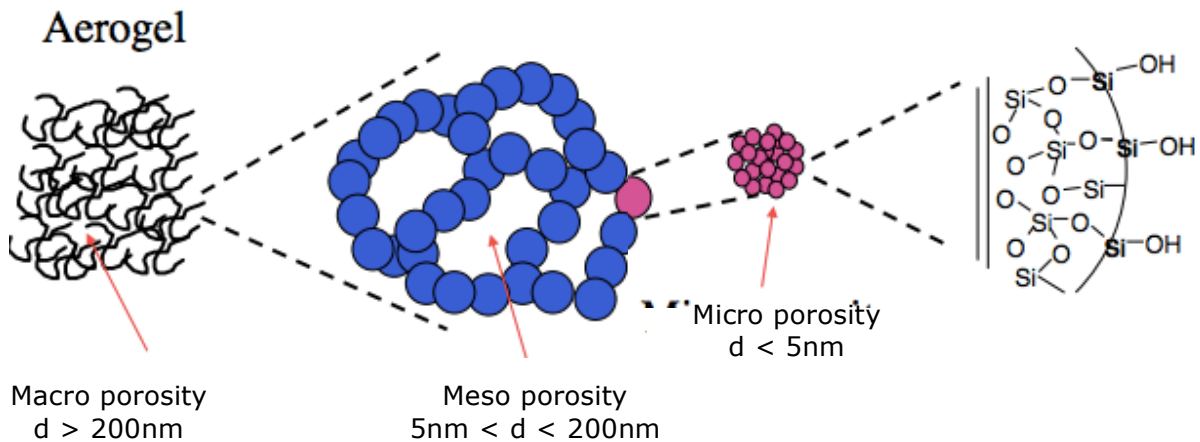


Figure 5: Porosity of aerogels at different scales

Nanomechanics of Carbon Nanotubes

Carbon nanotubes have great potential in material science due to their superior mechanical properties over conventional materials. Prototypes for nano-electro-mechanical devices such as high performance nanomotors, switches and oscillators based on carbon nanotubes have shown great promise. The sp^2 bonds present in the carbon nanotubes have a Young's modulus close to that of a diamond. The Van der Waal interactions are relatively weak between the graphitic shells and this acts as a form of lubrication. The carbon nanotubes possess a unique combination of small size, high stiffness, low density and high strength. They have a broad range of electronic properties from metallic to p- and n-doped semiconducting [8].

High quality nanotubes can be produced in small quantities by methods based on cooling carbon plasma that can be generated between two graphitic electrodes in an inert atmosphere during an arc discharge. Other methods capable of producing CNTs in industrial quantities are based on Chemical Vapor Deposition (CVD) and catalytic decomposition of various hydrocarbons like methane or acetylene mixed with nitrogen or hydrogen in the presence of

catalysts. This offers the possibility of controlling the growth of nanotubes by patterning the catalyst and is more suitable for producing nanoscale structures with integrated CNTs [8].

Structure of Carbon Nanotubes

Carbon Nanotubes are considered to be single sheet of graphite that is curved up. It is the most stable form of crystalline carbon rolled in to a cylindrical shape with axial symmetry. The approximate diameter is 1.2 nm and can range between 0.7-10 nm. Rolling the graphite sheet along one of the symmetry axis gives either a zigzag tube ($m=0$) or armchair tube ($n=m$). Rolling up the sheet in a direction other than the symmetry axis gives a chiral nanotube [6]. The chiral angle and circumference of the cylinder can be varied.

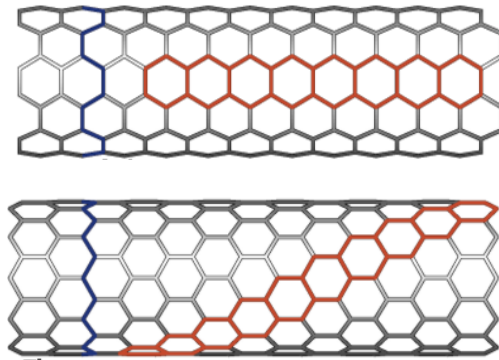


Figure 6: Carbon Nanotube consisting of 320 carbon atoms displaying the armchair and zigzag configurations generated by AViZ

The structure of a single wall Carbon Nanotube is expressed in terms of one-dimensional unit cell, defined by the vector:

$$\mathbf{v} = n \cdot \mathbf{a}_1 + m \cdot \mathbf{a}_2$$

Where \mathbf{a}_1 and \mathbf{a}_2 are unit vectors, and n and m are integers. Nanotubes that are constructed in this way are called (n, m) nanotubes. In most cases, $\mathbf{a}_1 = \mathbf{a}_2 = 2.49\text{\AA}$.

