Influence of Aerogel Morphology and Reinforcement Architecture on Gas Convection in Aerogel Composites

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Background:

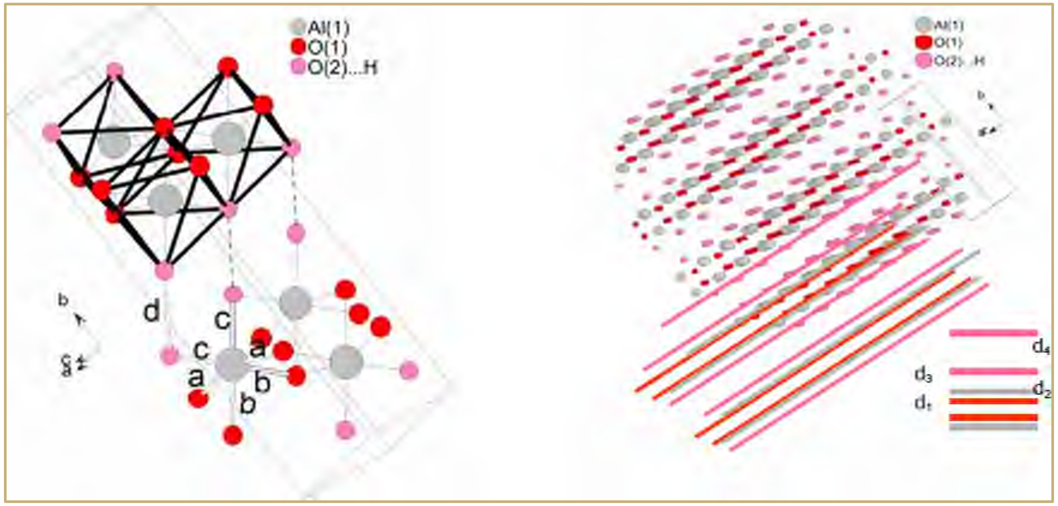
- Aerogels are very effective insulators, primarily in suppressing heat transfer by gas convection, but also in providing a tortuous path for solid conduction.
- Alumina and aluminosilicate aerogels can maintain mesoporous structure to temperatures of up to 1200˚C.
- These aerogels are fragile, but an be reinforced with fabrics, papers or foams to form composites in which the aerogel serves as the matrix.

Objective:

Characterize gas permeability of aluminosilicate aerogels with various ceramic fabric, paper or felt reinforcements.

Synthesis Approach: Boehmite alone to form alumina aerogel; Boehmite + TEOS co-condense to form aluminosilicate.

Boehmite [AlO(OH)]



AlO_6 octahedra

S. Brühne, Cryst. Growth Des., 2008, 8 (2), pp 489–493n

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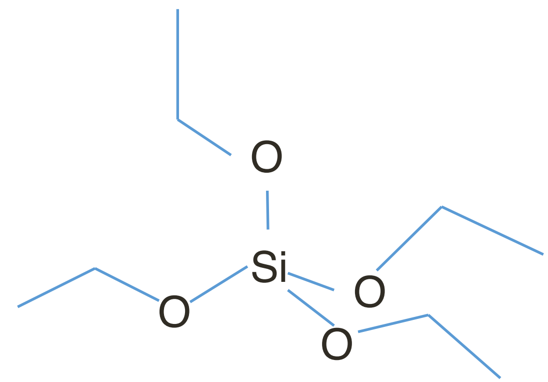
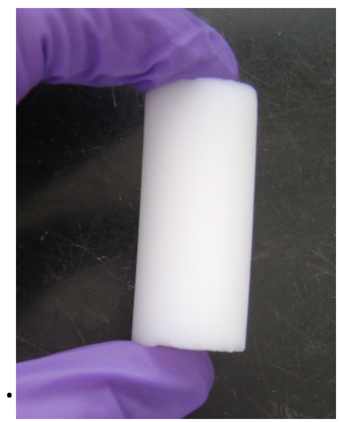
TEOS

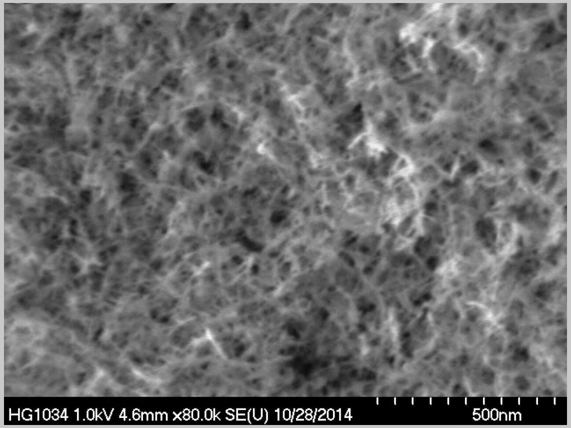




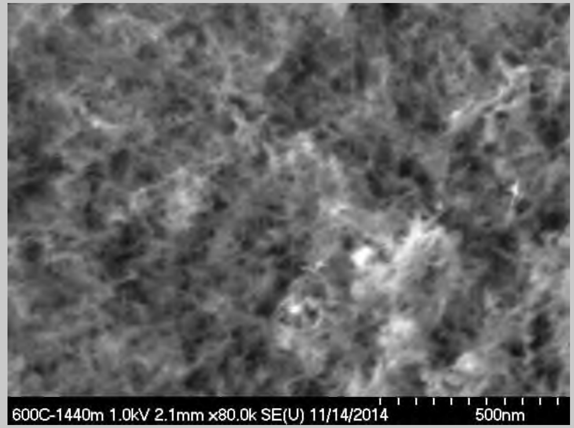
→ hydrogel $\xrightarrow{\text{supercritical } CO_2}$ **aerogel**

P. R. Aravind, *et al* Microporous and Mesoporous Materials **96** 14–20 (2006) .

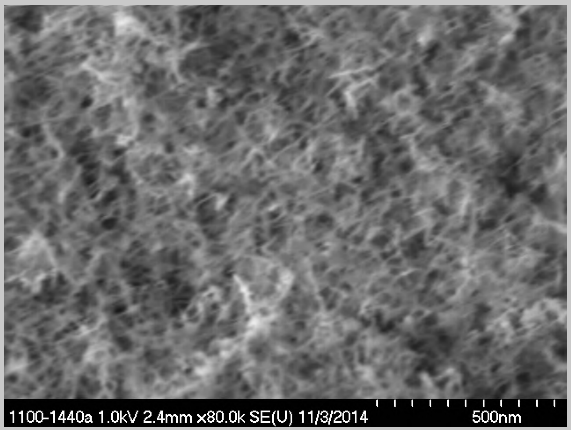
Introduction of silica into alumina lattice inhibits transformation to α-alumina (Hurwitz, *et al.*, manuscript in preparation)



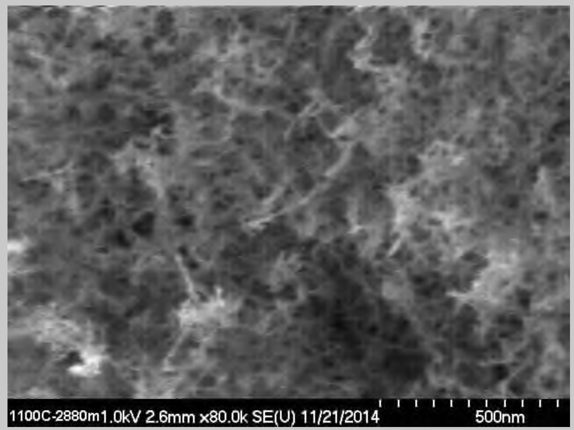
As supercritically dried



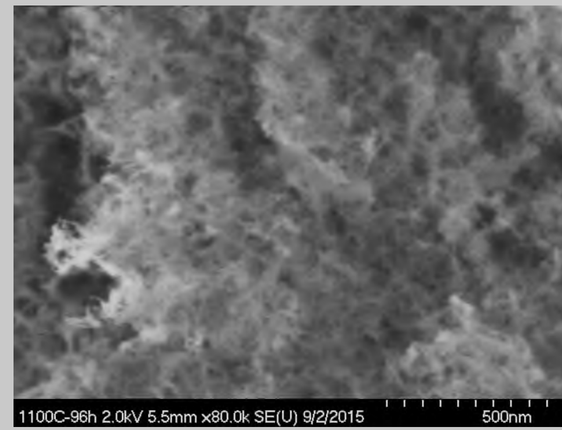
600˚C, 24h



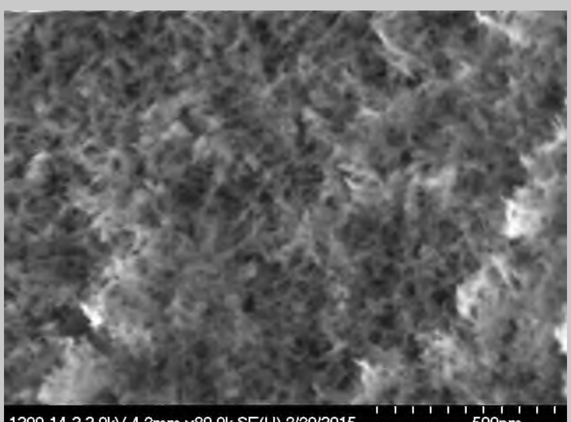
1100˚C, 24h



1100˚C, 48h



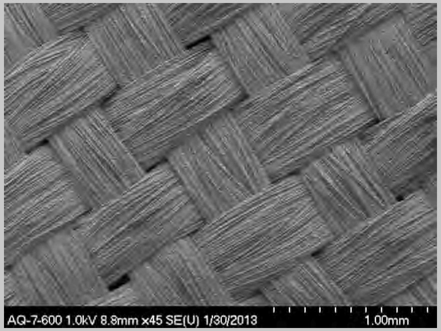
1100˚C, 96h



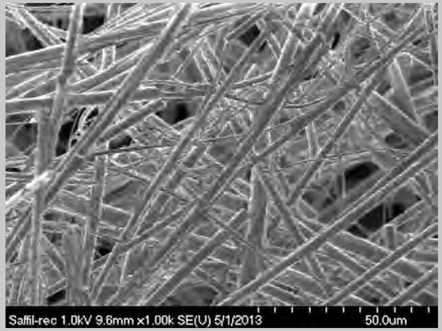
1200˚C, 24h

Micrographs showing persistence of mesoporous structure within aluminosilicate aerogel heated at 1100 and 1200˚C.

