Development of Aluminosilicate Aerogel Impregnated Oxide Foams for Structurally Integrated Thermal Protection Systems

Frances I. Hurwitz
Anna R. Palczer
Richard B. Rogers

NASA Glenn Research Center
Cleveland, OH 44135

Feliks Peysakhov, SUNY Stony Brook
Scott P. White, U Iowa

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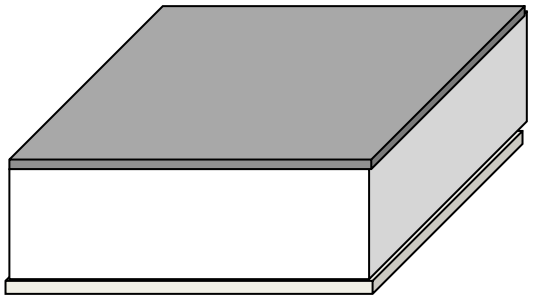
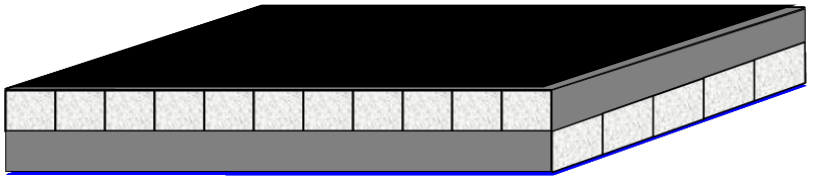
Insulating Cores for Structurally Integrated TPS (Integrated mechanical and thermal loads)

- Light-weight
- High volumetric heat capacity
- Low effective thermal conductivity
- Load bearing or non-load bearing
- Non-oxidizing
- Dimensional stability

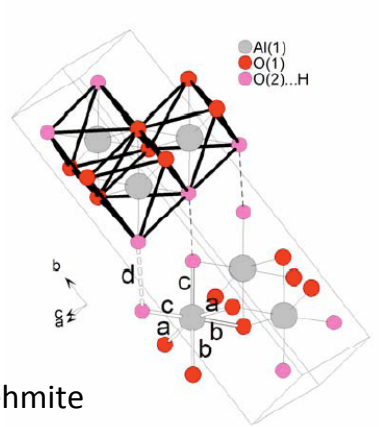


Can aerogel incorporation reduce thermal conductivity while maintaining dimensional stability, allowing for lighter weight structural elements?



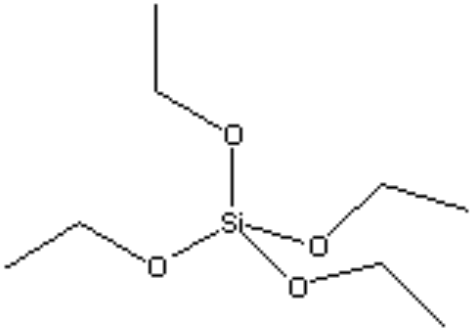
Susan White, Dan Rasky, "Fibrous-Ceramic/Aerogel Composite Insulating Tiles", NASA Tech Briefs 2004