Classical laws can be applied to describe the mechanical properties by introducing concepts like Young's Modulus at the nanoscale level. Hooke's Law can be applied to a series expansion of any potential around the equilibrium position. Stress Energy can be used to define the Young's Modulus.

$$E_{Young} = \frac{1}{V_0} \cdot \frac{\delta^2 E}{\delta \epsilon^2}; \quad \epsilon = 0$$

Nanotubes can be semiconducting or metallic, depending on the chirality of the graphene sheet. Carbon Nanotubes are produced with a distribution of chirality's and electrical properties, which cannot be controlled [6].

Polymer Based Hybrid Aerogels

Hybrid aerogels are fabricated in order to strengthen the nanostructures and suppress nanopores for collapsing. Nanofilters such as SiO₂, graphene, MoS₂, WS₂, and Multi Wall Carbon Nano Tubes (MWCNT) are usually incorporated in to the fabrication techniques to prepare polymer hybrid aerogels [9]. These hybrid aerogels generally exhibit enhanced thermal and mechanical properties as well as multi functions compared to pure polymer aerogels.

Graphene/CNT Based Carbon Aerogels

Novel carbon aerogels such as CNT and graphene aerogels are the most suitable candidates to make ultra light aerogels, which are elastic and conductive in nature. Some enhanced properties that these hybrid aerogels exhibit include good mechanical strength, low density, good electrical conductivity, good elasticity and high aspect ratio. Even though CNT and graphene aerogels have ultra light densities, they are mechanically strong and can support a counterweight up to 900 times its own weight. Some CNT or graphene aerogels are known to exhibit super elasticity,

which can be compressed multiple times and still recover most of the volume after the compression release. CNT Aerogels can typically have an electrical conductivity of $3.2 \times 10^{-2} \text{ S.cm}^{-1}$, which can be enhanced to 0.67 S.cm^{-1} by a high current pulse method while maintaining the internal structures. This property has the potential to further expand the application fields of aerogels materials including solar cells, sensors, electrodes and cold electron field emission [10].

Graphene based aerogels was first prepared by Wang in 2009. Thus was done by converting GO solution in to the graphene aerogel by method of ultrasonic-induced gelation followed by drying and thermal reduction. The graphene aerogels exhibit improved electrical conductivity around $1 \times 10^2 \text{ S.m}^{-1}$, which is more than two orders of magnitude compared to graphene assemblies with physical crosslinks. Graphene aerogels have a large surface area $\sim 584 \text{ m}^2.\text{g}^{-1}$ and a pore volume of approximately $2.96 \text{ cm}^3.\text{g}^{-1}$ thus being suitable candidates for energy storage and sensing applications. Graphene aerogels can be modified in to different macroscopic structures like 1D graphene aerogel fibers besides the 3D foam structures. Graphene aerogel fibers have high specific area and good electrical conductivity. They also possess great flexibility and can be folded, bent or stretched back to its original shape without any internal fracture as shown in figure 7.