Examples of Alternative Reinforcements for Aerogel Composites (Partial List)



Astroquartz Fabric

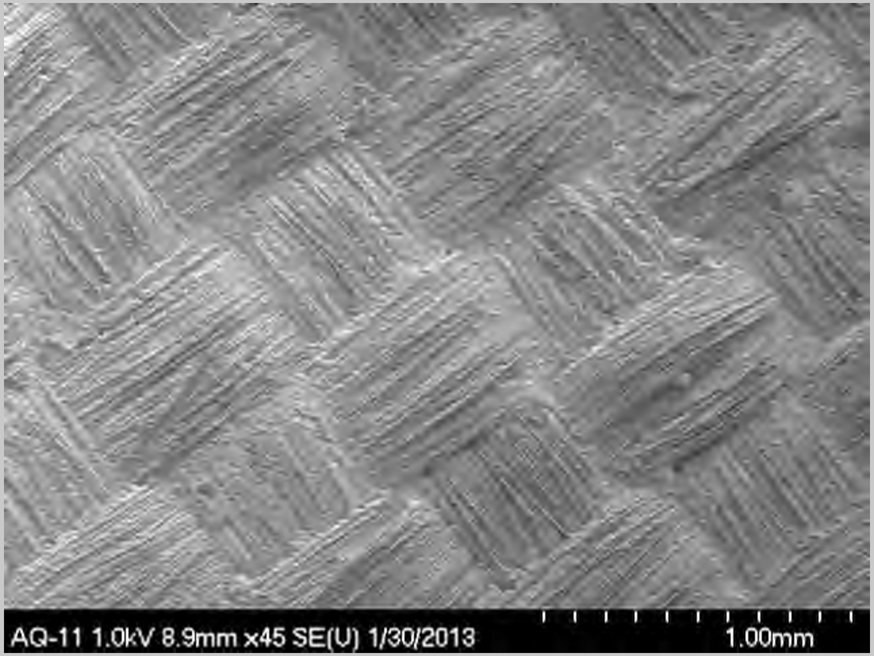


Saffil Paper

APA-2

Fiberfrax 972AH

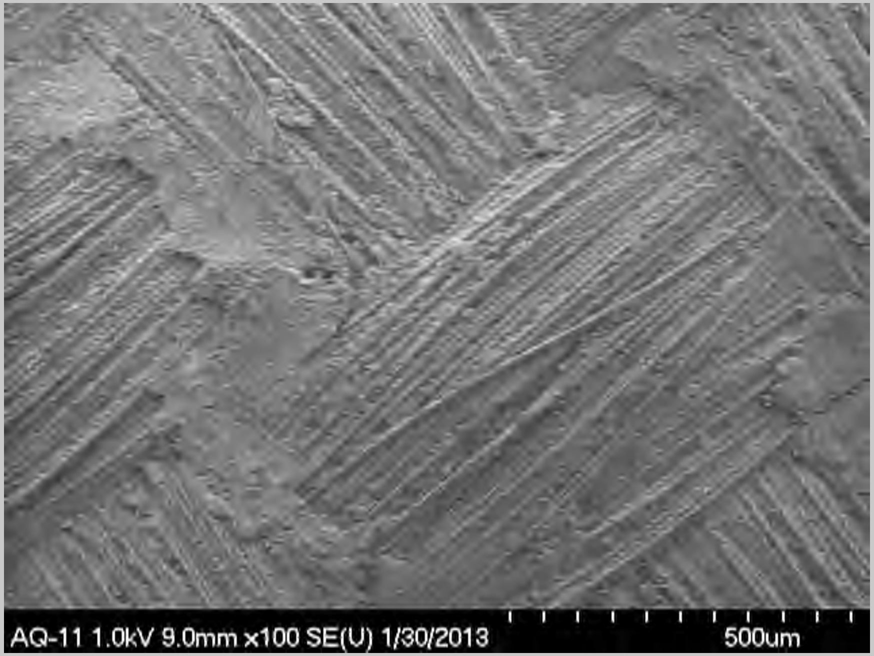
Ceramic Reinforcement	Thickness (mm)	Density (g/cm^3) As received	Composite Density (g/cm^3)	Upper Use Temperature	Composition (%)
Astroquartz (503 plain weave) Unsized	0.11	0.95	0.610-0.997	1070° C	99.99 SiO_2
Nextel 440	0.3	1.0	1.09	1100-1200˚C	γ-alumina + mullite + amorphous SiO_2
ZYW30A					
Saffil Paper	0.5, 1.0	0.15 (w binder) 0.125 (1000C)	0.144-0.194	1600° C	95-97 Al_2O_3, 3.0-5.0 SiO_2, <0.5 trace elements
APA-2 Paper	1.25	0.11	0.127-0.163	1650° C	86 Al_2O_3, 10 SiO_2, 4 other oxides
Fiberfrax 972AH	0.8	0.192	0.184-0.207	1176° C	47-52 Al_2O_3, 48-53 SiO_2, <0.5 Na_2O_3, <0.5 Fe_2O_3

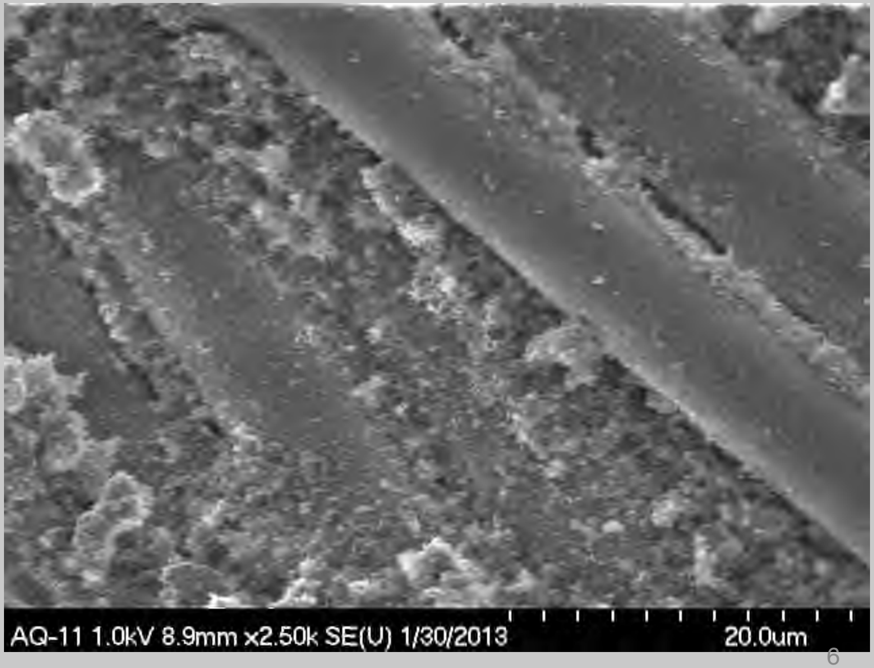


2-D Fabric composites

Aerogel impregnation into woven fabrics fills crossover points between tows as well as inter-fiber spacing within tows, decreasing gas permeability.

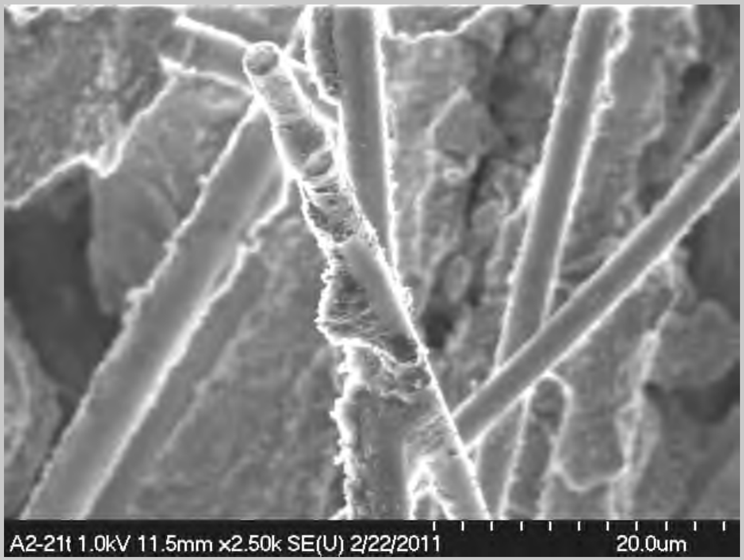
Example shown in Astroquartz 503 (plain weave)