Boehmite [γ-AlO(OH)]+ TEOS → hydrogel → aerogel

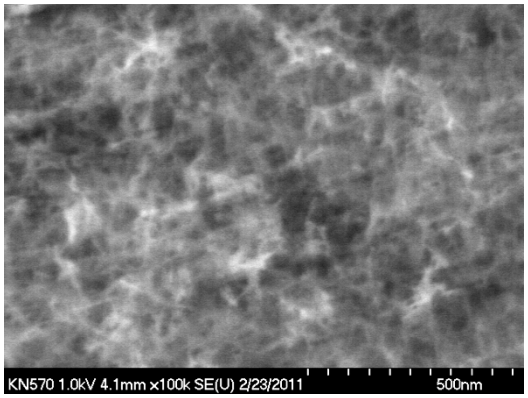


Boehmite

+

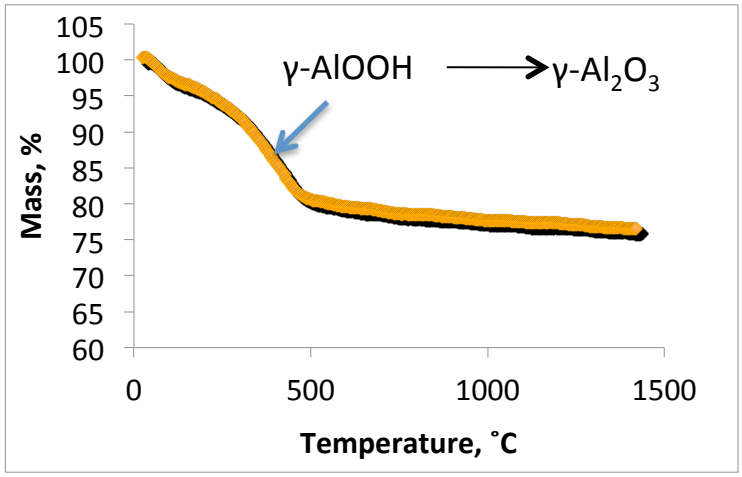


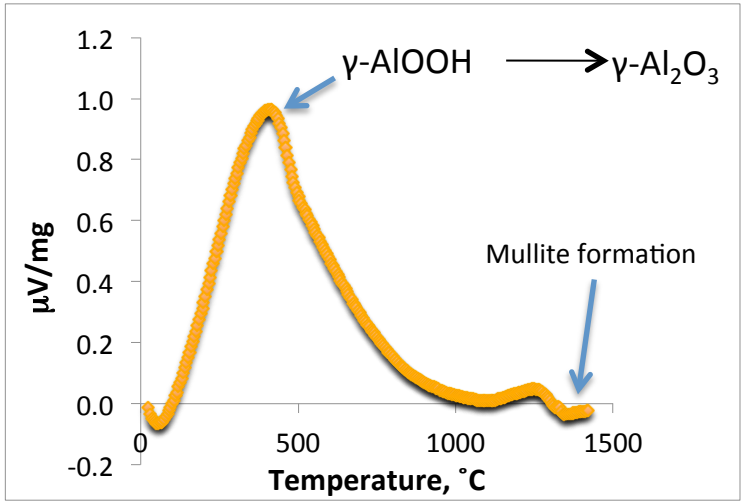
TEOS



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

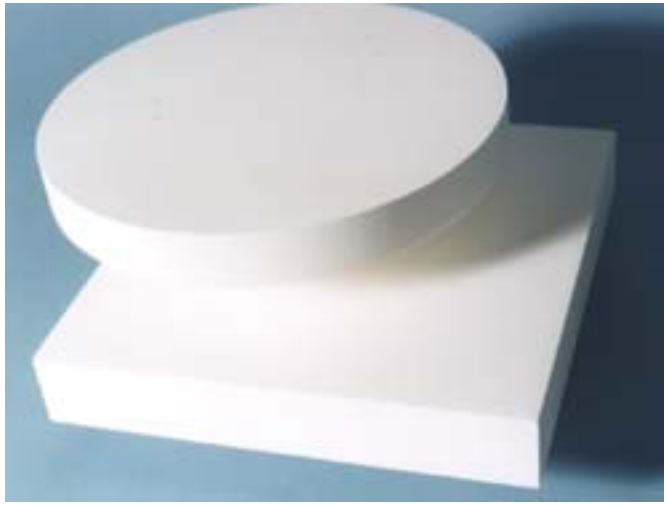
P2W 3Al:1Si, 412 m^2/g



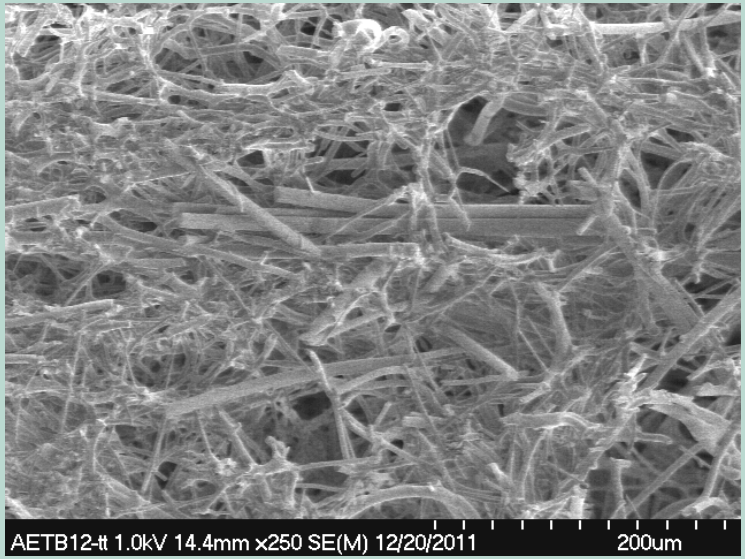


Oxide Foam Properties

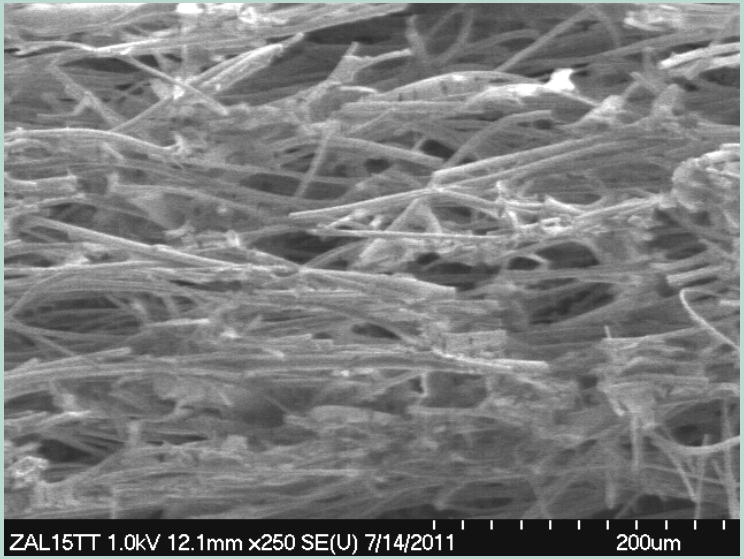
Material	Density (g/cc)	Thermal Conductivity (W/m-K)	Specific Heat (J/kg-K)	Al_2O_3	SiO_2	Binder	Source
AETB-12	0.192	0.064 (predicted)	628	20%	68%	Glassy phase; Inclusion of silica and aluminoborosilicate fibers provides bonding	TPSX
M15	0.240	0.16	1050	85%	15%	High Purity Silica	Zircar Zirconia
M2-35	0.624	-	1050	85%	15%	Mullite	Zircar Zirconia
ZAL-15	0.240	0.16	1047	85%	15%	High Purity Silica	Zircar Ceramics