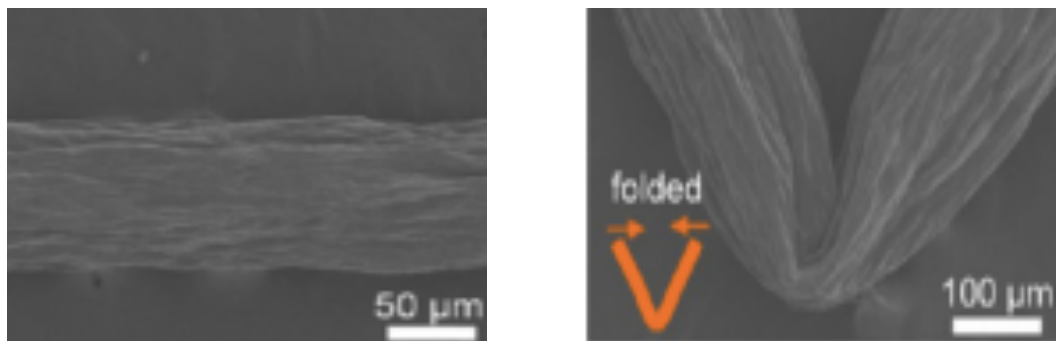


Figure 7: SEM images of graphene aerogel fibers, straight and folded up

Graphene and CNT's are two allotropes of carbon that has been investigated extensively in recent years. Both allotropes individually possess multifunctional properties, such as high electrical conductivity up to 10^4 S/cm, superior thermal conductivity of around 2000-5300 W/mK and a Young's Modulus of ~ 1 TPa.

Fabrication of Graphene-CNT Hybrid Aerogels

Graphene/CNT hybrid aerogels have been successfully developed for applications such as conductors, capacitive deionization and LIBs. The CNT's can prevent graphene nanosheets from stacking thereby enhancing the surface area of the gas. Chemical reduction method is the preferred technique for fabrication of CNT-graphene hybrid aerogels because of the high quality results obtained. The CNT's are dispersed in the GO aq. Suspension by sonication. LAA or hydrazine hydrate is used to reduce the mixture. A chemical bond between the graphene nanosheets and the CNTs is formed. Heat treatment removes the defects from graphene nanosheets and the CNTs. By simultaneous reduction, it is possible to chemically bond the two carbon allotropes [11].

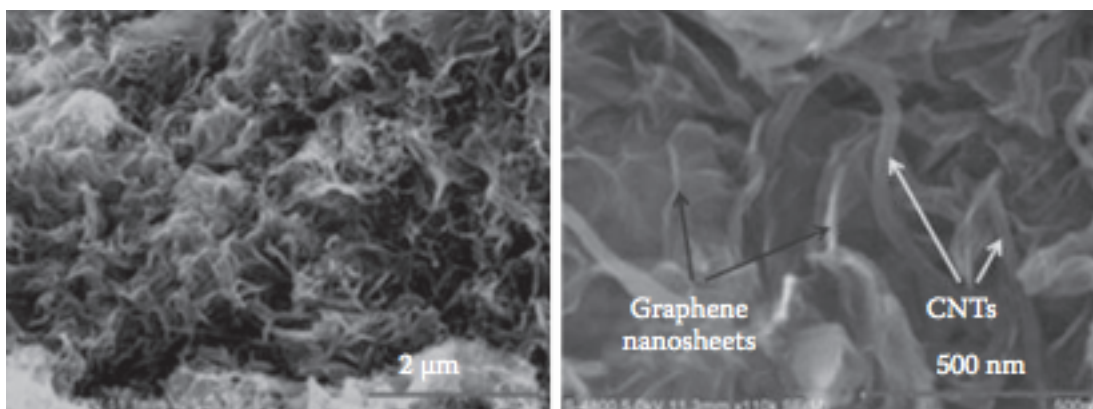


Figure 8: Network Structure of graphene-CNT hybrid aerogels

Other fabrication methods of CNT-graphene aerogels include directly freeze-drying the CNT-Go mixture, CVD method or the hydrothermal method.

Properties of Graphene/CNT Aerogels

Graphene and CNT aerogels exhibit various multifunctional properties like high electrical conductivity, low density, large surface area and good mechanical stability. The components required to fabricate graphene/CNT aerogel are now commercially available because of extensive research already done in this field. The novel properties that make aerogels an interesting area of research are described in next section [3].

Electrical Properties

Graphene aerogels generally report an electrical conductivity within a large range from 0.1-100 S/m. The electrical conductivity of graphene aerogels fabricated from the RF method can be measured by a four-probe technique. The values obtained by conducting measurements average up to 87 S/m. Graphene aerogels synthesized from the hydrothermal method apply the two-probe technique to measure electrical conductivity. The measurements generally return a value ~ 0.1 S/m. There is a large variation between the two methods due to different bonding formed during different fabrication conditions. Various drying and reducing agents also affect the electrical conductivity of graphene aerogels. The Supercritical Drying method yields a more conductive aerogel than that of the freeze-drying process. By using a more efficient reducing agent, the electrical conductivity of the aerogel can be enhanced [3].

