PMI
POROSITY & PERMEABILITY TESTING

Porous Materials and Their Structure

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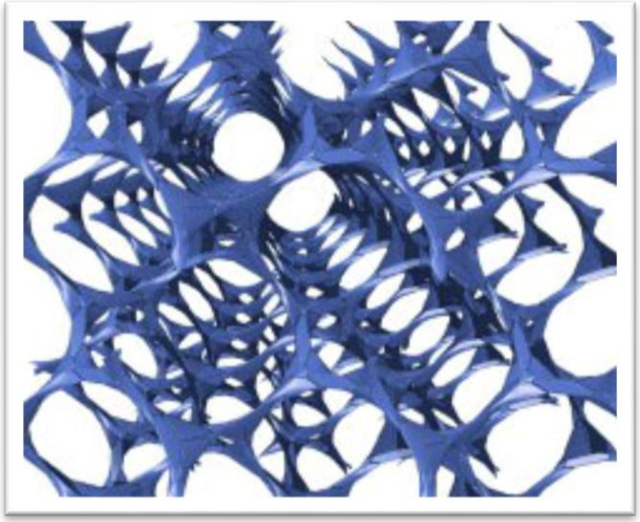
Porous Materials, Inc.

Porous Materials And Their Structure



Introduction

Pore Structure

- Porosity
- Pore Cross-section
- Pore Shape
- Pore Size
- Pore Size Distribution
- Pore Path
- Pore Surface Area

Characteristics of Pore Structure

Characterization Techniques

Summary and Conclusion

Applications of Porous Materials in the Industry

Industry	Applications	Role of porous structure
BIOTECH 	• Implants • Body parts • Filters for body fluid • Barriers for bacteria & viruses • Barrier for pollens • Storage of body parts • Tissue growth • Artificial skin	• Removal of unwanted materials • Blood vessel growth • Separation of constituents of blood • Selective permeation of gases and nutrients • Prevention of passage of bacteria, viruses, pollens etc.

Applications of Porous Materials in the Industry

Industry	Applications	Role of porous structure
PHARMACEUTICAL 	• Pharmaceutical powders • Drugs • Bandages • Barriers for bacteria • Dressings • Packaging material • Drug delivery pouches	• High surface area for fast dissolution • Vapor and oxygen permeation • Controlled liquid permeation • Removal of bacteria
FOOD 	• Filters for purification & separation • Material for packaging & storage • Powdered food	• Removal of bacteria • Separation of constituents • Prevention of O_2 permeation • Absorption

Applications of Porous Materials in the Industry

Industry	Applications	Role of porous structure
ENVIRONMENTAL 	• Filters for water purification • Filter for toxicity reduction • Filters for desalination • Air filters • Gas masks	• Removal of solids, ions, & bacteria • Selective gas permeation • Adsorption
HYGIENE, GARMENT, & HOUSEHOLD 	• Barriers to mite • Breathable fabric • Diapers • Hygienic napkins • Adsorption of gas • Facial tissue • Cosmetic powders • Paper towels • Bed sheets	• Moisture transmission • Gas transmission • Prevention of mite migration • Retention of liquid • Odor removal

Applications of Porous Materials in the Industry

Industry	Applications	Role of porous structure
FILTRATION 	• Felts • Wire mesh filters • Textiles filters • Perforated material	• Removal of solids from liquids and gases • Removal of liquids
POWER SOURCE 	• Separators • Electrodes • Chemical powders	• Surface area for reactions • Selective ion permeability
CONSTRUCTION 	• Geotextiles • Barriers for water seepage and soil erosion • Powders in paints • Water storage • Cement	• Keeping out solids • Controlled liquid permeation • Moisture transmission • Prevention of water transmission

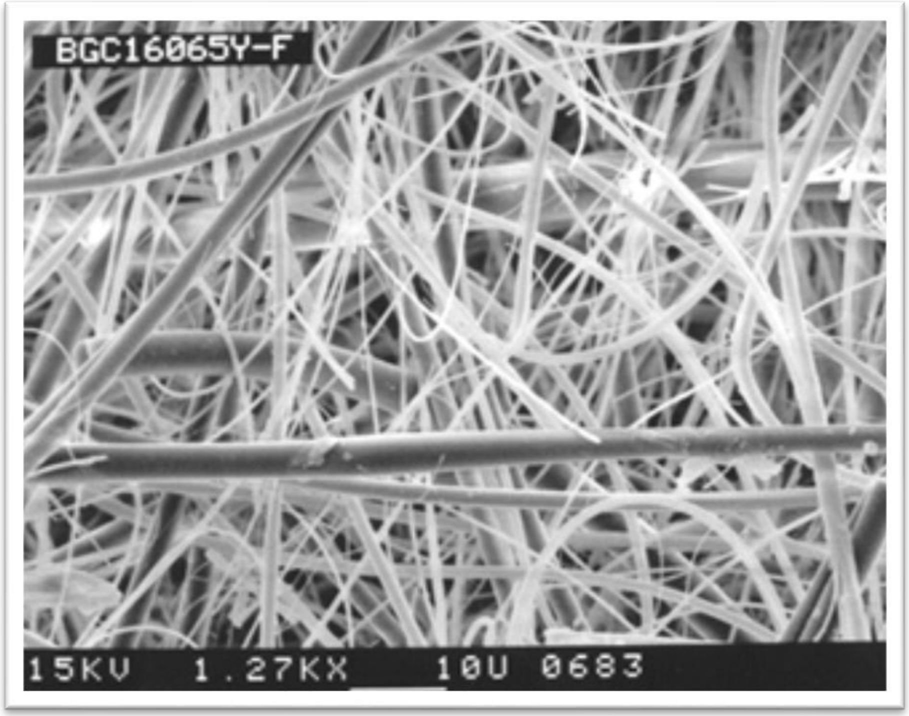
Applications of Porous Materials in the Industry

Industry	Applications	Role of porous structure
PAPER/PRINTING 	• Coated paper • Packaging paper • Felt	• Controlled ink flow • Differential ink flow in x, y, or z directions • Vapor transmission
CHEMICAL 	• Zeolites • Ion exchange resins • Catalysts • Carbon black • Abrasives • Fertilizers • Metal powders • Ceramics	• High surface area • Controlled particle size • Controlled porosity

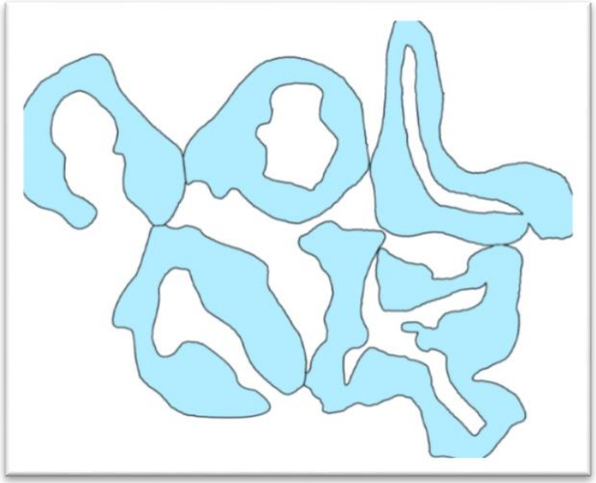
Various Kinds of Porous Materials

Fibrous material

Porosity due to voids between fibers

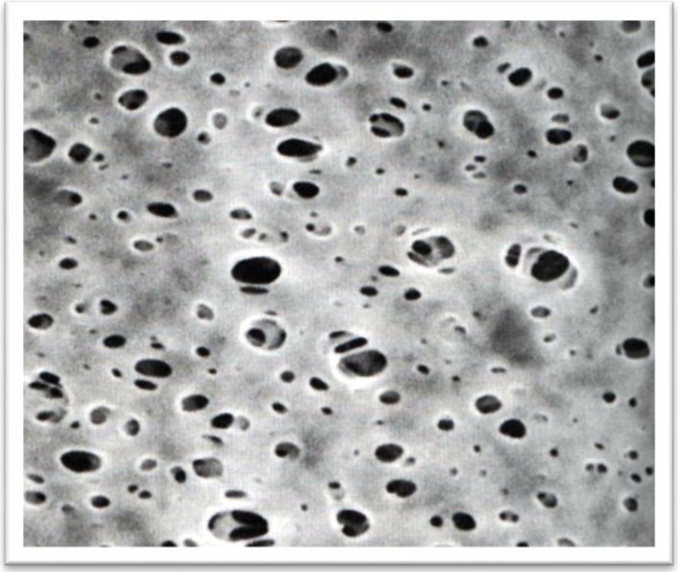


Various Kinds of Porous Materials



Powder bed

Porosity due to voids between particles and internal voids inside particles.