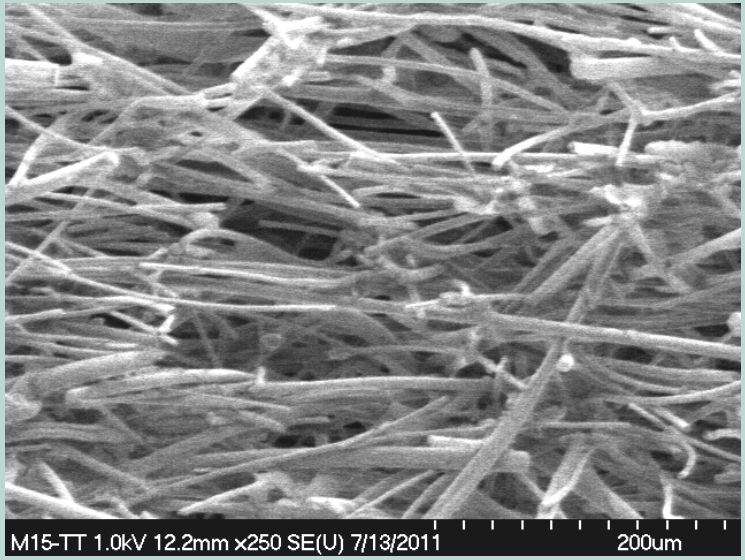
Microstructure: As-received foams



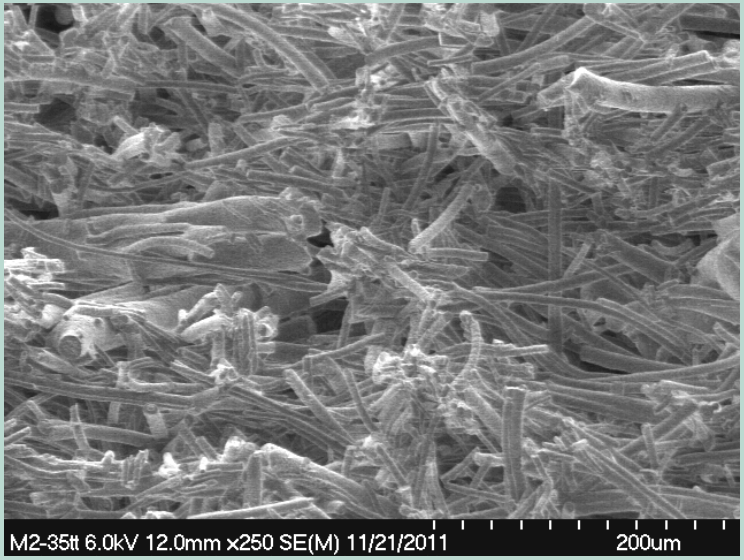
AETB-12



ZAL-15

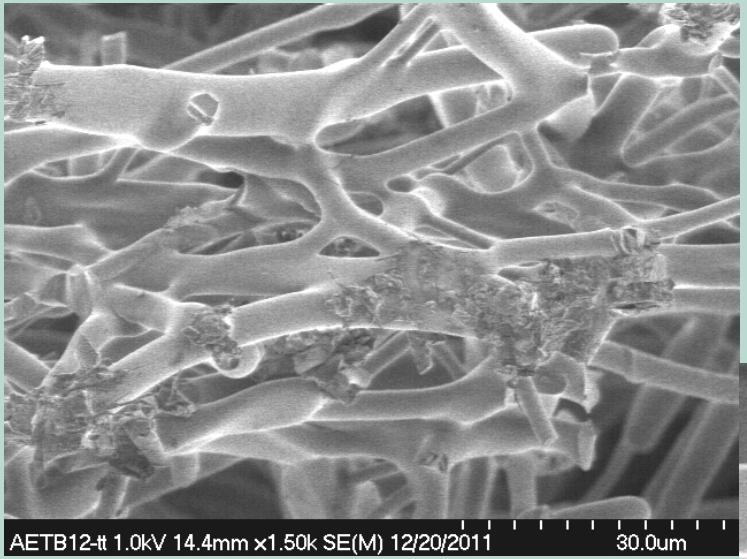


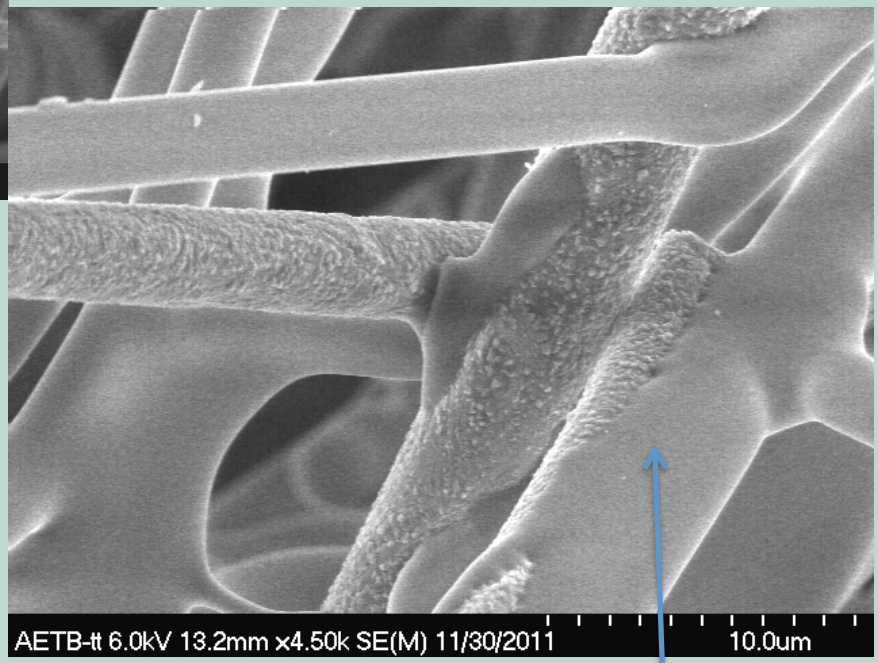