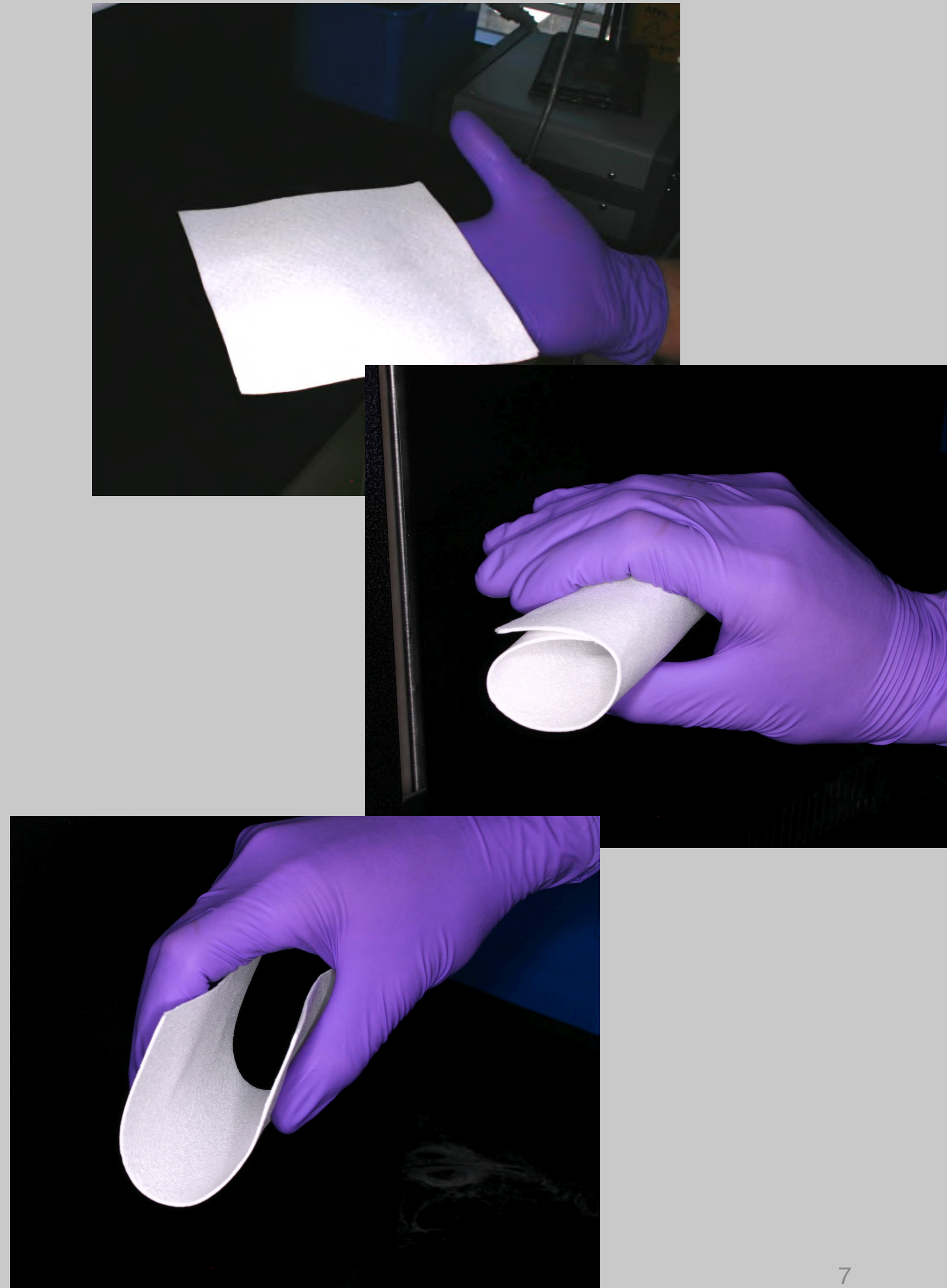


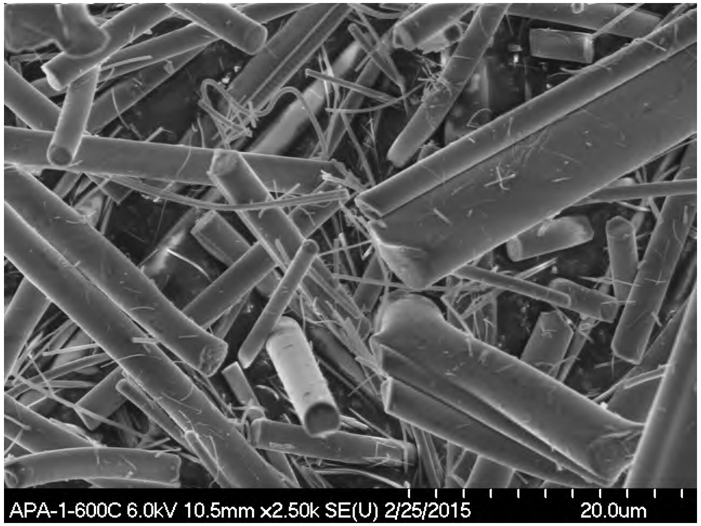
APA-2/ aerogel composite

Density of 0.14 g/cm^3, lighter than Microtherm HT (0.3428g/cm^3)

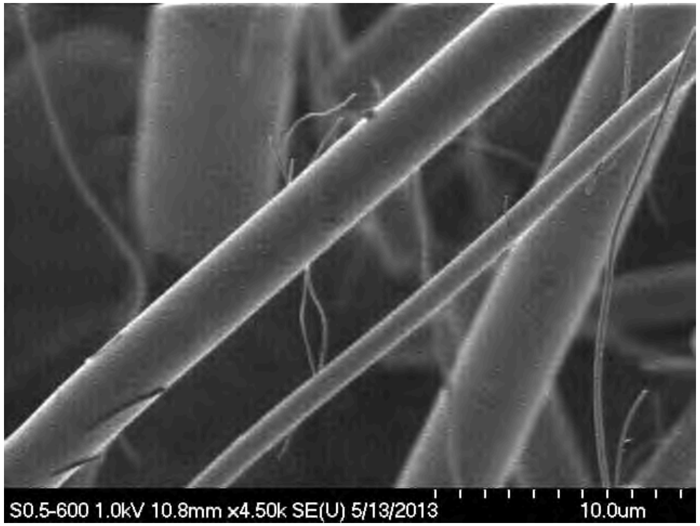


Aerogel bonds to fibers; unlike commercial materials, particles do not spall. Aerogel/fiber bond achieved by heat treatment of alumina paper to remove all binders prior to sol impregnation.

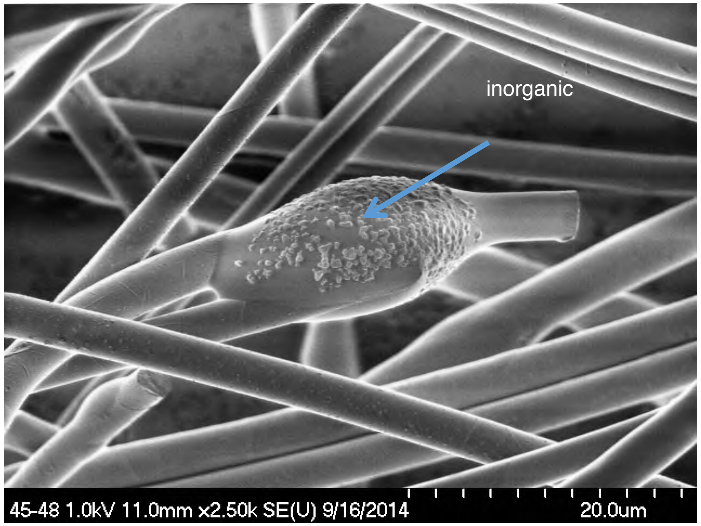




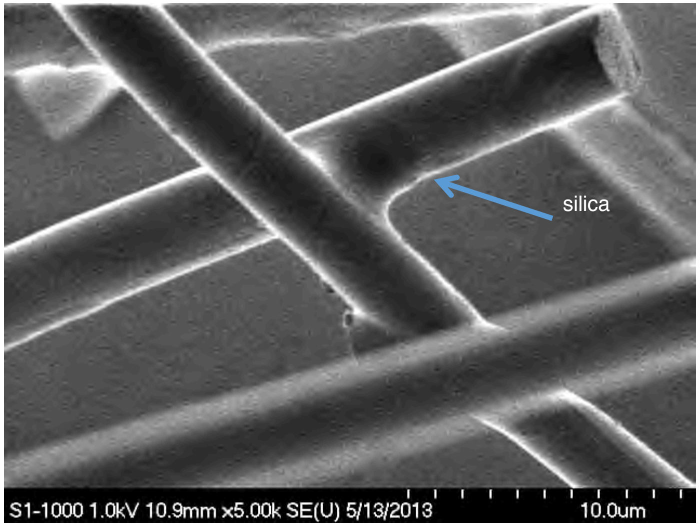
APA-1, binder removed



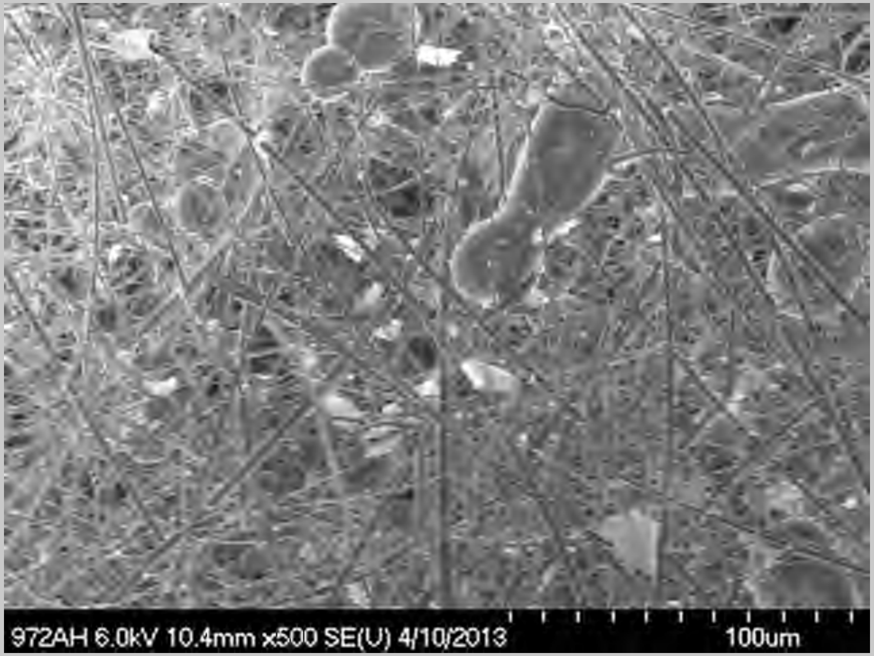
Saffil paper, binder removed



APA-2, no organic binder (inorganic may include Si, S, Ba, K,Ca)



Saffil paper, 1000˚C

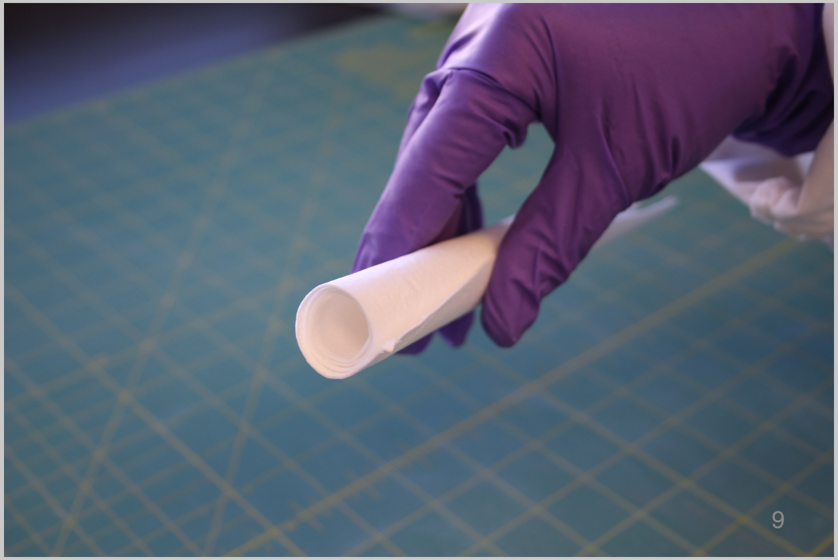


Fiberfrax 972AH

47-52 Al_2O_3, 48-53 SiO_2, <0.5 Na_2O_3, <0.5 Fe_2O_3
Paper can be handled without tearing after binder removal.
Can be treated to remove sodium from aluminosilicate “flakes”.