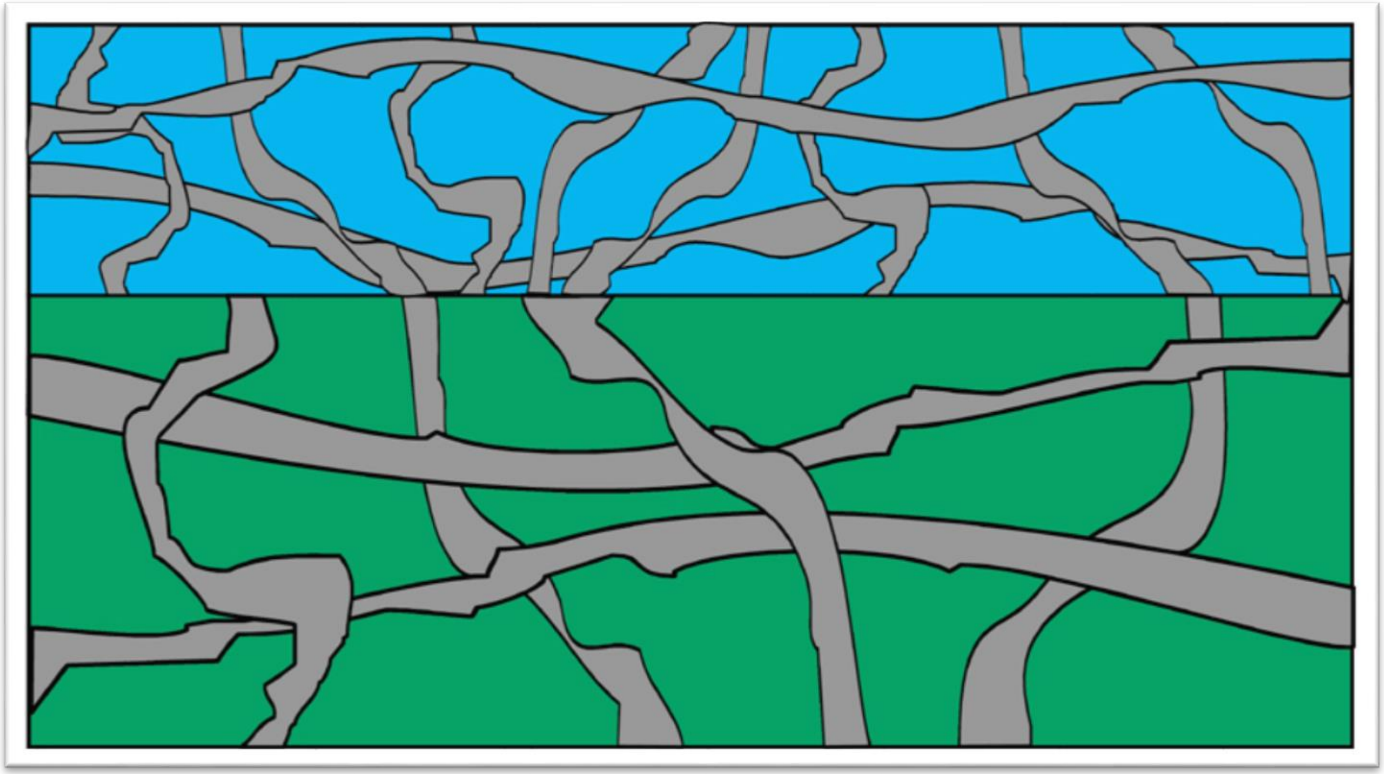
Consolidated material

Porosity in membrane created by voids left inside material

Various Kinds of Porous Materials

Coated porous material and laminated porous material

Structure of coated or laminated porous material.



Various Kinds of Porous Materials

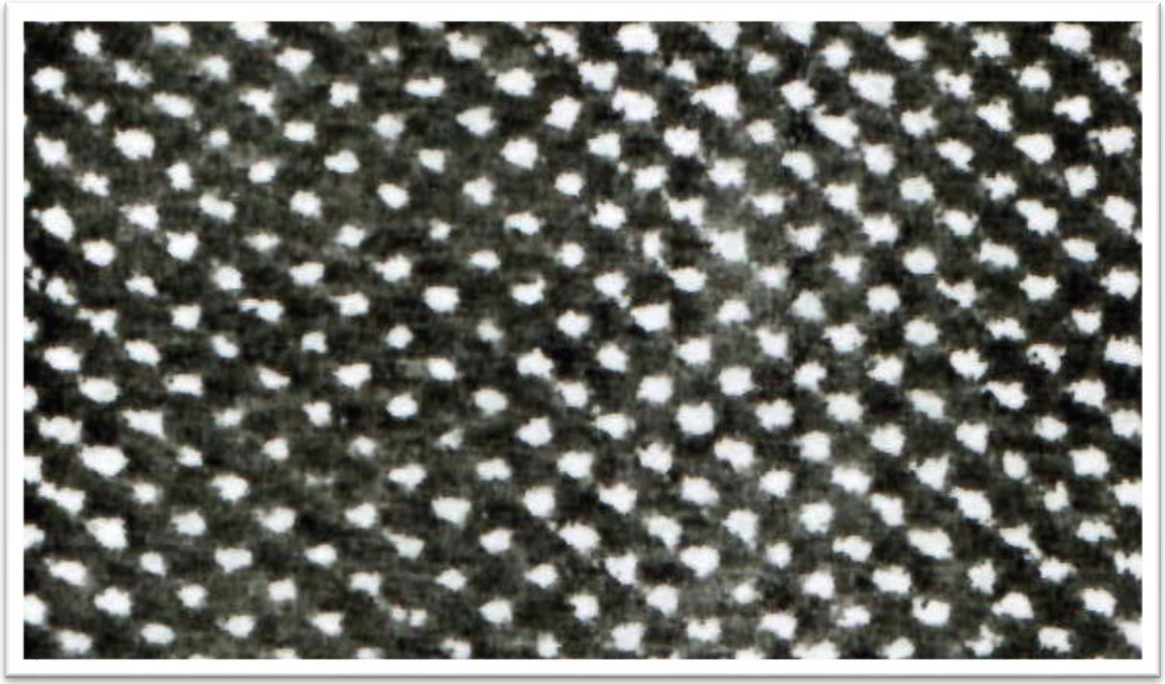
Structural classification of porous materials

Porous Materials

- Fibrous
- Particulate
- Consolidated
- Coated & Graded
- Laminated

Pore Structure

Porosity - Interstitial Void



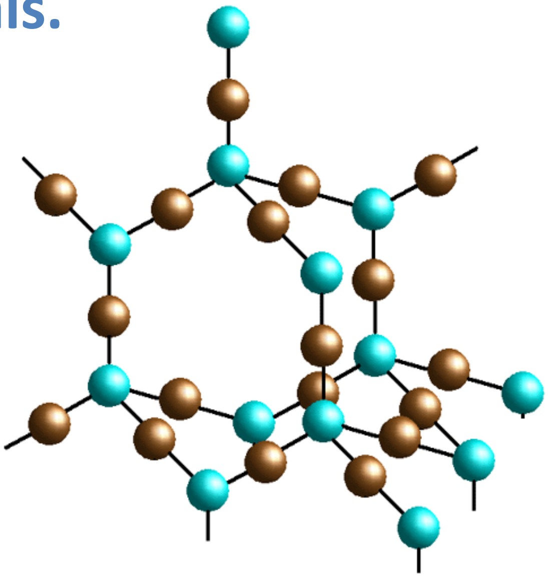
High-resolution electron micrograph of the [110] plane of crystalline silicon showing interstitial openings in the plane of atoms

Pore Structure

Porosity - Interstitial Void

SILICON: Interstitial space 66 % of its volume

Arises because atoms cannot be compacted closer together due to strong repulsive forces resulting from overlap of outer electron orbitals.

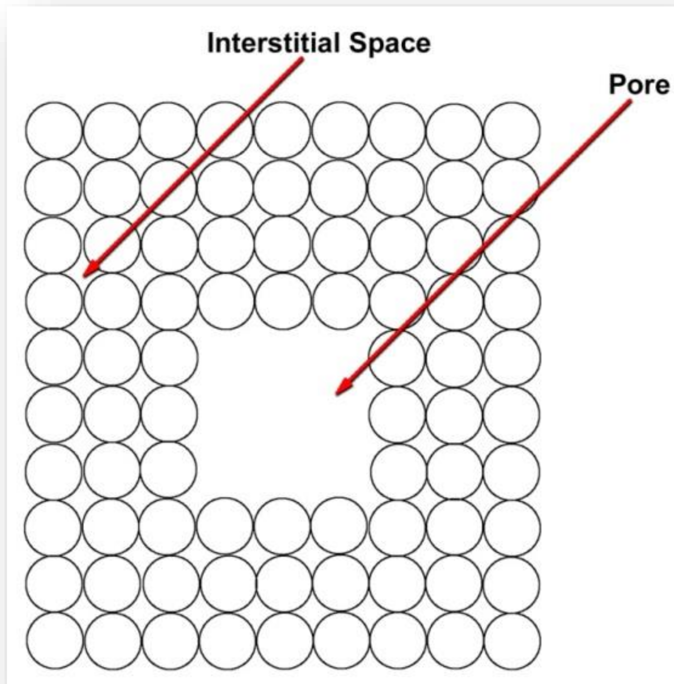


Pore Structure

Porosity

Pores

- Created by atoms missing from the neighboring lattice sites
- Porous materials contain pores and interstitial openings