M-15



M2-35

AETB-12

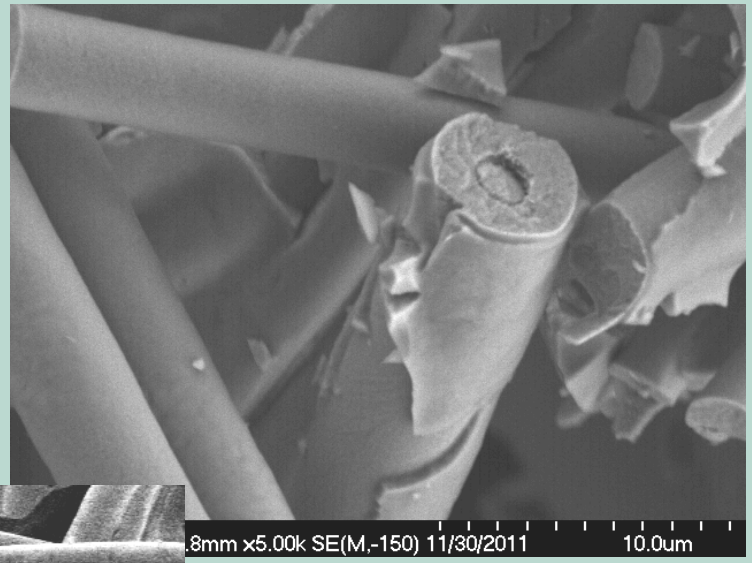


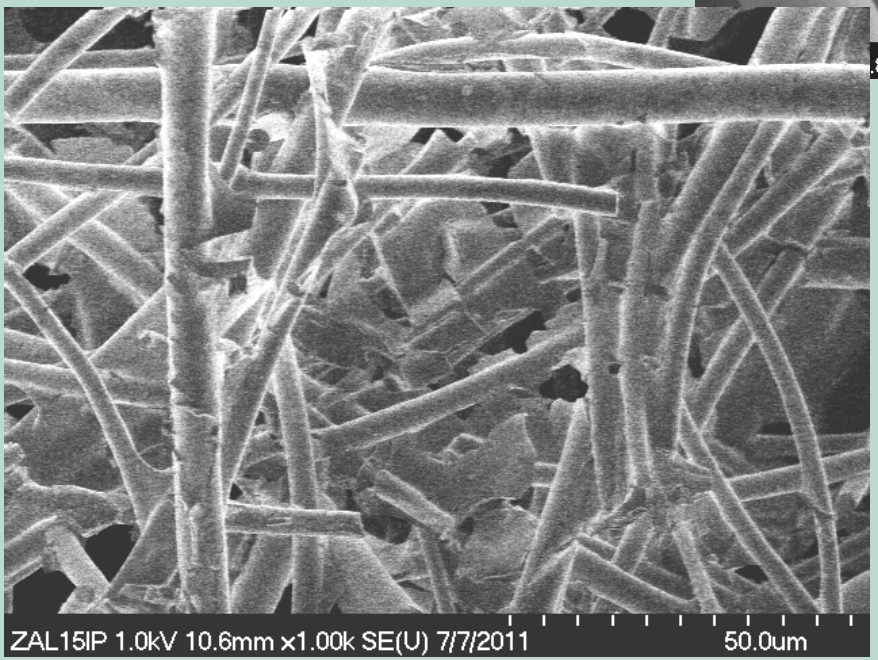


Microstructure:
As-received foams

SiO_2

Microstructure:
As-received foams