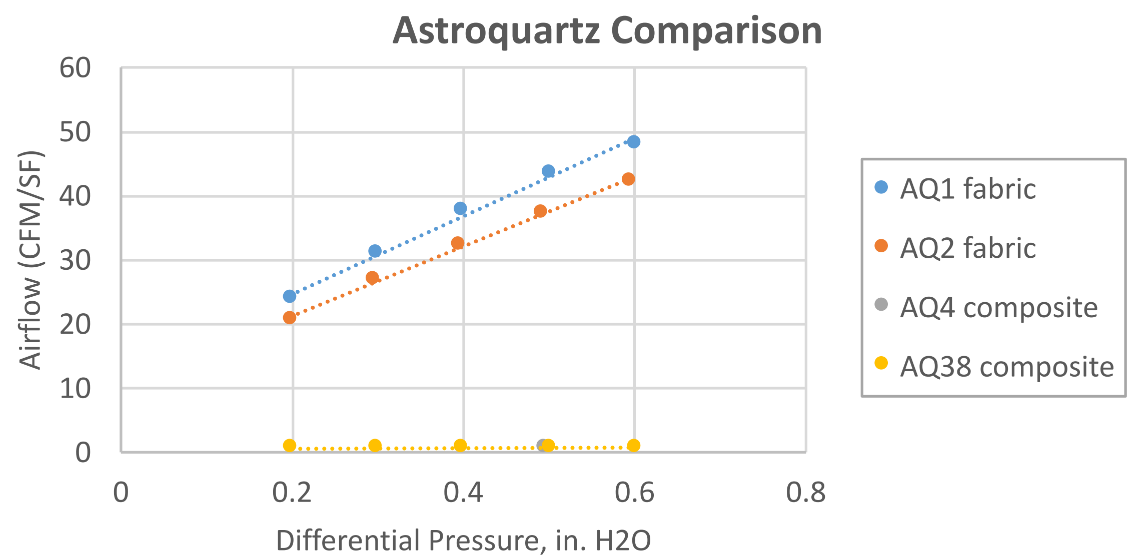
Operating temperature: 1176 C

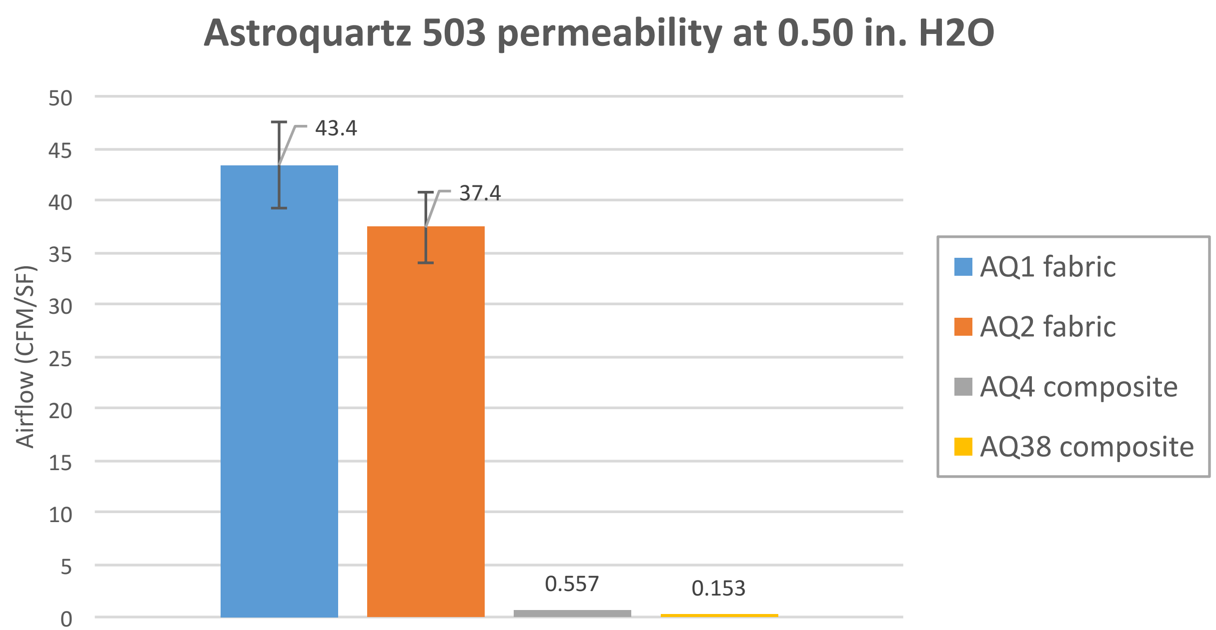
ISSUE: Most flexible, but contains small diameter silica fibers; composite will not tolerate sharp fold-but will fold over “slip layer”.



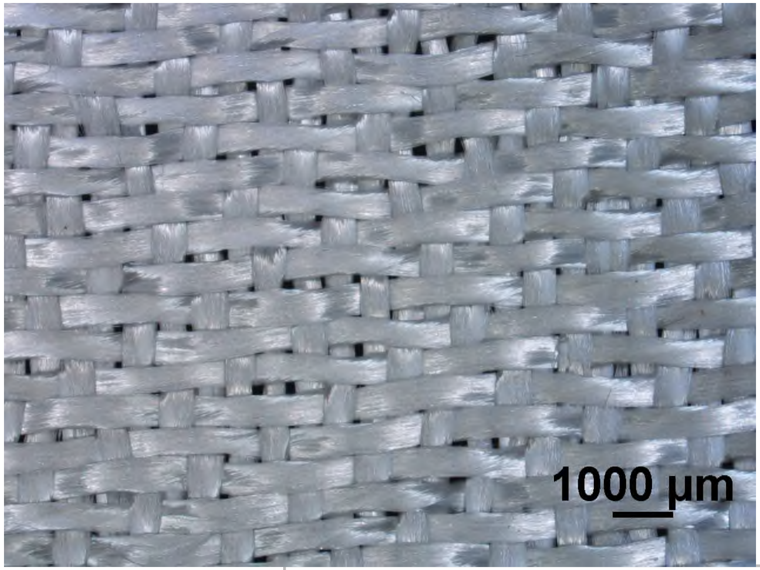
Reinforcement	Mass fraction aerogel
Astroquartz	0.10
Nextel 440	0.05
ZYW30A	0.06
APA-2	0.37
Saffil	0.30
Fiberfrax	0.28
Quartz papers	0.52-0.62
Quartz felt	0.6-0.9