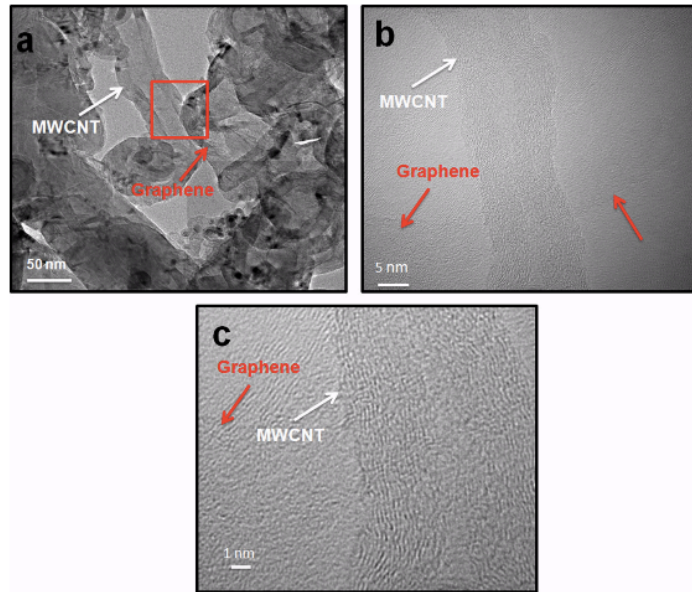


Figure 9: Interaction between graphene and MWCNT in aerogels

CNT aerogels can achieve a slightly higher electrical conductivity compared to graphene aerogels because of a more critical graphitic lattice. Different reactants during processing cause this difference between the two kinds of aerogels. DWCNT aerogels can achieve an electrical conductivity of up to 8 S/cm by the four probe measuring technique [5].

Mechanical Properties

Graphene/CNT aerogels are made up by individual nanosheets so mechanical properties like strength and Young's Modulus are far less than graphene and CNT monolayers. Therefore, graphene/CNT aerogels are generally considered brittle. A study conducted by Zhang revealed that graphene aerogels developed from supercritical drying was capable of supporting more than 14,000 times their own weight without much deformation. He also concluded that graphene aerogels processed by freeze-drying methods could only support 3,300 times its own weight.

Thus, supercritical drying achieves four times more strength than the freeze-drying method. CNT aerogels can support up to 1000 times its own weight after thermal treatment.

Most experiments conducted to test mechanical properties for graphene/CNT aerogels are compressive in nature due to the simple experimental setup involved and the brittle nature of aerogels. The compressive stress-strain curve for graphene/CNT aerogels can be divided in to three regions: elastic, yield and densification. The elastic region generally has 10% compressive strains, the yield region experiences ~60% compressive strains and the densification region experiences strains beyond 60%. From compressive tests, the Young's Modulus for graphene aerogels varies from 0.26 to 20 MPa, while those of CNT aerogels were between 0.12-65 MPa [10].

Thermal Properties of Graphene/CNT Aerogels

The mesoporous structure of graphene/CNT aerogels may reduce the internal thermal resistance and enhance the thermal efficiency by materials. However, thermal measurements and thermal transport mechanisms of graphene/CNT aerogels is an area of limited research. Under the right conditions, the thermal conductivity of graphene aerogels at 11% vol. of graphene and a low surface area of 43 m²/g was 2.18 W/mK. To measure the thermal conductivity of graphene aerogels, infrared microscopy techniques can be used. The measurements can be obtained using 1D steady-state heat conduction equation based on Fourier's Law.

$$\textit{Fourier's Law of heat conduction } Q_{cond} = -kA \frac{dT}{dx} (\textit{Watt})$$

The thermal conductivity of graphene aerogels can be between 0.12-0.36 W/mK. This is much lower than pure graphene because of the low quality and small size of chemical derived graphene nanosheets. Heat conduction through different forms of an aerogel scales as the density and

sound velocity. Figure 8 below shows that high-density silica will have a high heat transfer value. The low-density silica will have a low heat transfer. By adding dopants to the aerogel, a longer path is created thereby influencing a lower sound velocity and heat transfer.