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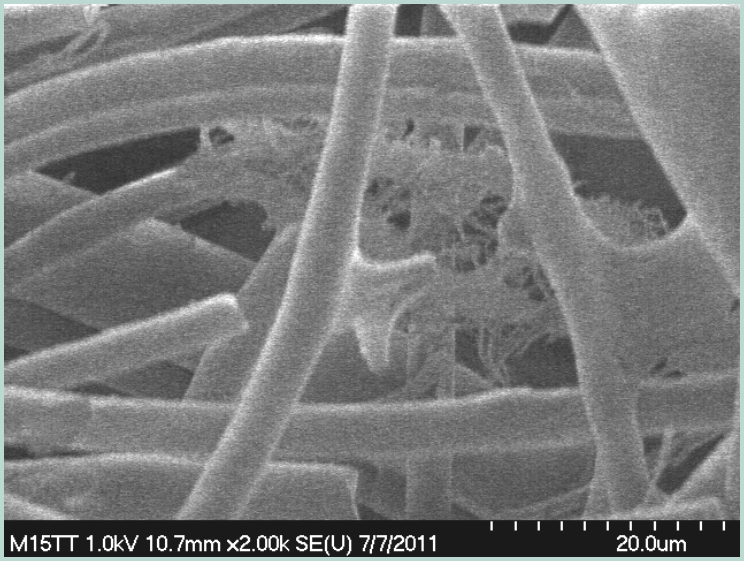
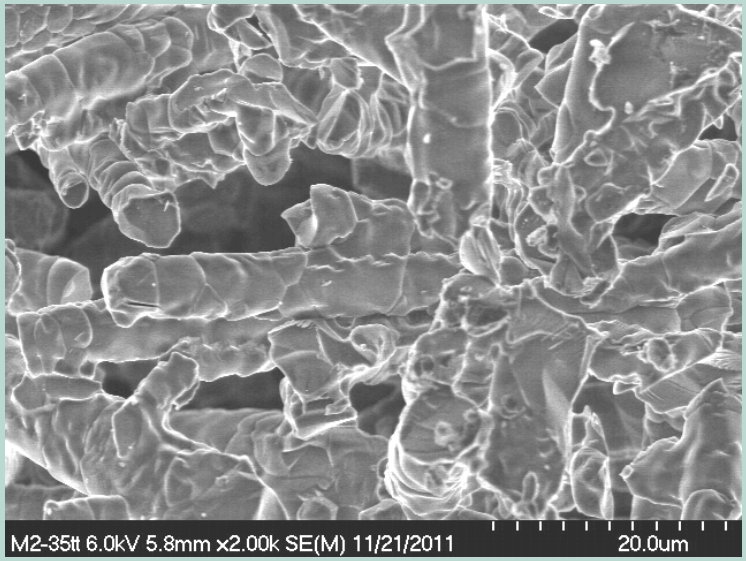
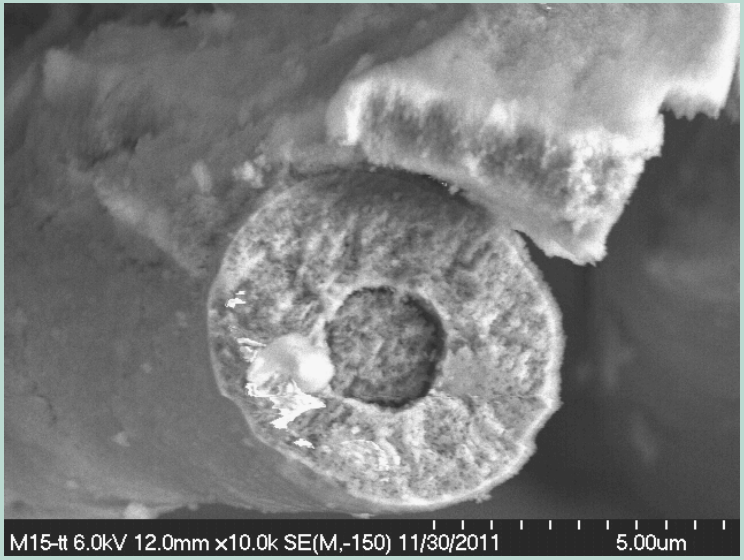
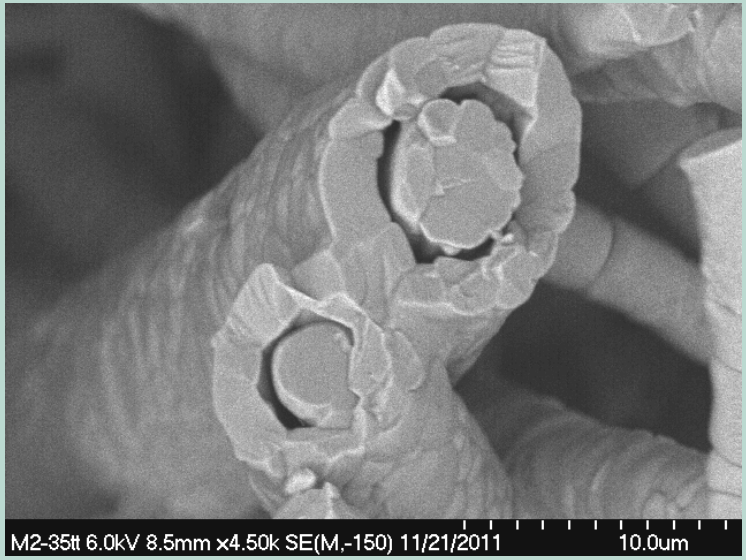