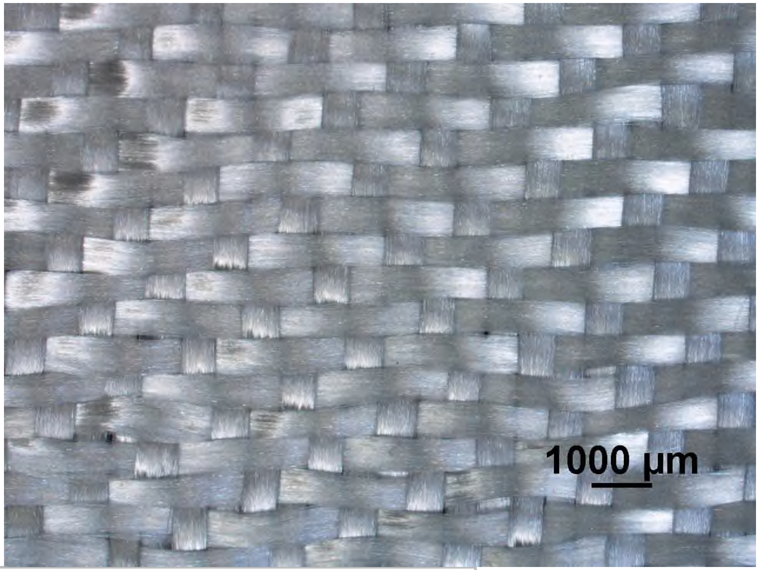
Astroquartz



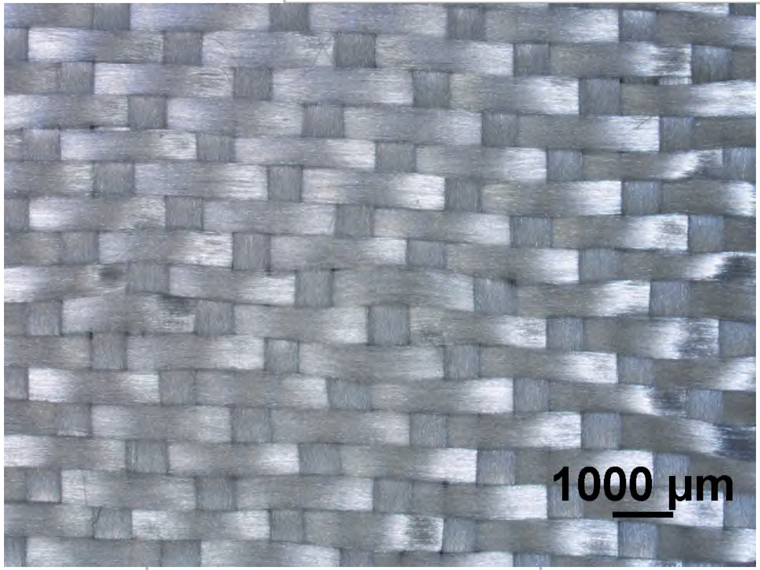


Nextel fabrics

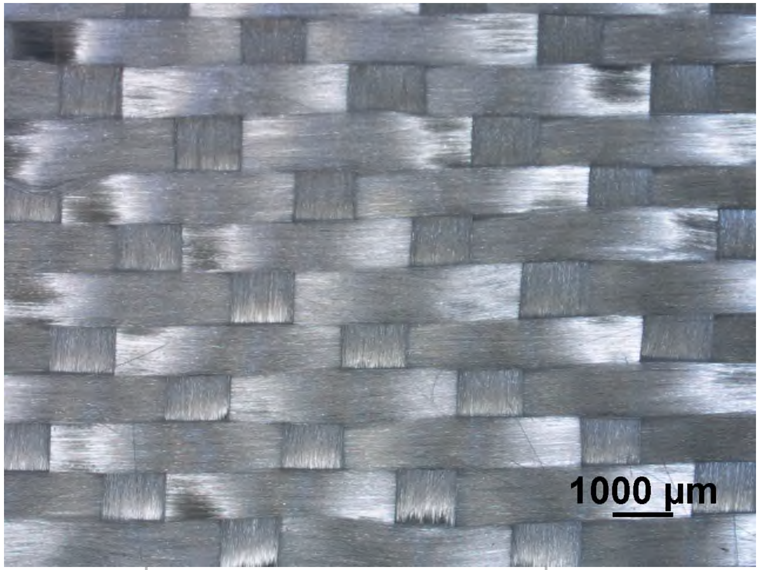




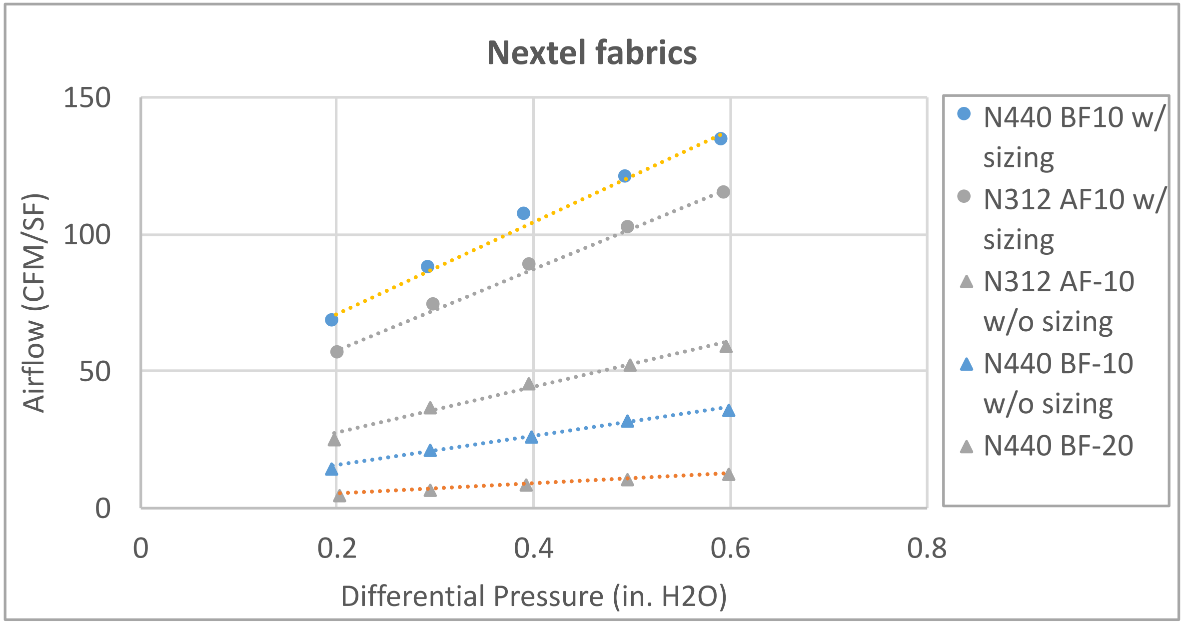
AF10 with sizing (left) and without sizing (right).