Pore Structure

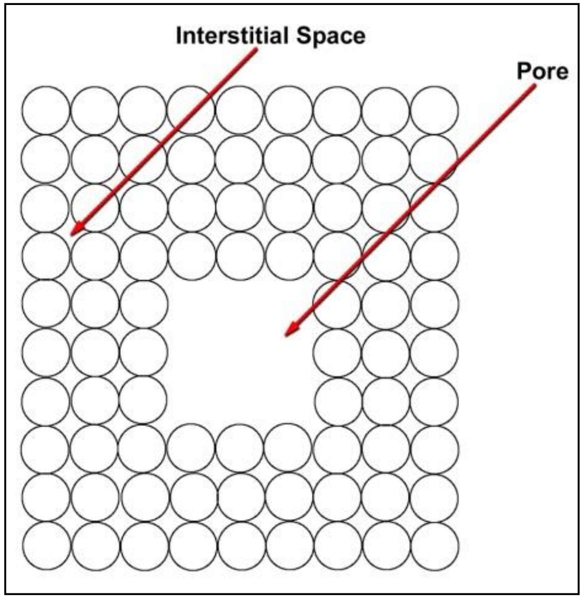
Porosity

Definition of Porosity

Interstitial void volume in the solid ignored.
Only pore volume considered

$$P = V_v / [V_v + V_s]$$

P = porosity
V_v = volume of pores
V_s = volume of solid in the material



⇦ Interstitial void Ignored

⇦ Pore Volume considered

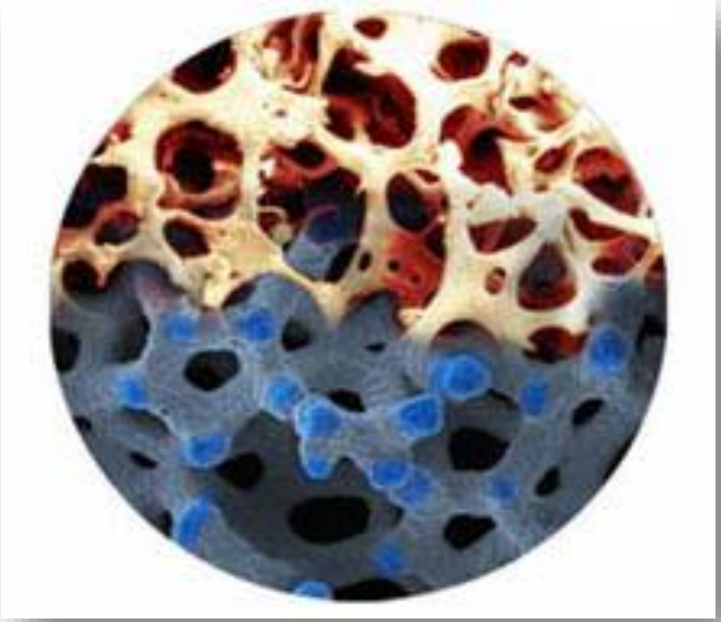
Pore Structure

Porosity

Wide Range of Porosity

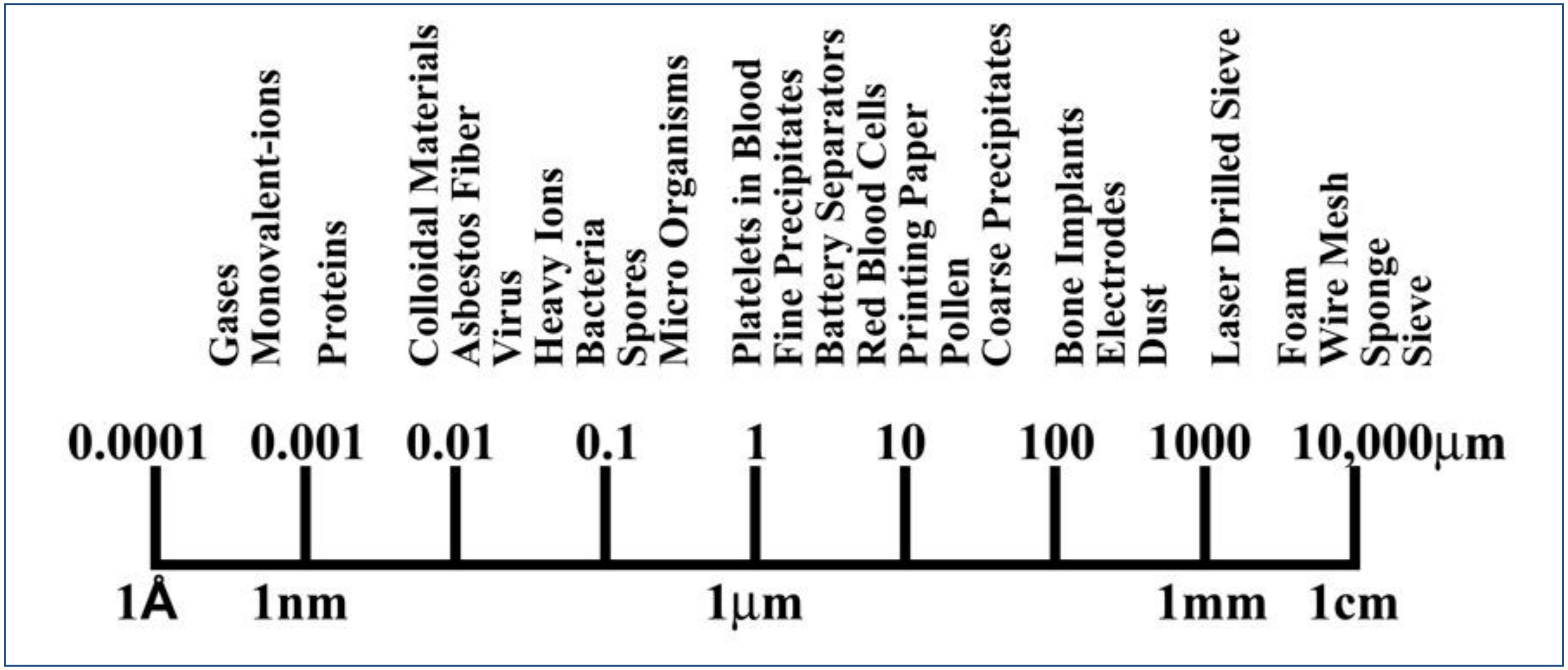
Almost zero to very large

Large 5 mm size pore in ceramic foam



Pore Structure

Porosity

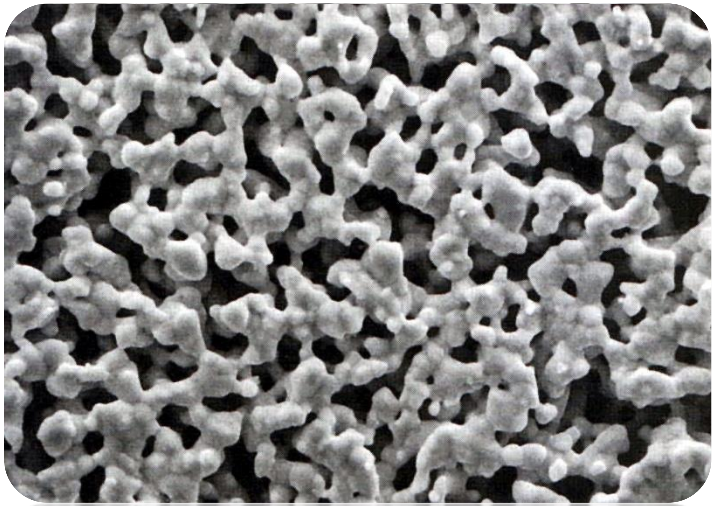


Typical void size ranges and particle size ranges in some materials of interest.