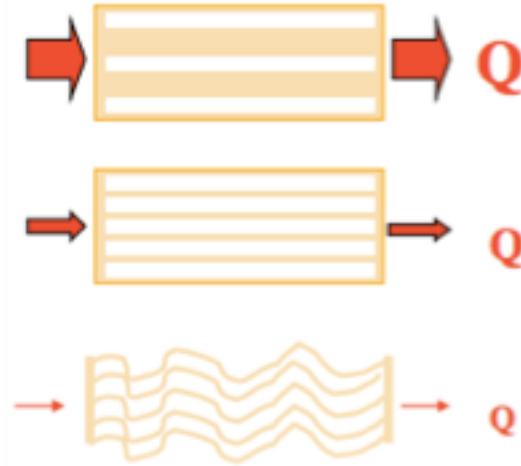


Figure 10: Heat Conduction Scaling in aerogels

There is no heat transfer in aerogels through convection. The pores in the aerogels are too small to support convection cells. Also, at low pressures like 10 kPa, there may or may not be a gas molecule within a given pore. Insulations made from aerogels experience no convection at the walls [7].

Silica aerogels are transparent or translucent to visible light unless a dopant material like graphene is added. The aerogels block most of the IR allowing its use as a Greenhouse covers. However, IR around $4\mu\text{m}$ passes through the pores reducing effectiveness of high temperature insulation. By loading the aerogel with dopants, the IR windows can be closed. This can drastically affect the heat insulation of the aerogel. Figure 11 below shows the transmission % graph in silica aerogels vs carbon loaded aerogels.

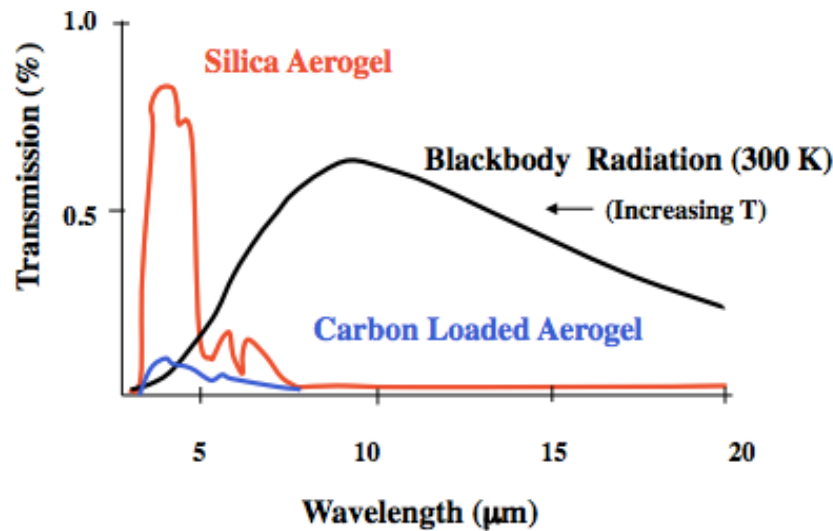


Figure 11: Transmission % of IR in aerogels through radiation

Specific Surface Area of Graphene/CNT Aerogels

The Specific Surface Area (SSA) of the graphene/CNT aerogel can be determined using the methylene blue (MB) adsorption method by UV-Vis spectroscopy. The test is conducted by adding a known mass of graphene/CNT aerogel in to a known volume of methylene blue (MB) solution of standard concentration. The process is followed by 2 hours sonication and stirring for 24 hours. This is done to reach the adsorption-desorption equilibrium of the methylene blue. By centrifuging the mixture, the suspended particles are removed. The MB concentration is measured through spectroscopy at a wavelength of 665nm compared with the initial concentration. The SSA of the aerogel can be calculated using the following equation.

$$\text{Specific Surface Area of the Aerogel SSA} = \frac{N_A A_{MB}}{M_{MB}} \frac{(C_0 - C_e)V}{m_s}$$

Where N_A represents Avogadro number ($6.02 \times 10^{23}/\text{mol}$), A_{MB} is the covered area per MB molecule typically assumed to be 1.35nm^2 , V is the volume of MB solution, M_{MB} is the relative

molecular mass of MB, m_s is the mass of the sample and C_0 and C_e are the initial and equilibrium concentration of MB.