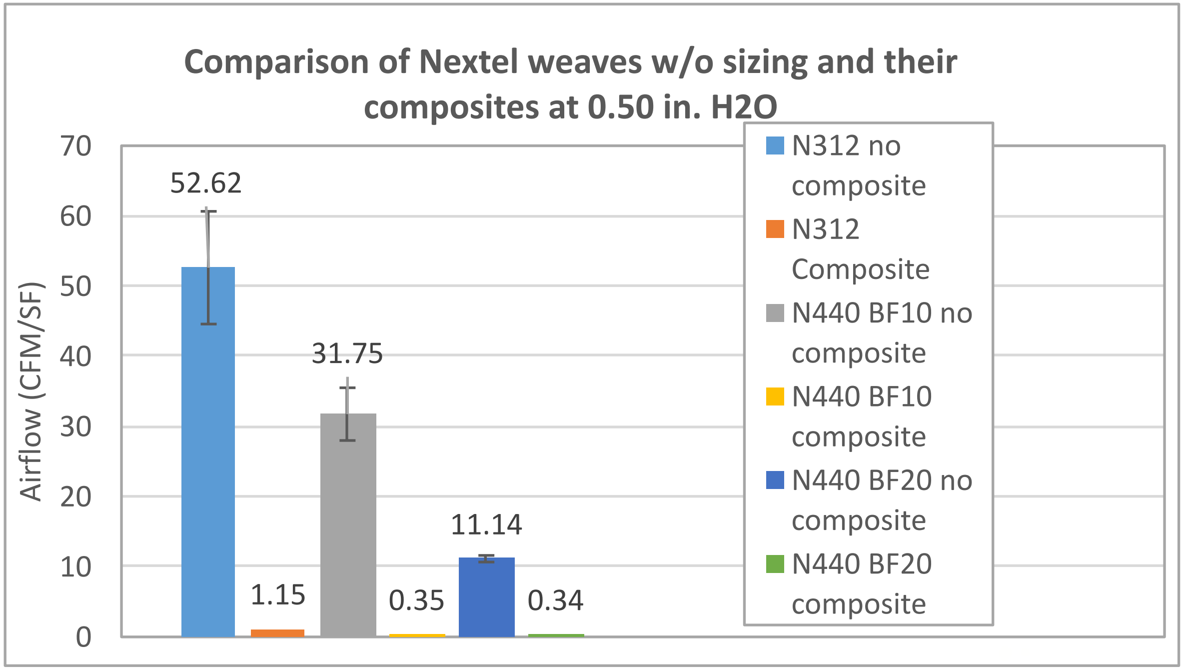


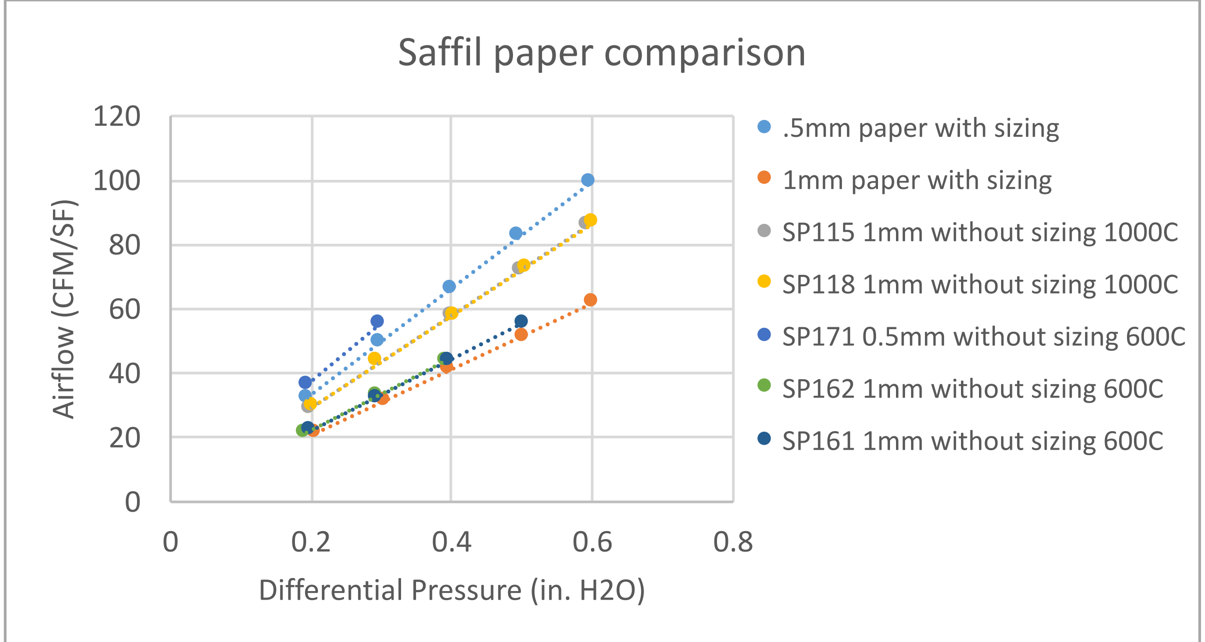
BF10 without sizing



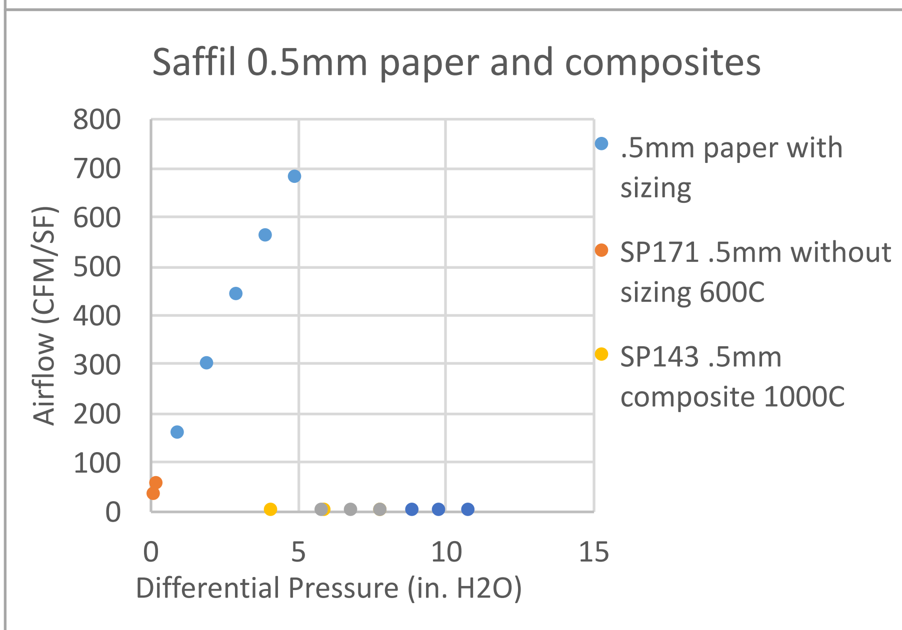
BF20 without sizing

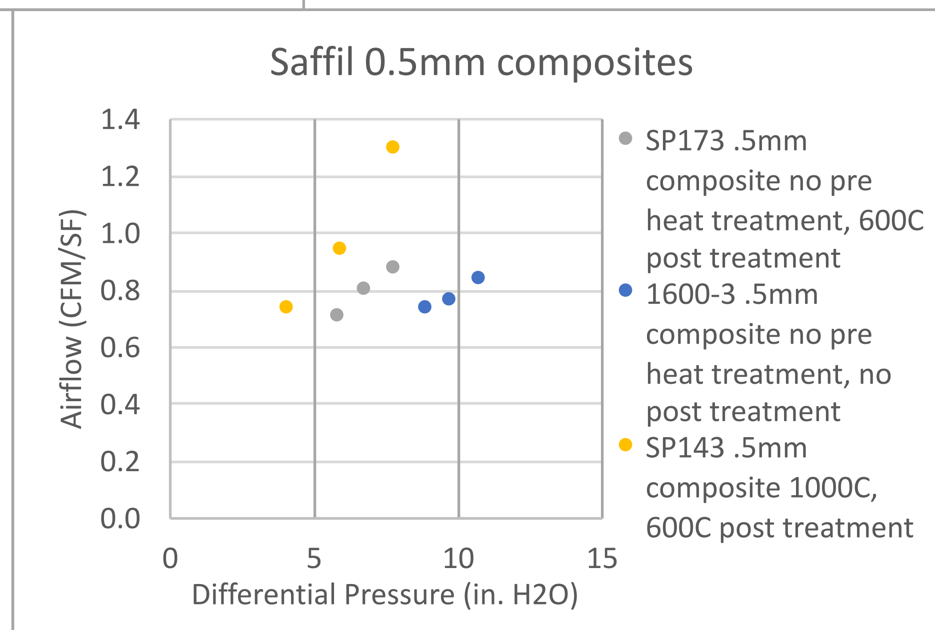

Nextel fabrics
Airflow (CFM/SF)
Differential Pressure (in. H2O)
150
100
50
0
0
0.2
0.4
0.6
0.8
N440 BF10 w/ sizing
N312 AF10 w/ sizing
N312 AF-10 w/o sizing
N440 BF-10 w/o sizing
N440 BF-20

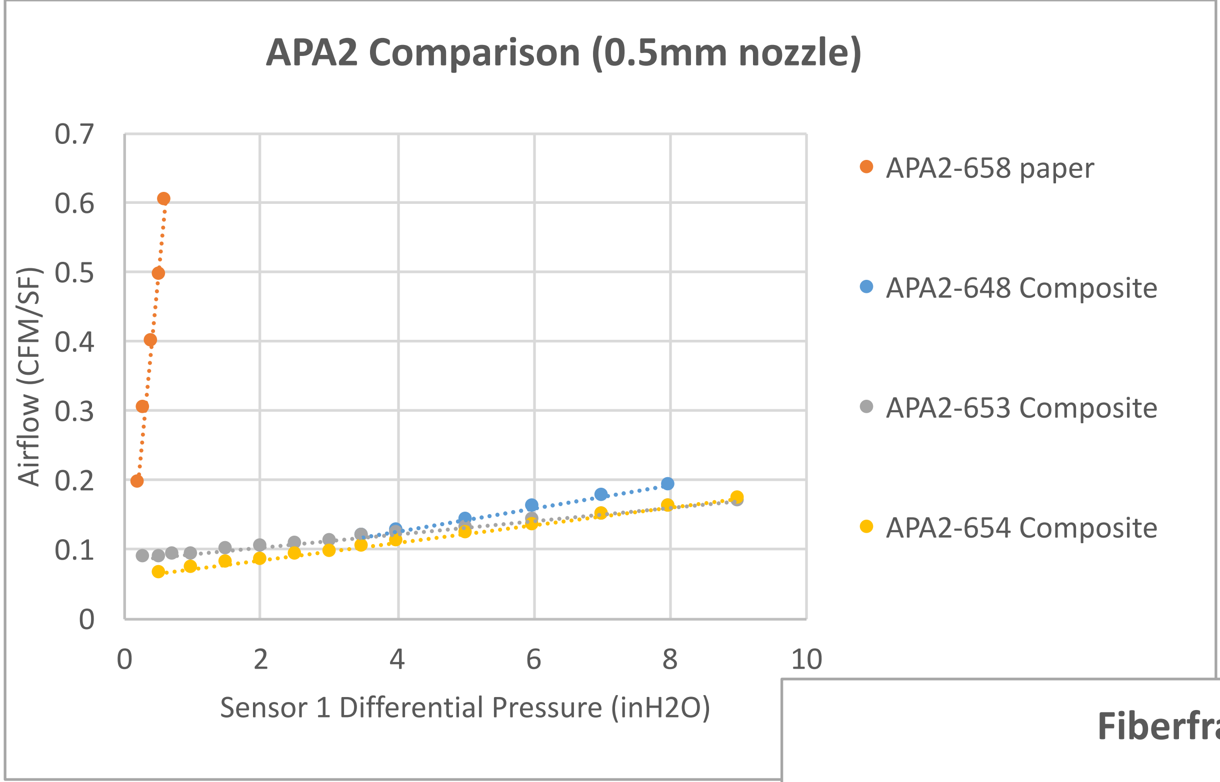

Comparison of Nextel weaves w/o sizing and their composites at 0.50 in. H2O
Airflow (CFM/SF)
70
60
50
40
30
20
10
0
52.62
1.15
31.75
0.35
11.14
0.34
N312 no composite
N312 Composite
N440 BF10 no composite
N440 BF10 composite
N440 BF20 no composite
N440 BF20 composite



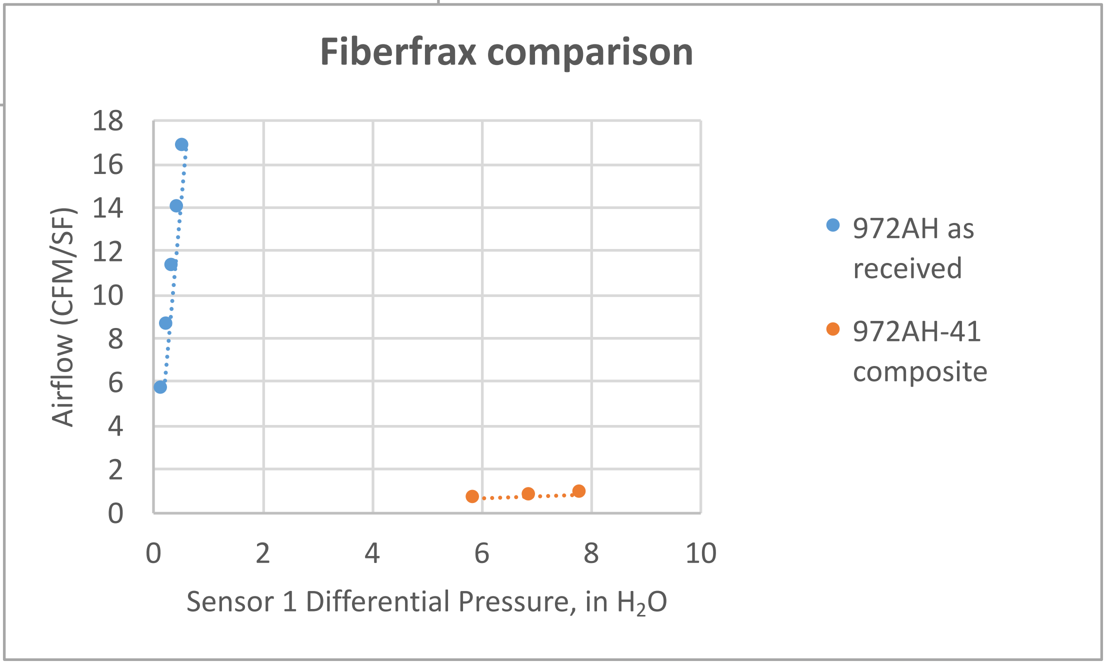
Note variations in scale!