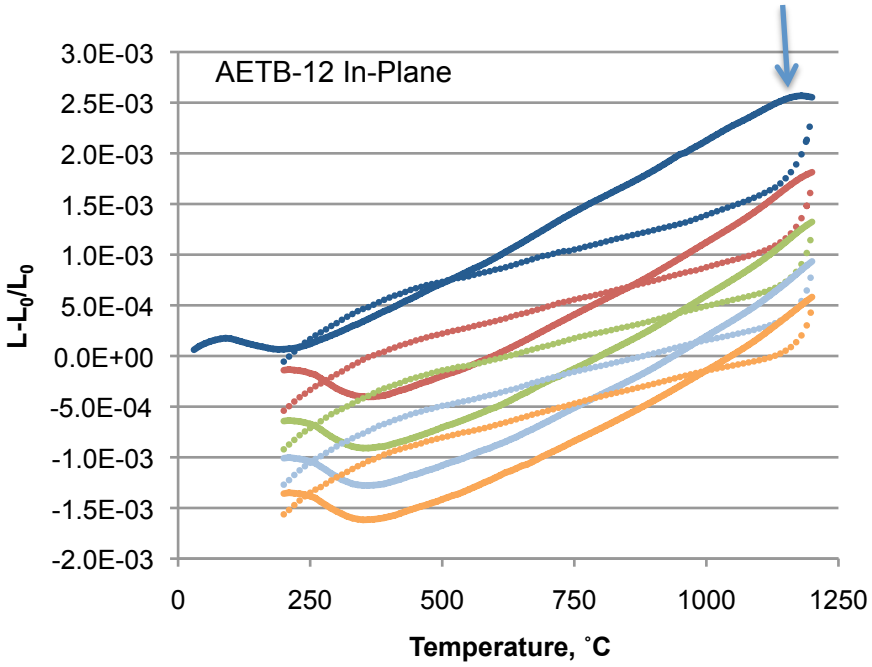


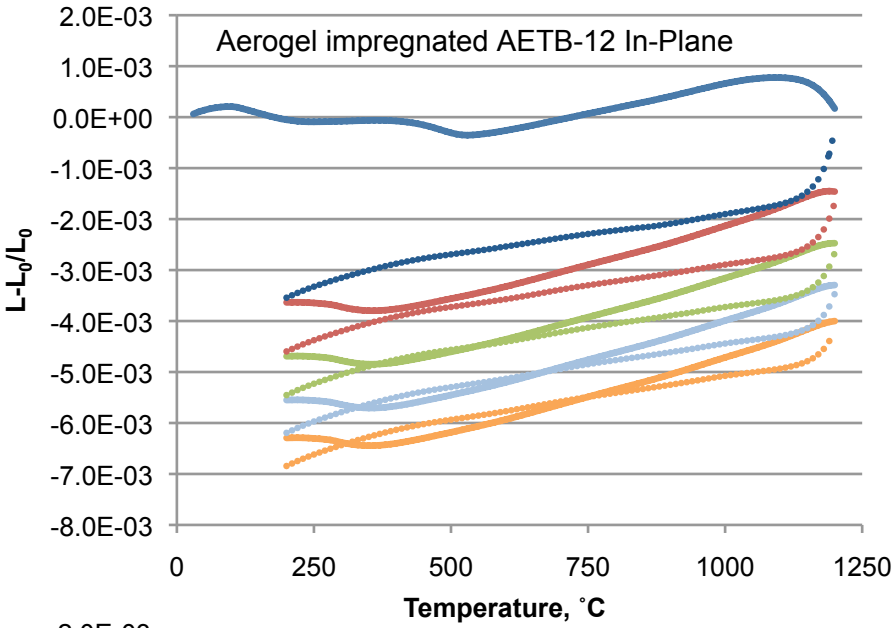
M-15

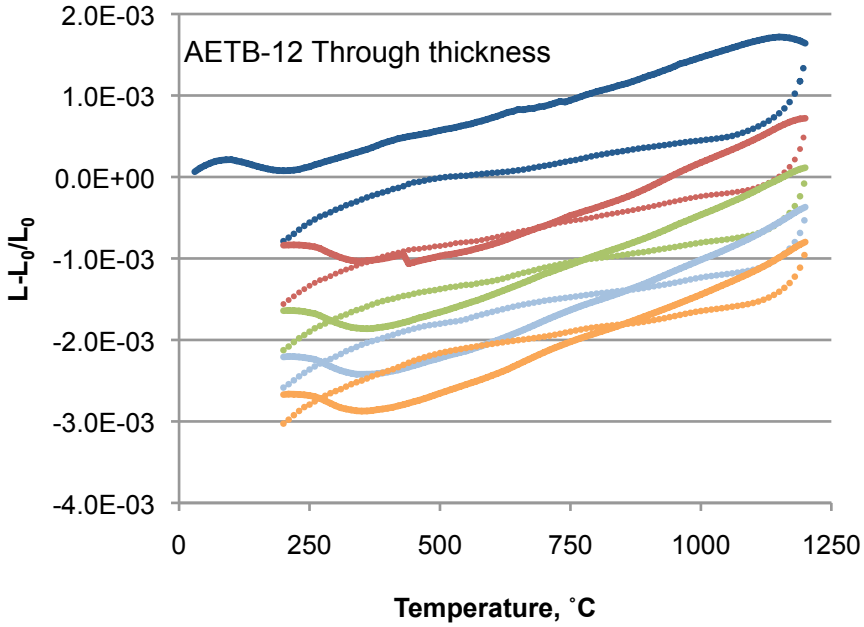
M2-35

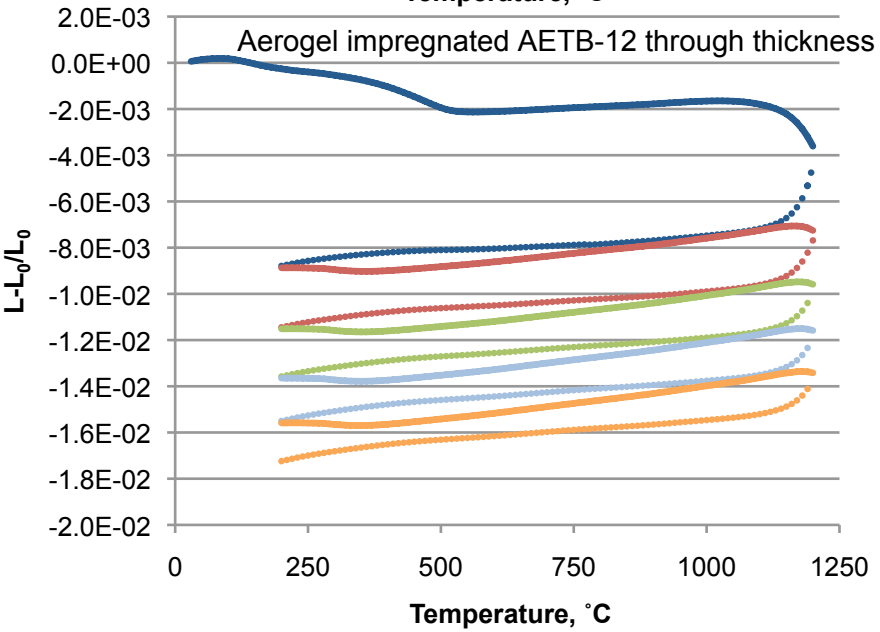