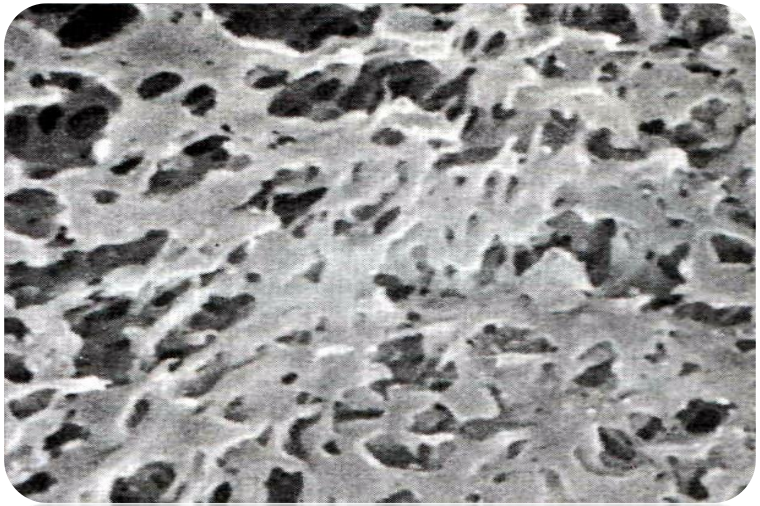
Pore Structure

Pore Cross-Section

Normally quite irregular



Sintered metal disc

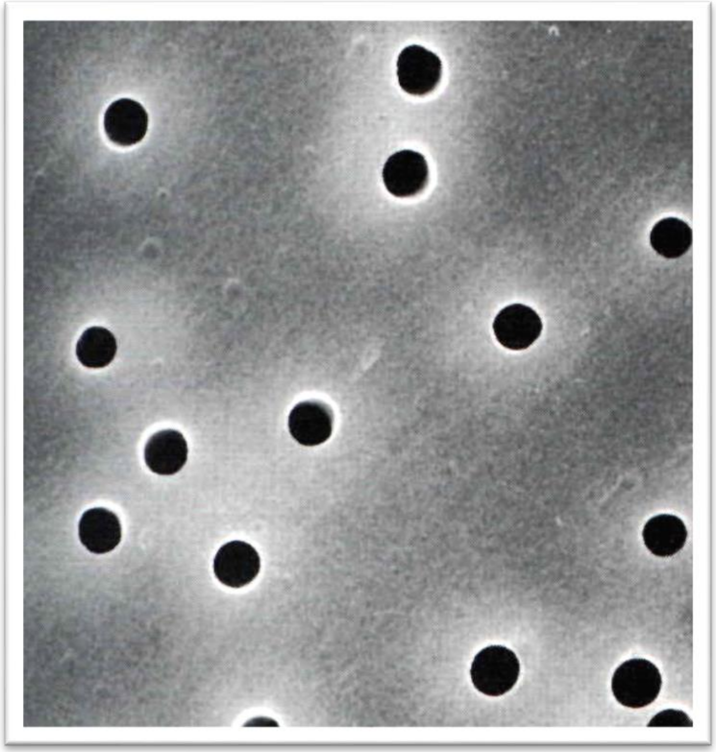


Membrane

Pore Structure

Pore Cross-Section

Circular in some unique materials

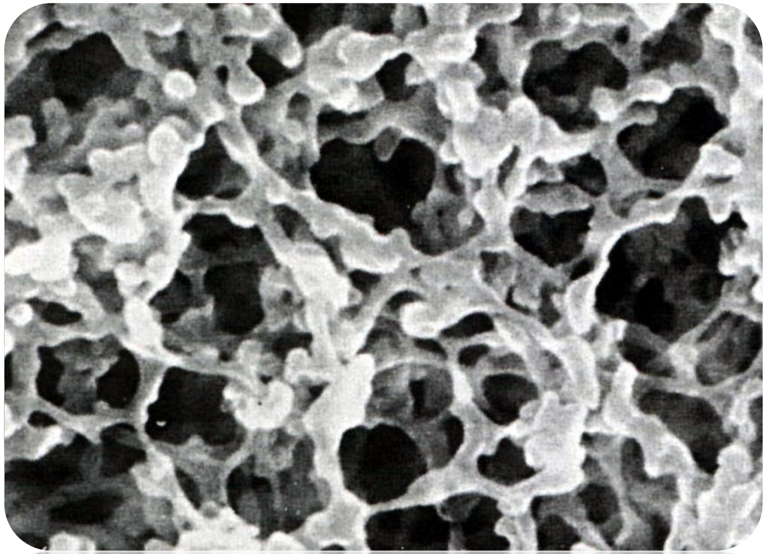


Track-etch membrane

Pore Structure

Pore Shape

Pores normally form highly interconnected and tortuous channels



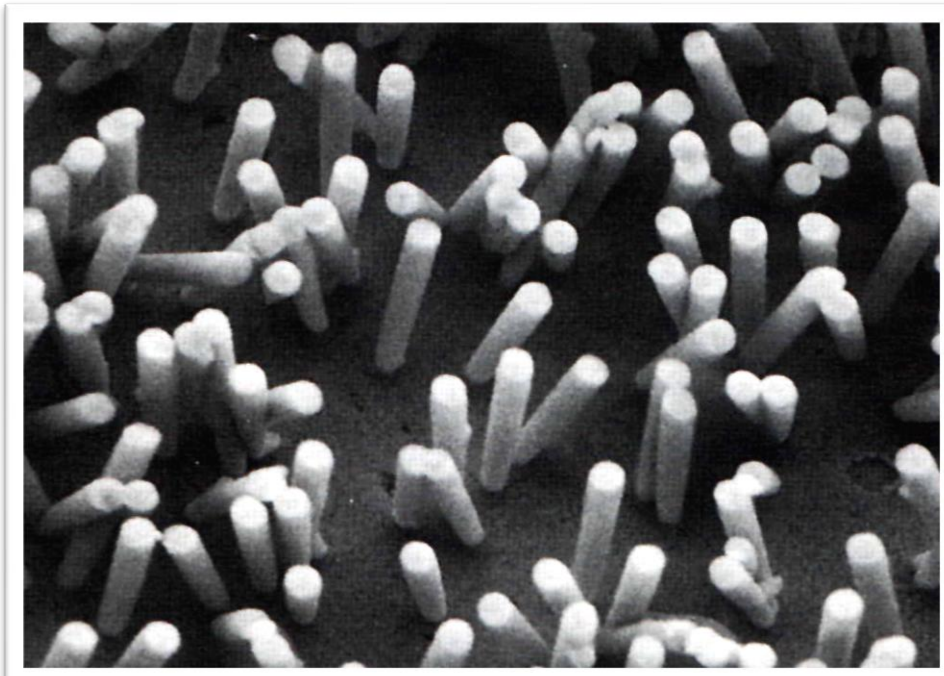
High depth of focus reveals irregular cross-section and variation with depth

Tortuous channels in cross-section of membrane

Pore Structure

Pore Shape

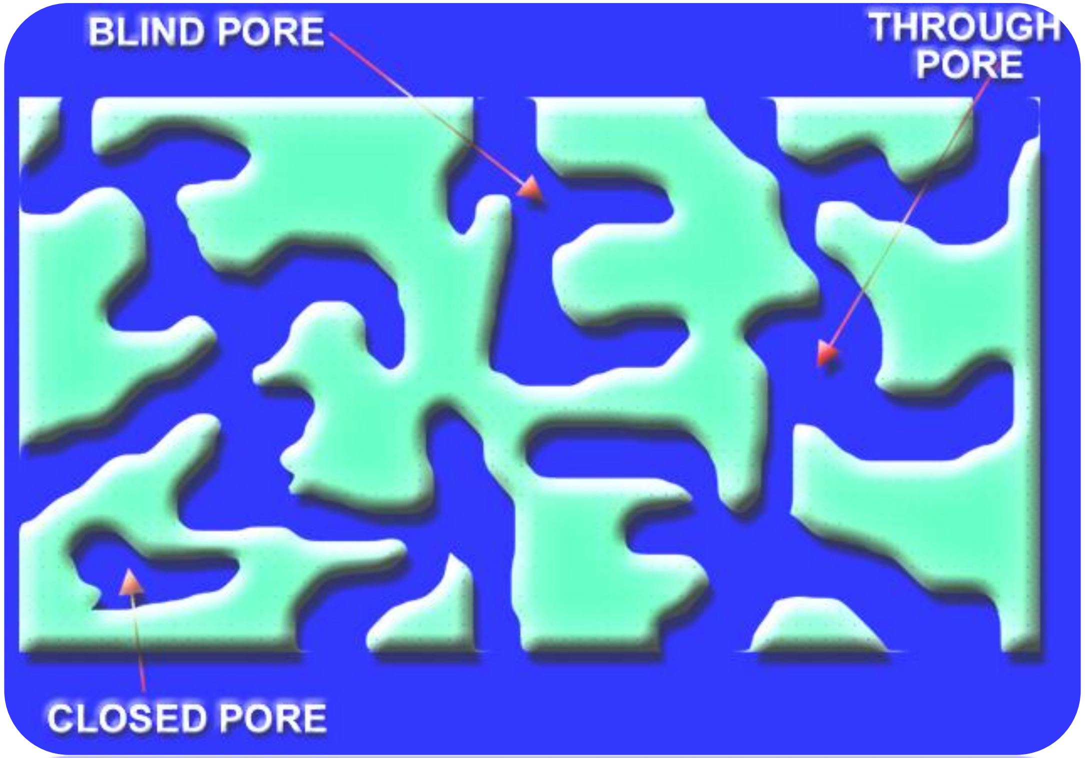
Cylindrical pores without much interconnectivity in membranes can be produced by special techniques



Structure revealed by filling pores with insoluble material and dissolving the matrix