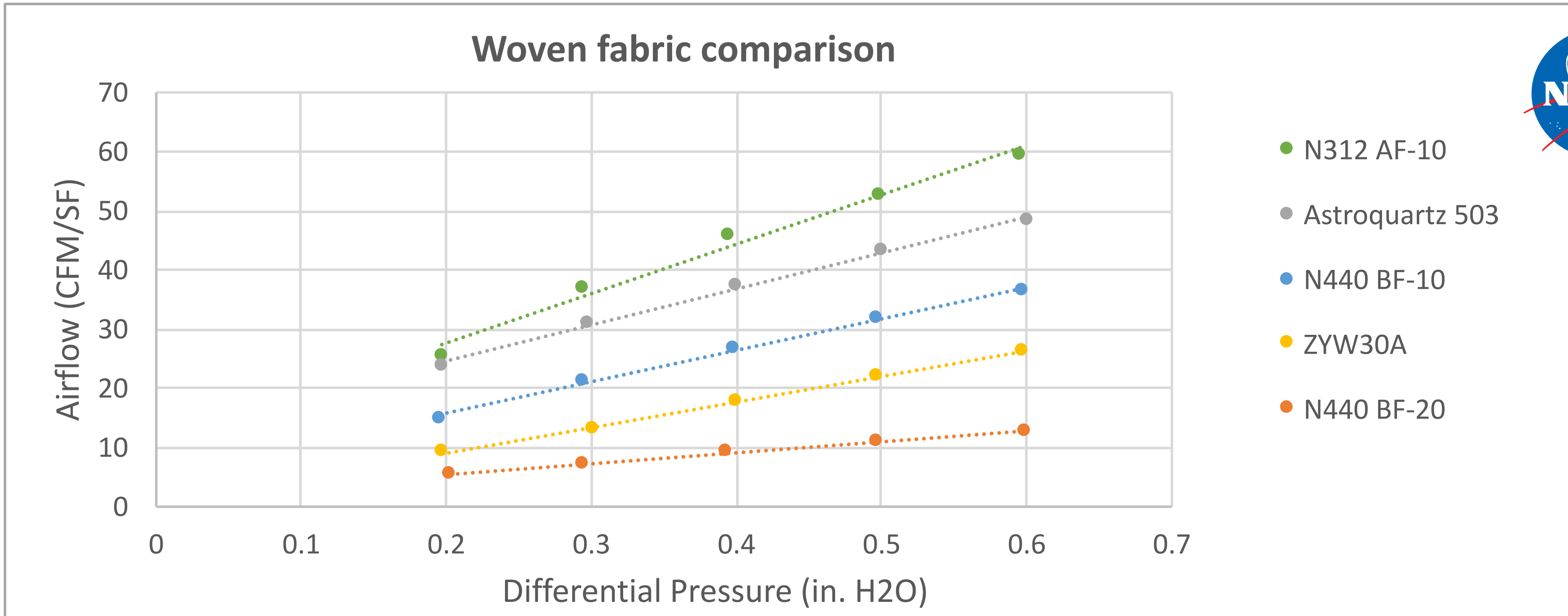


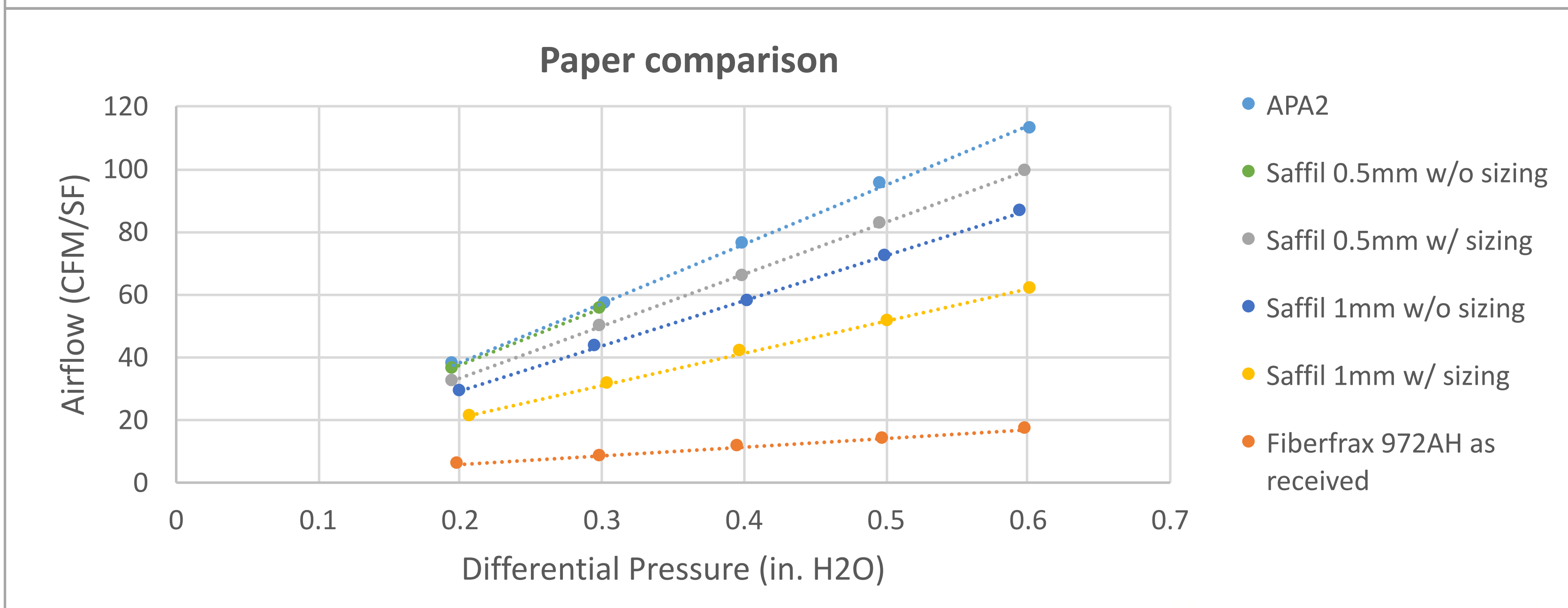


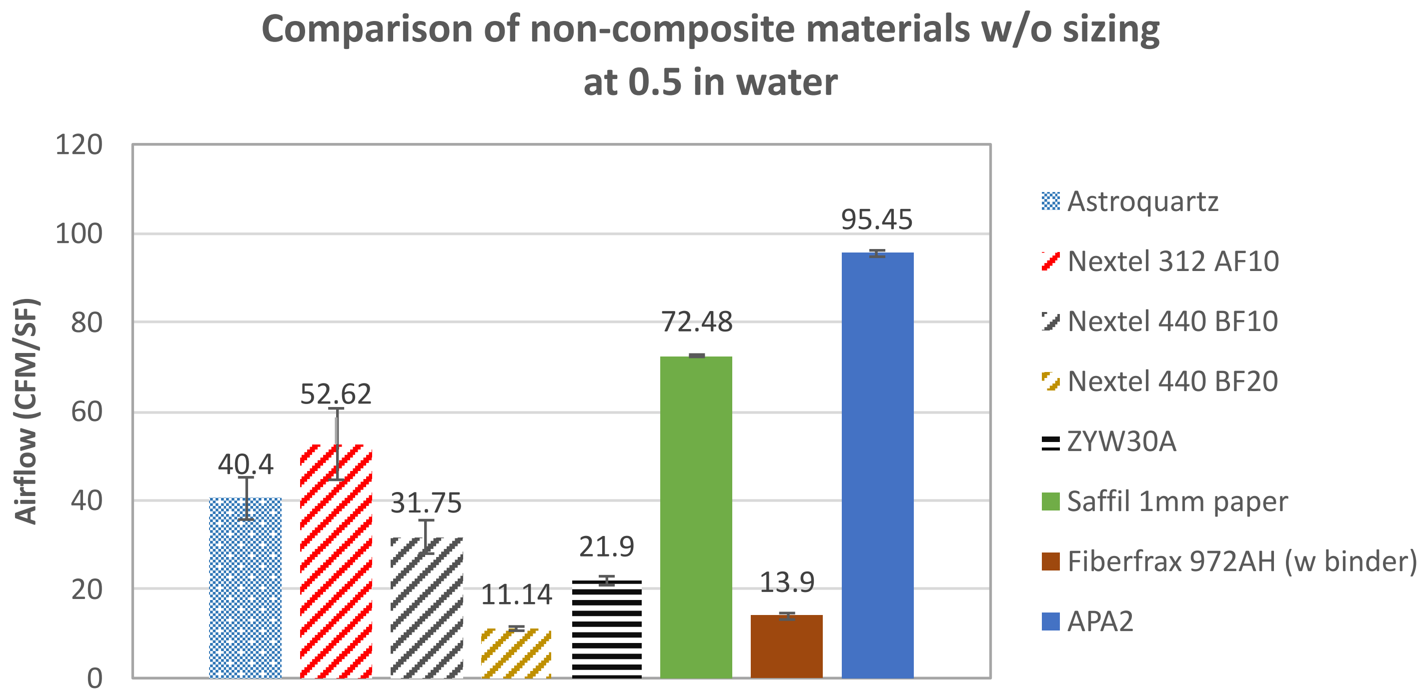


Incorporation of aerogel provides marked reduction in permeability for both papers, but APA2 paper has much lower permeability than Fiberfrax paper alone.