Table 1: Standard Properties of Aerogels

	Aerogel¹	Silica²	Air
Dielectric Constant	1.007-2	----	1.0
Density (kg/m ³)	3-400	2200	1
Surface Area (m ² /g)	600-1500	----	----
Thermal Conductivity (W/mK)	0.01-0.3	1.3	1
Refractive Index	1.0-1.4	1.4	1.0
¹ Silica Aerogel			
² Sintered Silica (Polycrystalline)			

Reusability Test and Adsorption Capacity of Graphene/CNT Aerogels

The ASTM F726-99: Standard Test Method for Sorbent Performance of Adsorbents can be used to measure the adsorption capacity of aerogels. This test uses different kinds of oils such as gasoline, organic solvents and fats. The weight of graphene/CNT adsorbents is recorded after immersing it in the oil for 1 hour and compared with the initial weight of the aerogel. The adsorption capacity (Q) can be calculated using the weight of the aerogel before and after the adsorption test by:

$$\text{Adsorption of Aerogels } Q = \frac{wt_{final} - wt_{initial}}{wt_{initial}}$$

The most efficient results are obtained when the experiment is conducted three times and an average value of adsorption capacity can be calculated.

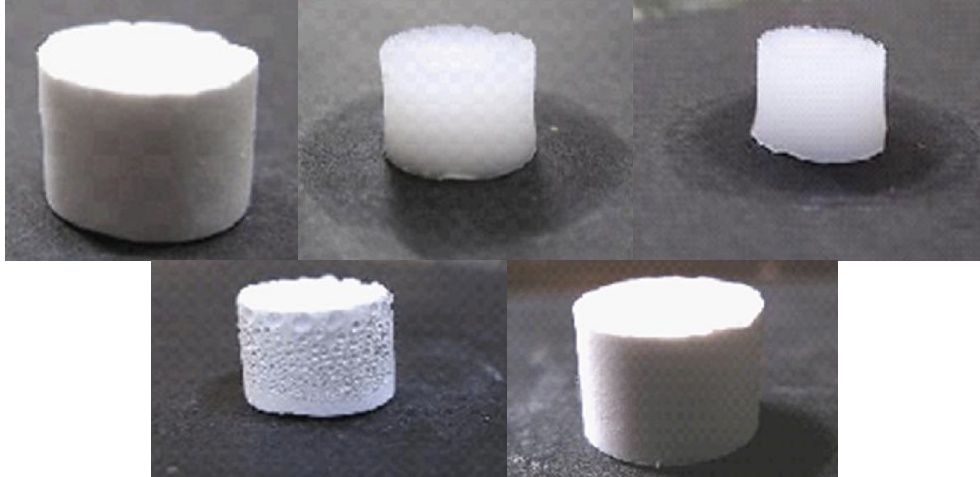


Figure 12: Various stages of absorption and desorption of organic liquids in aerogels

Process Variables

Graphene hydrogels are formed by the pi-pi stacking during reduction. The reducing agent (LAA, HI or NaHSO₃) impacts the electrical properties and thermal stability of graphene aerogels. CNT-graphene aerogels formation can also take place due to pi-pi bond interaction between the graphene nanosheets as well as between the graphene and CNTs. During the reduction process, majority of the CNTs wrap inside the 2D network of graphene hydrogel, while the remaining CNTs forms pi-pi interaction with the reduced graphene oxide sheet [5].

Effect of Reducing Agents and CNTs on GAs

Graphene Aerogels are characterized by nitrogen adsorption/desorption measurements to get values like specific surface area and pore size. This can be obtained by the **Brunauer-Emmett-**

Teller method. The surface area of graphene aerogels reduced by LAA, HI and NaHSO₃ give different values. By implementing the process of annealing, the functional groups can be further removed causing the aerogels to condense further. There is no clear explanation for the difference in values due to different reducing agents. It is possible that by annealing, the pi-pi interaction among graphene sheets may decrease [5].

Effect of CNTs on GAs

Different concentrations of CNTs can be added to the Gas to form different CNT-graphene hybrid aerogels. The surface area of CNT-graphene aerogels increase due to a higher concentration of CNTs up to 1 mg/mL . This is due to the additional surface area of the dispersed CNTs and the separation of graphene nanosheets by the CNTs during the formation processes. However, higher CNT concentrations may reduce the surface area of the aerogel as it may block the pores by bundling [5].