Pore Structure

Pore Path

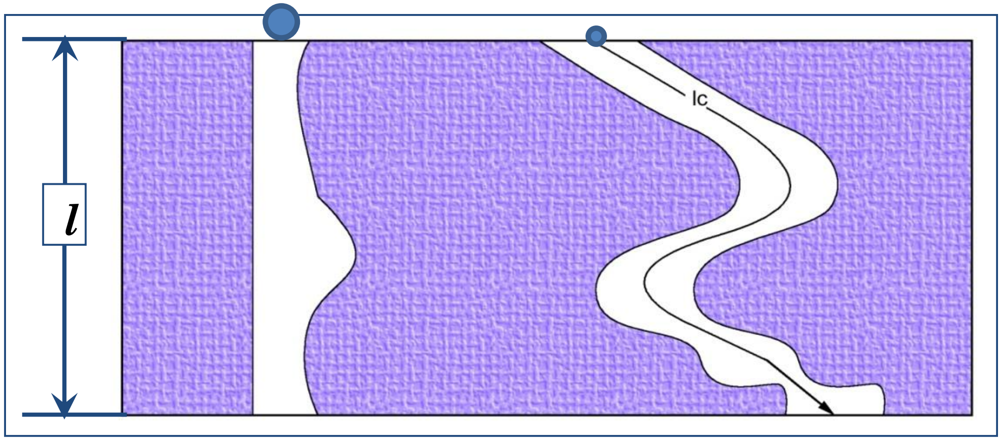


Pore Structure

Pore Path

$$\tau = (l_c / l)$$

τ = tortuosity
l = thickness of porous medium
l_c= length of pore path



(Flow Rate) $\propto$ $[(D^4 \Delta p) / l_c]$

Flow through tortuous path is less

Pore Structure

Pore Size

Varies along the course of the channel

Pore size at any location determined by cross-section at that location

Pores with irregular cross-sections: Pore size not defined

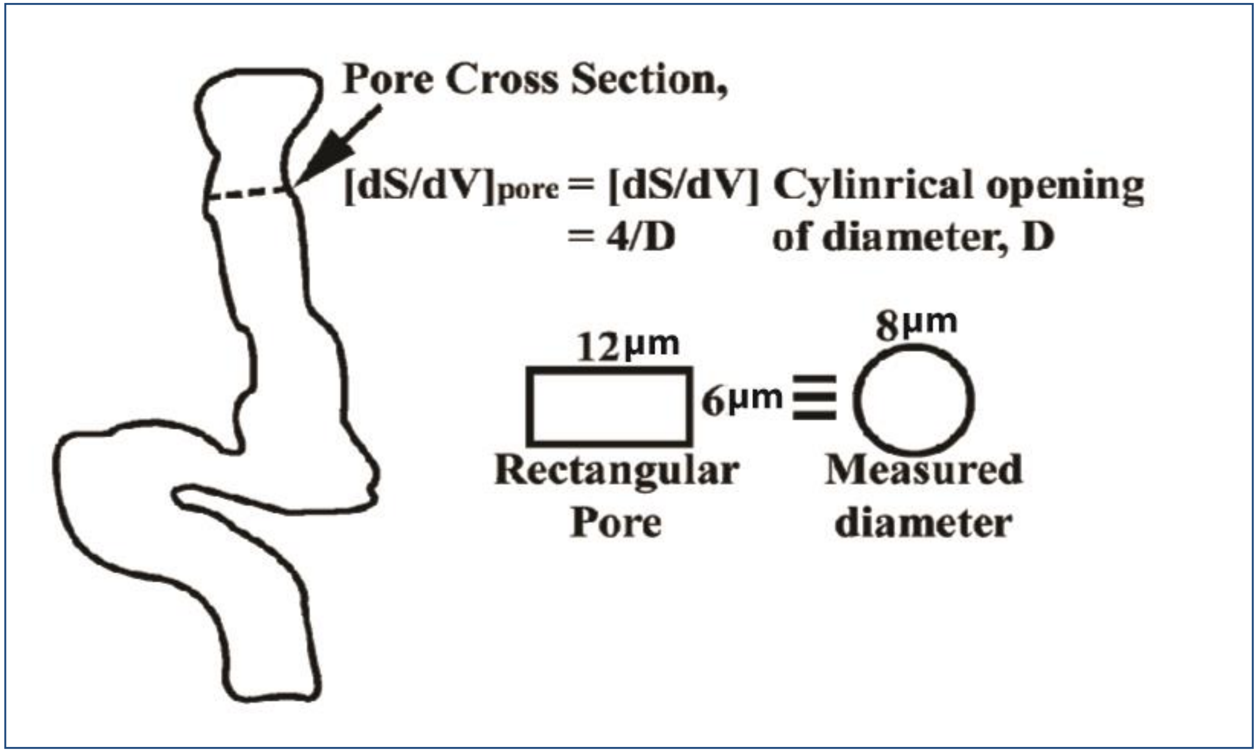
Pore Structure

Pore Size

In all measurement techniques **pore diameter (D)** at the desired location in the pore is defined as the **diameter of an equivalent cylindrical pore**

Pore Structure

Pore Size

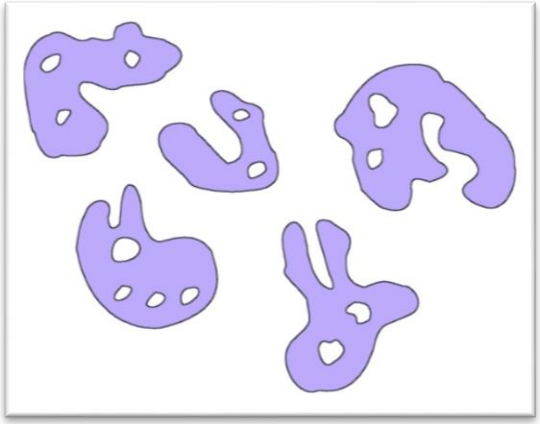


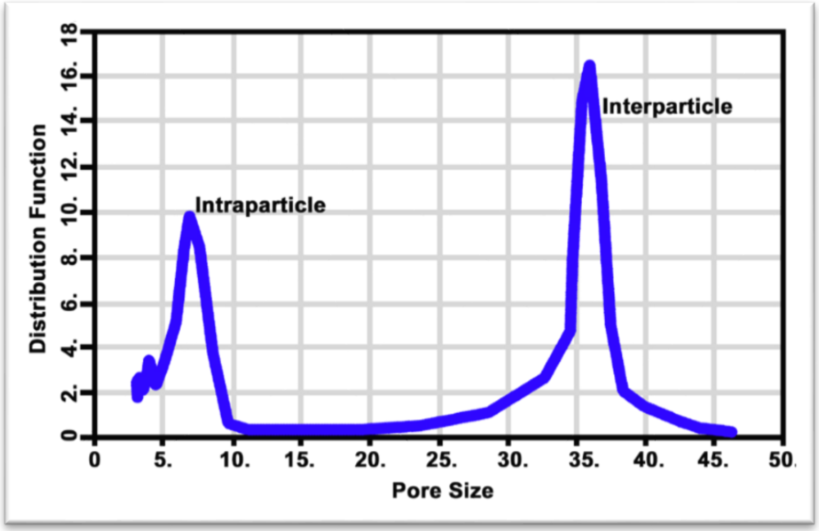
Definition of measured pore size

Pore Structure

Pore Size Distribution

May be very narrow
May be broad
May be multi-modal





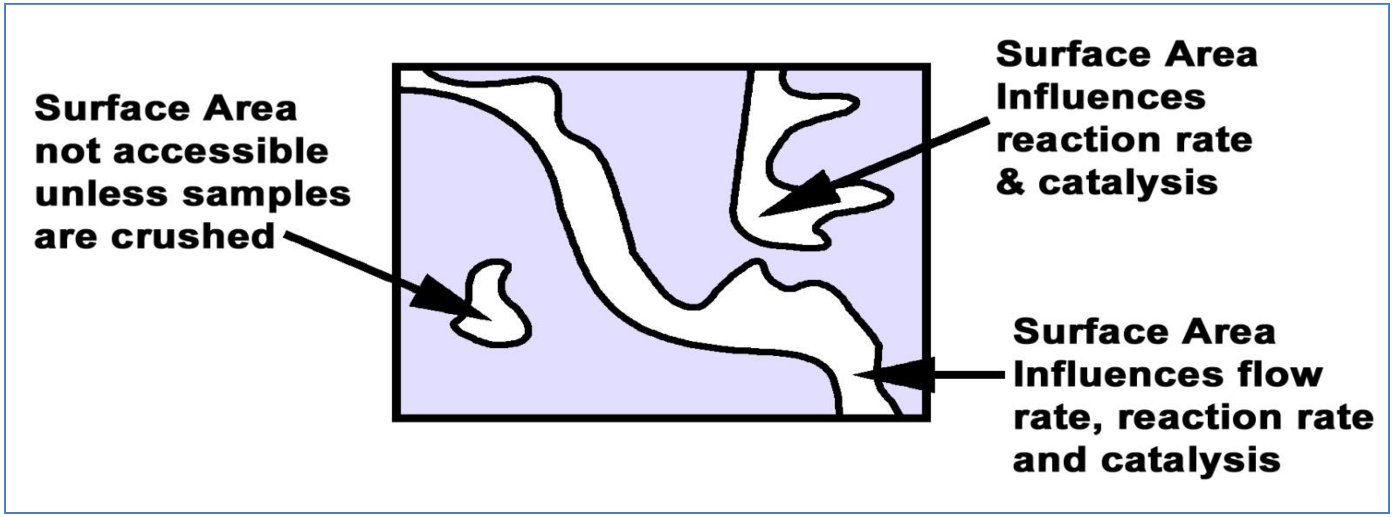
Bimodal distribution in a powder bed

Pore Structure

Pore Surface Area

Pore surface area is determined by the:

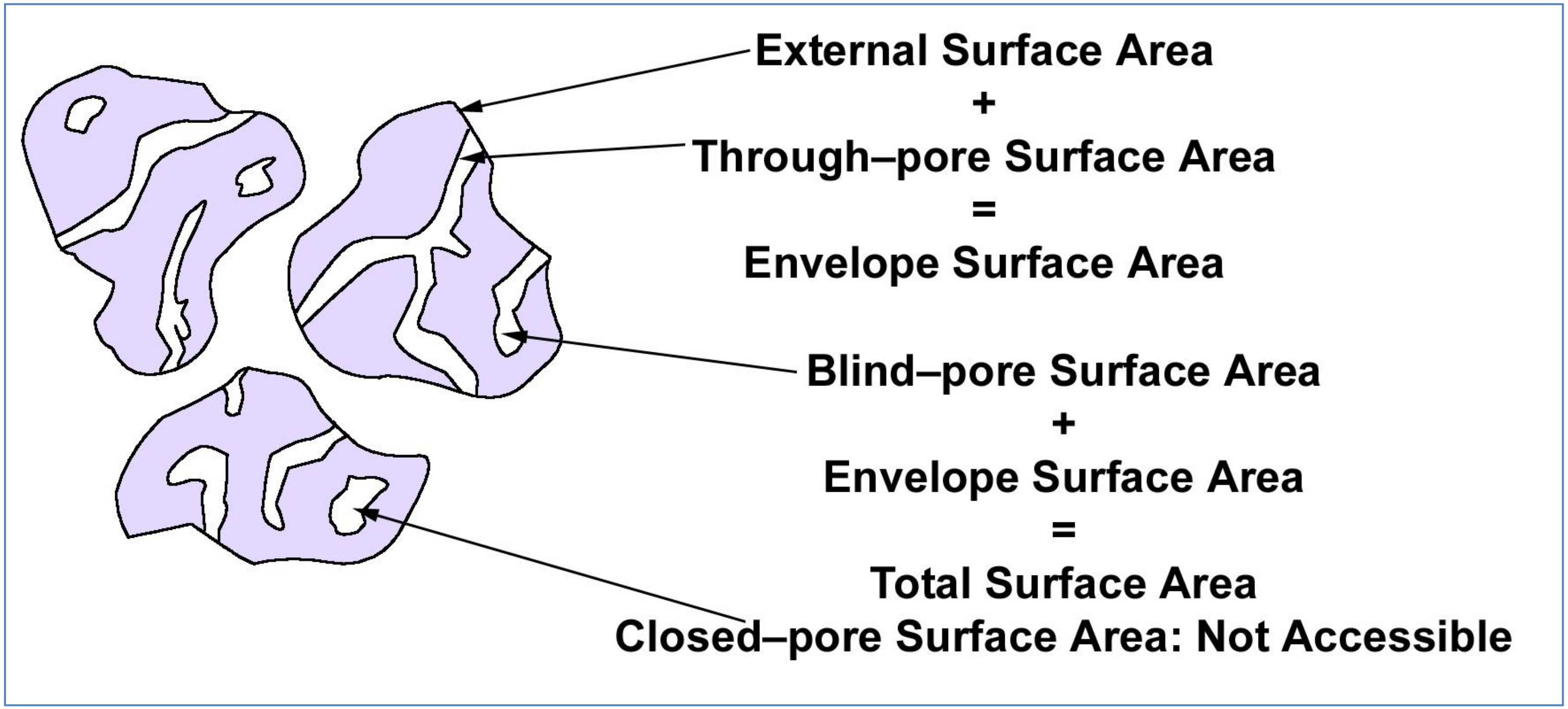
- Shape of pore
- Size of pore
- Roughness of pore surface



Role of the three different kinds of pores

Pore Structure

Pore Surface Area



Envelope surface area

Pore Structure

Pore Volume

The pore volume has the following components

Total pore volume

=

Volume of closed pores

+

Volume blind pores

+

Volume of through pores

Characteristics of Pore Structure

Largest through pore *throat* diameter (bubble point pore diam):	***Barrier properties***
Mean flow through pore throat diameter:	***Measure of performance***
Pore size distribution:	***Measure of efficiency***
Gas permeability***:***	***Gas flow rate, Rate of Process***
Liquid permeability:	***Flow rate of liquid chemicals***
Pore volume & pore volume distribution:	***Dirt holding capacity***
Diffusion permeability:	***Small flow rates or leak rates***
Envelope surface area:	***Filtration***
Total surface area:	***Reaction rates***

Characteristics of Pore Structure

Simulation of true service conditions

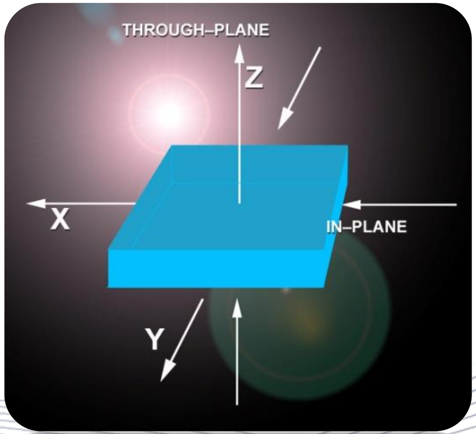
Effect of Compressive Stress

Effect of Cyclic Stress

Effect of Temperature

Effect of Chemical Environment (Use of different humidity and chemicals)

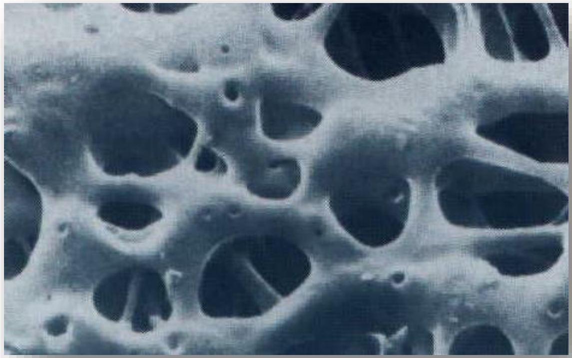
Effect of Orientation (Characteristics of in-plane pores and pores in the x & y directions)