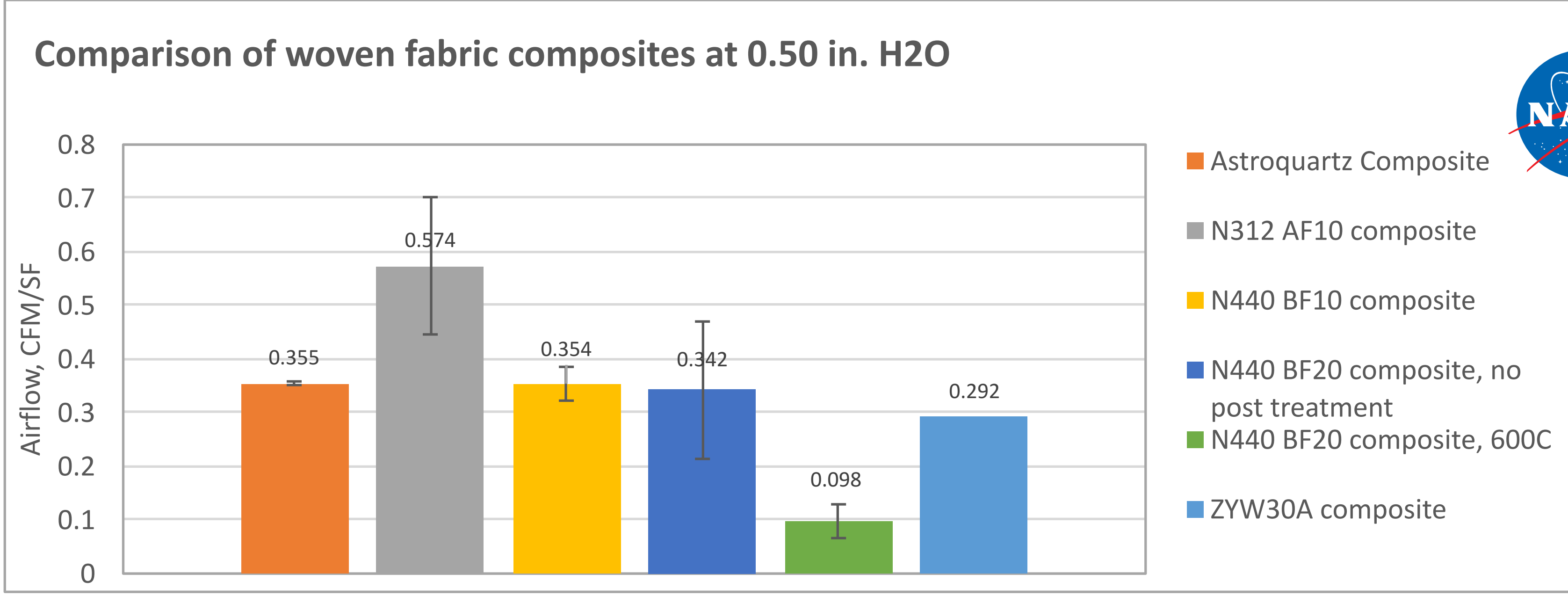


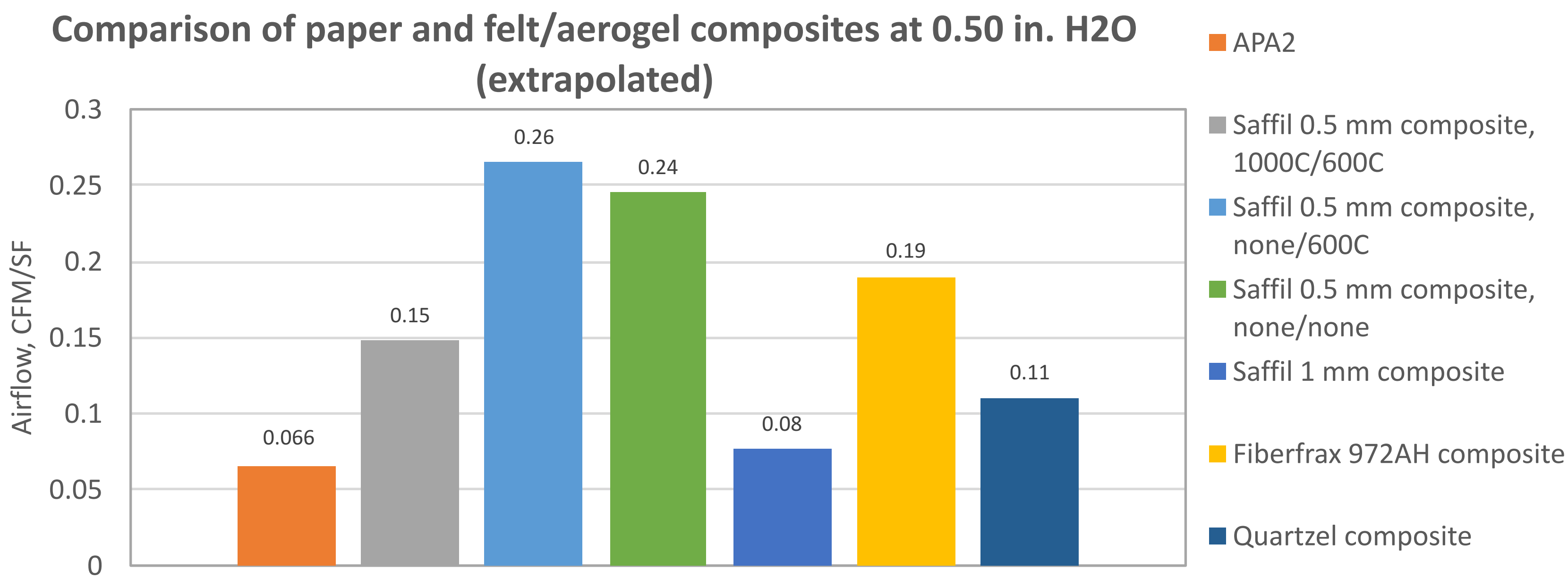

Woven fabric comparison
Airflow (CFM/SF)
70
60
50
40
30
20
10
0
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
Differential Pressure (in. H2O)
N312 AF-10
Astroquartz 503
N440 BF-10
ZYW30A
N440 BF-20


Paper comparison
Airflow (CFM/SF)
120
100
80
60
40
20
0
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
Differential Pressure (in. H2O)
APA2
Saffil 0.5mm w/o sizing
Saffil 0.5mm w/ sizing
Saffil 1mm w/o sizing
Saffil 1mm w/ sizing
Fiberfrax 972AH as received

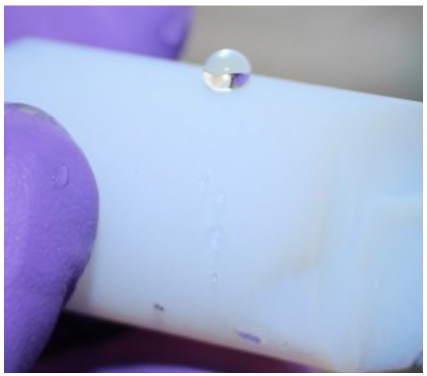


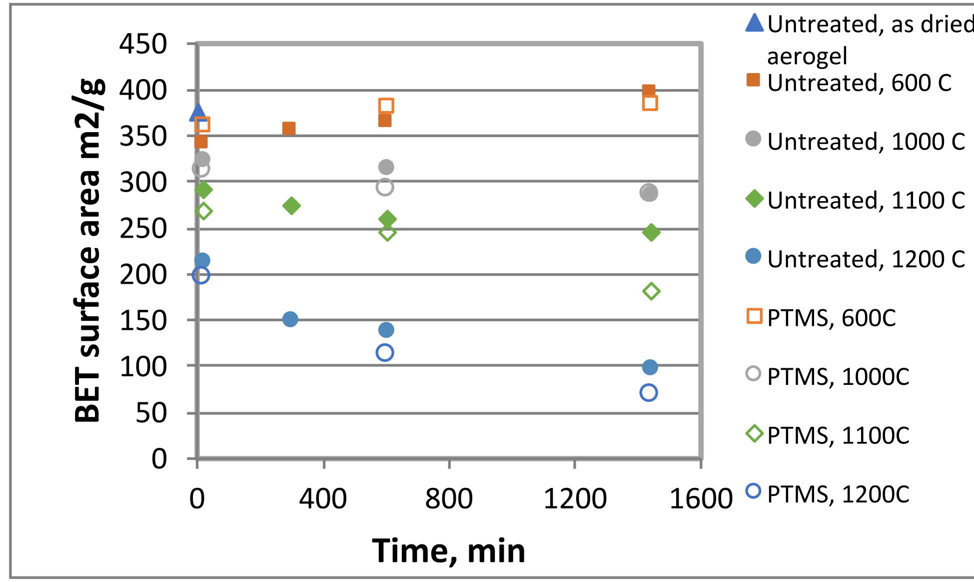
Saffil 0.5mm paper w/o binder was too fragile to be tested at 0.50 in. H2O.


Comparison of woven fabric composites at 0.50 in. H2O
Airflow, CFM/SF
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0.355
0.574
0.354
0.342
0.098
0.292
Astroquartz Composite
N312 AF10 composite
N440 BF10 composite
N440 BF20 composite, no post treatment
N440 BF20 composite, 600C
ZYW30A composite

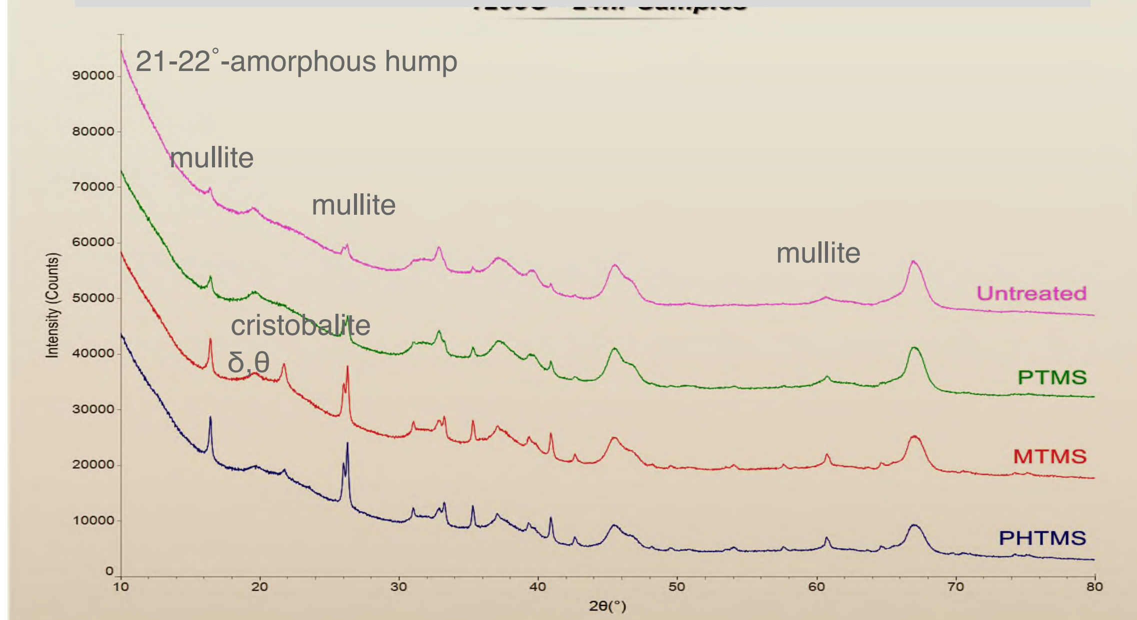

Comparison of paper and felt/aerogel composites at 0.50 in. H2O (extrapolated)
Airflow, CFM/SF
0.3
0.25
0.2
0.15
0.1
0.05
0
0.066
0.15
0.26
0.24
0.08
0.19
0.11
APA2
Saffil 0.5 mm composite, 1000C/600C
Saffil 0.5 mm composite, none/600C
Saffil 0.5 mm composite, none/none
Saffil 1 mm composite
Fiberfrax 972AH composite
Quartzel composite

Hydrophobic treatments available
Use of PTMS shows some reduction of surface area at high temperature.
Some additional silica incorporation occurs; PTMS does not produce cristobalite seen with other silanes.





X-ray Diffraction of Hydrophobic Samples Heated at 1200C, 24h



Trades:

- Woven fabrics incorporate low fraction of aerogel, but can reduce gas permeability, reducing convective heat transfer.
- Alumina fiber provides opacification; alumina papers offer best thermal performance.
- Saffil paper contains some small silica fibers which bridge fiber cross-over points on heating to 1000˚C, stiffening paper. Aerogel composite thermal properties and permeability are comparable to APA-2. Saffil available as 0.5 mm paper, thinner than 1 mm APA-2, and therefore slightly more flexible.
- Fiberfrax 972AH offers highest flexibility, but slightly higher thermal conductivity and permeability and contains respirable fiber. (APA-1 and Saffil also contain small diameter fibers). Most flexible, particularly with “slip” layer.
- Quartz felts offer highest aerogel incorporation on a volume fraction basis (fiber volume fraction is low), and offer lowest density and more robust mechanical strength than many of the papers.

Conclusions:

- In 2-D fabrics, weave architecture strongly influences permeability. Removal of organic sizing permits spreading of tows to reduce gas flow.
- Impregnation of 2-D fabrics with aerogel provides more than an order of magnitude reduction in permeability. N440 (BF20) provided lowest permeability of options tested, but is a heavy option (1.1 g/cm^3).
- Ceramic papers generally offer further reduction in permeability and lighter weight than fabrics, but are more fragile mechanically, and composites of most papers are less flexible than the 2-D weaves.
- Waterproofing techniques have been developed; however, influence on permeability has not as yet been evaluated.