Aerogel Use in Reduction of Casimir Force

Aerogels are highly porous materials typically fabricated from SiO₂ using sol-gel techniques. Due to properties like low thermal conductivity, good mechanical strength and luminescence, low density, and porosity as high as 95%, aerogels are fast becoming a promising material in the semiconductor industry. Aerogels have the lowest refraction index of all solids in the visible range $n \sim 1.05$ depending on the porosity. Air voids in the aerogels dominate the dielectric behavior rather than the solid part. The Casimir effect is a small attractive force that acts between two parallel-uncharged conducting plates due to quantum vacuum fluctuations of the electromagnetic field. Aerogels can be used to reduce the magnitude of the Casimir force on a

nanoscale. In order to calculate the magnitude of Casimir Force, we will need to analyze the dielectric response of the aerogel in a broad frequency range. Methods such as **Clausis-Mossotti** approximation and **Looyenga’s** approach can be used to study the effective optical properties of high and low porous media respectively [11].

$$\text{Clausis – Mossotti for dielectric permittivity } \frac{\epsilon - 1}{4\pi} E = \sum_k N_k \alpha_k (E_i)_k$$

Hybrid Aerogel Applications

Hybrid Aerogels have some unique characteristics that make them attractive to use in science and technology. Some general applications of Hybrid Aerogels perspective to properties are listed below in Table 2.

Table 2: Potential applications of hybrid aerogels

Property	Feature	Application
Acoustic	1. Low speed of sound	1. Sound proof rooms 2. Ultrasonic distant sensors
Optical	1. Transparent 2. Multiple composition 3. Low refractive index	1. Light weight optics 2. Light guides 3. Cherenkov detectors
Mechanical	1. Elastic 2. Light Weight	1. Energy absorber 2. Velocity particle trap
Thermal conductivity	1. Good insulating solid 2. High Temp. Threshold	1. Construction and insulation 2. Automobiles, space vehicles
Density	1. Lightest synthetic solid 2. High Surface Area	1. Catalysis 2. Sensors 3. Fuel Storage 4. Filters for gaseous pollutants
Electrical	1. Low dielectric constant 2. High dielectric strength	1. Dielectrics for Integrated Circuits 2. Capacitors

Aerogels as an Absorbent

Synthesis of flexible and super hydrophobic aerogels can be used as an absorbent. The absorption and desorption capacity of doped silica aerogels can be investigated using eleven solvent and three oils. A. Venkateshwara Rao introduced this method and can be applied to our methodology [4]. The mass of a liquid that rises into the aerogel pores is given by the following formula.

$$**Mass of liquid in aerogel pores: $2\pi r\gamma\cos\theta = mg$**$$

When liquids are involved that completely wet the surface, the contact angle θ is zero and for such surfaces, the mass is represented by:

$$**Mass of liquid in aerogel pores with zero angle: $2\pi r\gamma = mg$**$$

or

$$\frac{\gamma}{\rho} = kV$$

Where r is the radius of aerogel pores, V is the volume of the absorbed liquid, ρ is the liquid density and $k=g/2\pi r$ is a constant. As the surface tension of the liquid increases, the mass of the liquid absorbed also increases linearly. Super hydrophobic aerogels can be efficient absorbents of oils and organic liquids [4].

Aerogels as a Sensor

Aerogels have a high porosity and surface area overall that make them potential candidates for use as a sensor. Silica aerogels doped with nanoparticles exhibits reduced electrical resistivity with increasing humidity. Hybrid Aerogels that have modified surfaces are less affected by humidity as compared to hydrophilic aerogels. They can be used as anticorrosive, hydrophobic agents.

Aerogels as Materials with Low Dielectric Constant

SiO₂ aerogel films have found great interest in Integrated Circuit applications because of properties such as low dielectric constant, high porosity and high thermal stability. Earlier investigations in this field conducted by Park revealed that the dielectric constant for silica thin films of silica aerogels was approximately 1.9. By using ambient drying process using n-heptane as a drying solvent, the SiO₂ aerogel films can achieve a low dielectric constant of 2.0 with 79.5% porosity [11].

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