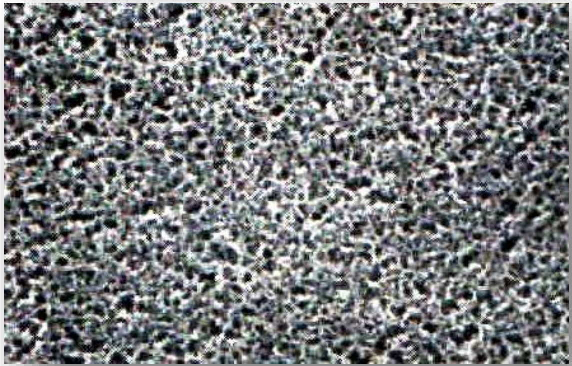


Characteristics of Pore Structure

Pore characteristics of graded, coated & laminated materials

Graded pore structure in membrane



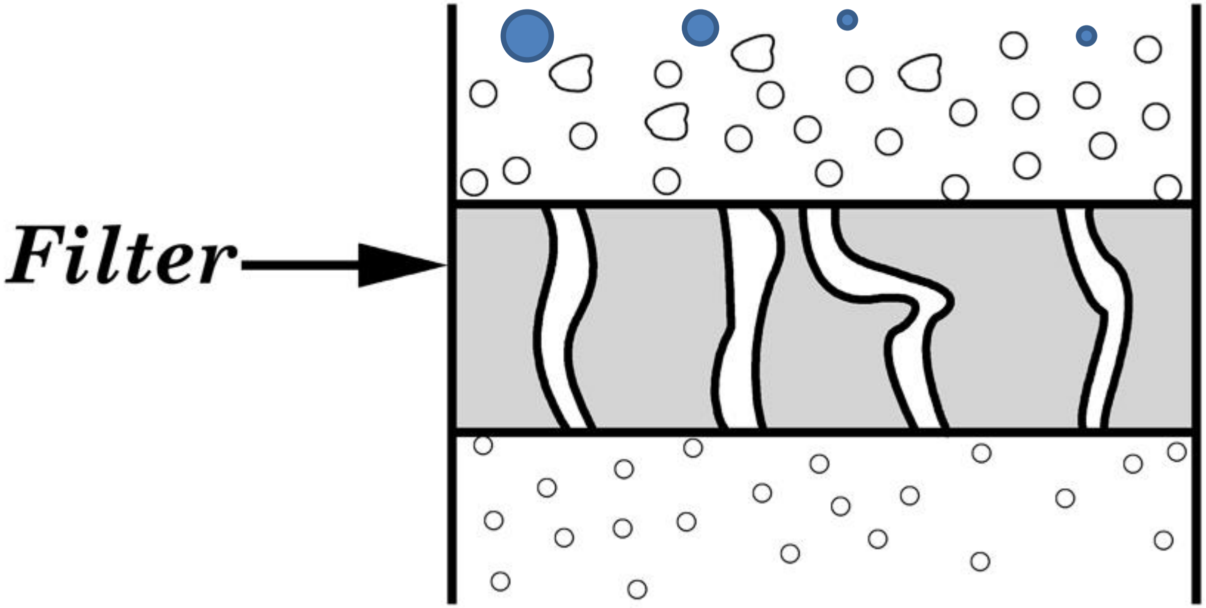


Characterization Techniques

Macroscopic Measurement Technique - For large pores

Microscopic Techniques - Very few pores are measurable

Particle Challenge Technique - Expensive & Time Consuming

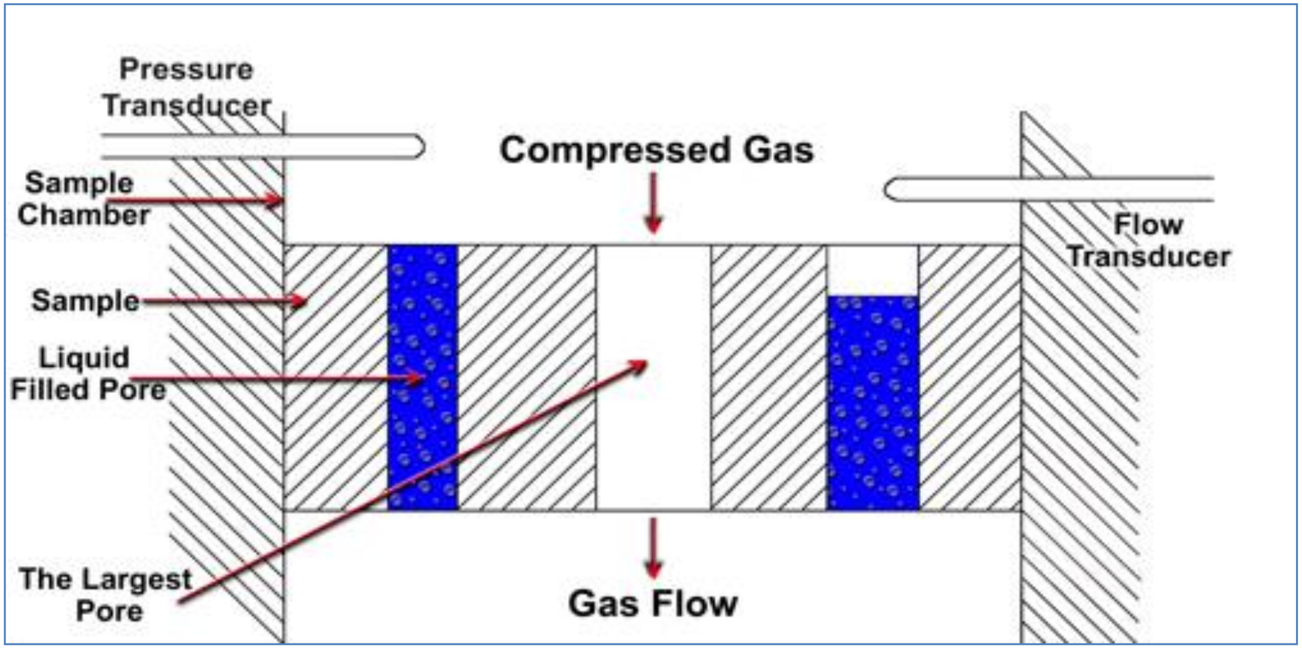


Particle challenge technique

Characterization Techniques

Capillary flow porometry

-Measures differential gas pressure and flow rates through dry samples and samples wetted by a wetting liquid
-Computes through pore throat diameters, distribution and gas permeability
-Widely used & highly versatile



Principle of Capillary Flow Porometry

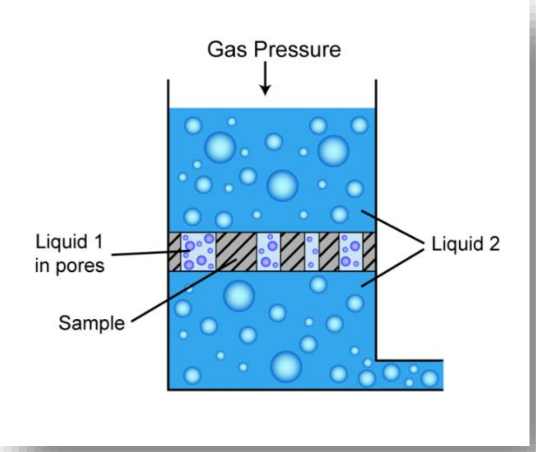
Characterization Techniques

Liquid - Liquid Porometry

One immiscible high surface tension wetting liquid displaces from pores another low surface tension wetting liquid

Pore structure computed from measured

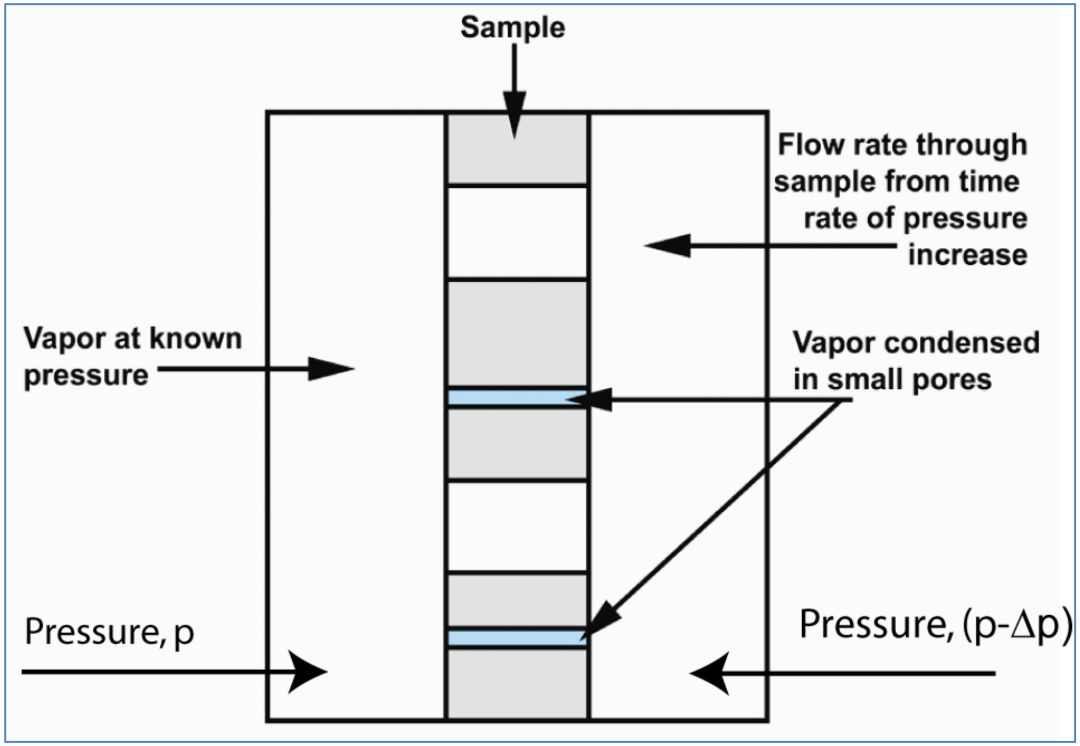
- **Differential pressure**
- **Flow rate of wetting liquid with higher surface tension**
- **Interfacial tension between the two liquids**



Characterization Techniques

Capillary Condensation Flow Porometry

- Measures flow through pores not blocked by condensed vapor
- Computes through pore diameter, distribution and flow rate

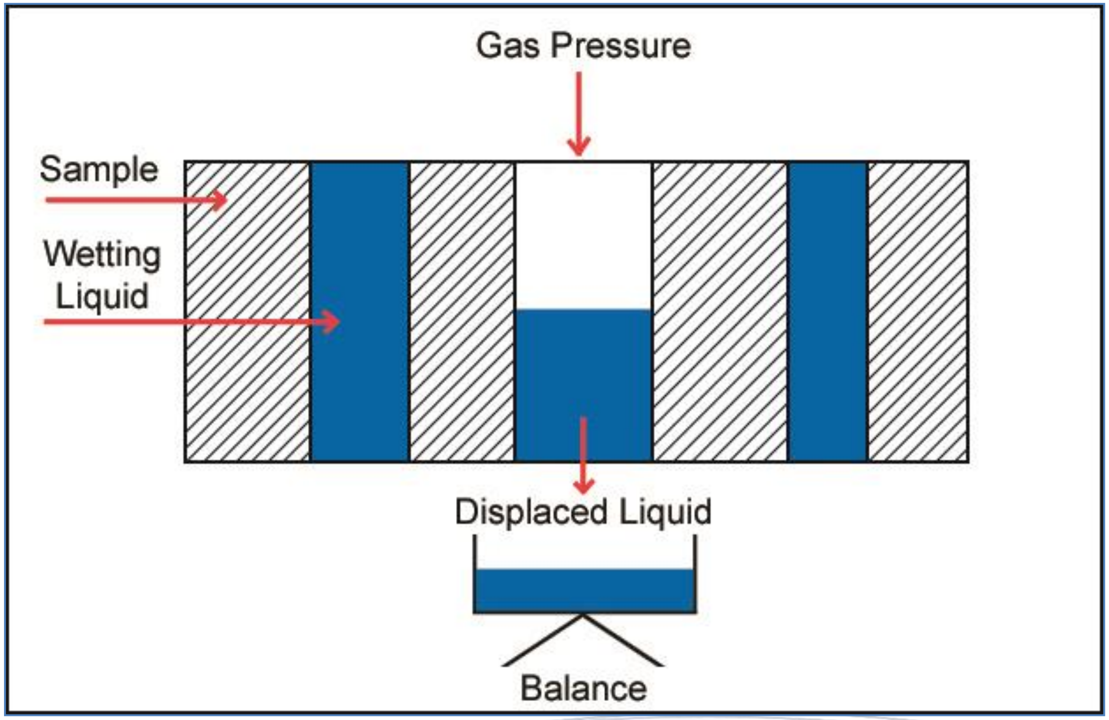


Characterization Techniques

Liquid Extrusion Porosimetry

-Measures differential gas pressure and volume of wetting liquid displaced from pores

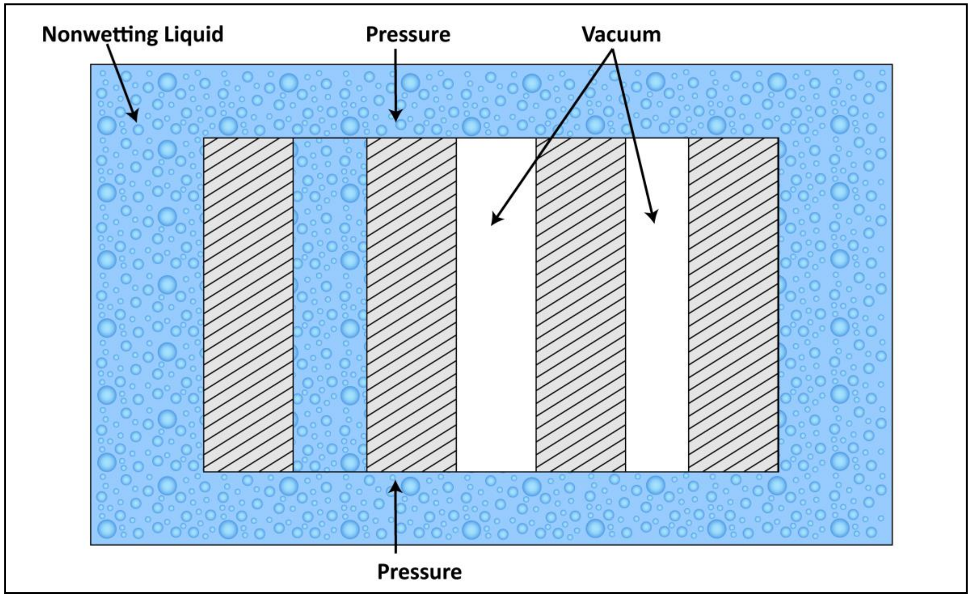
-Computes through pore volume, diameter and distribution



Characterization Techniques

Intrusion Porosimetry

- Increasing pressure forces non-wetting liquid in to pores
- Measures pressures and intrusion volume
- Computes pore volume and diameter

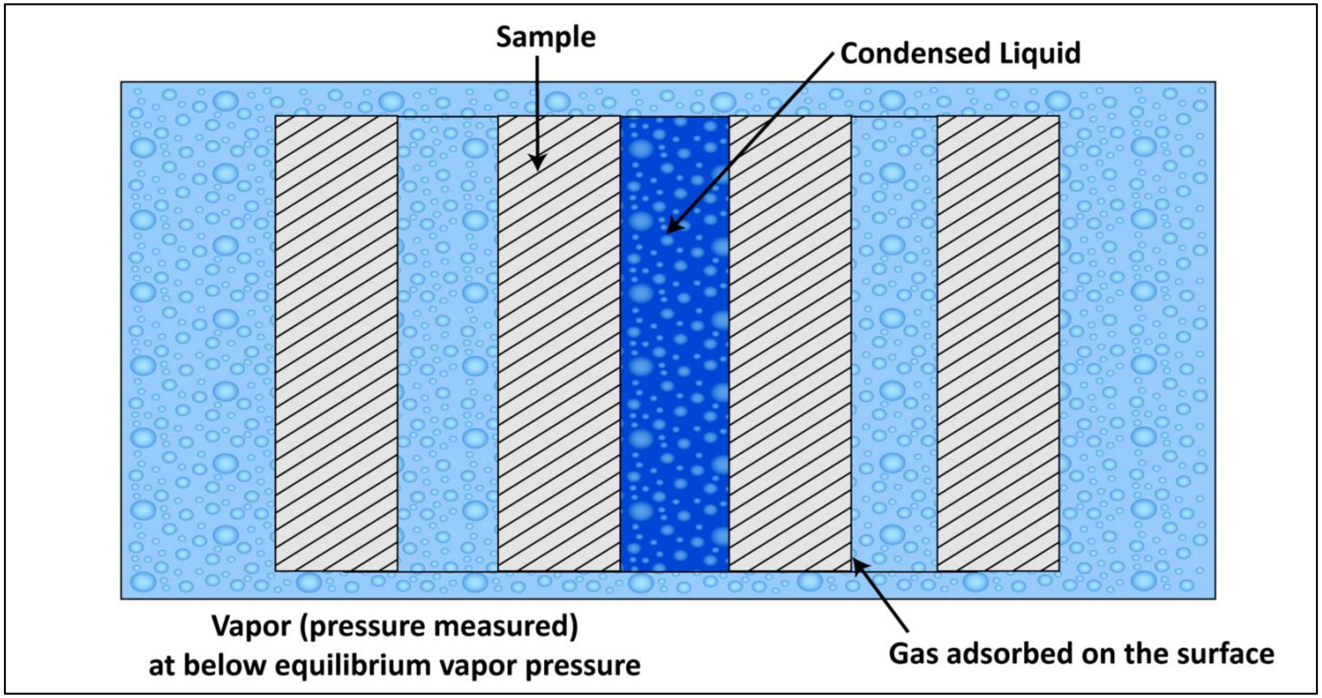


- Mercury (Mercury porosimetry)
- Water (Aquapore, Vacuapore)
- Other non-wetting liquids

Characterization Techniques

Gas Adsorption (BET) & Condensation

- Measures surface area from amount of gas adsorption
- Measures pore volume and diameter from amount of gas condensation



Principle of the gas adsorption technique

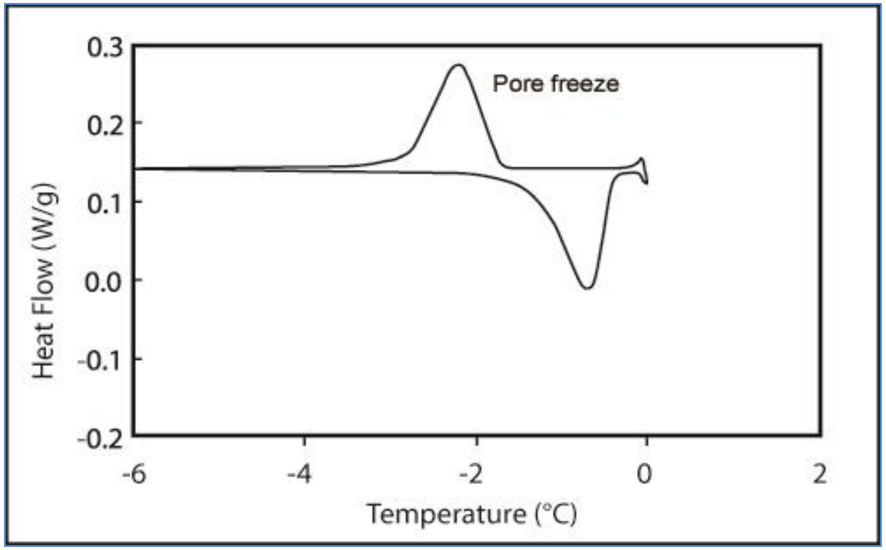
Characterization Techniques

Novel Techniques

Thermoporometry

Measures freezing points of a liquid in pores having different diameters

Pore diameter and distribution are computed



Gas permeation technique

- Measures flow rates of gases of differing molecular diameter
- Computes pore diameter and distribution

Characterization Techniques

Pore Structure Characteristics (Size, Volume, & Distribution)			
Size: > 500 μm	Size: < 500 μm	Size: 500 – 0.002 μm	Size: < 0.002 μm
1. Macroscopic techniques	1. Microscopy	1. Particle challenge	1. Capillary condensation flow porometry
		2. Flow porometry	2. . Liquid – Liquid porometry
		3. Extrusion porosimetry	3. Gas permeation porometry
		4. Gas adsorption	4. Thermoporometry
		5. Intrusion porosimetry	5. Gas adsorption
Pore Structure Characteristics (Fluid Permeability, Porosity)			
1. Gas/Vapor Permeametry			
2. Liquid Permeametry			
3. Pycnometry			

Summary

1. The pore structure is normally very complex. Characteristics must be carefully defined

2. Characteristics need to be measured:

   Bubble point (largest pore size)
   Mean flow pore size
   Pore size distribution
   Pore volume
   Pore volume distribution
   Liquid/gas permeability
   Total surface area
   Envelope surface area

3. A number of commercial techniques are available

4. The techniques, their capabilities, their limitations and all new developments will be critically examined in the following presentations

Thank You

PMI

POROSITY & PERMEABILITY TESTING