Microstructure: As-received foams

Dilatometry: AETB-12

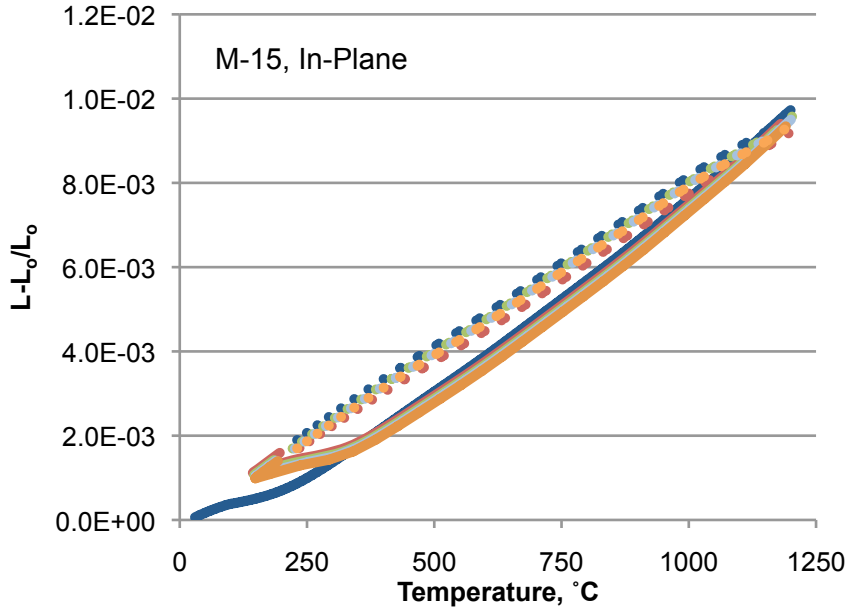


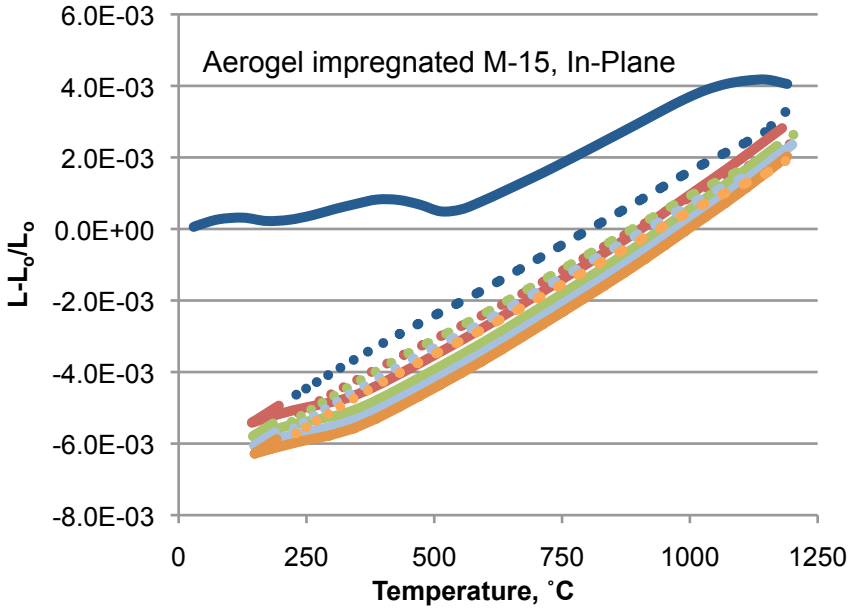


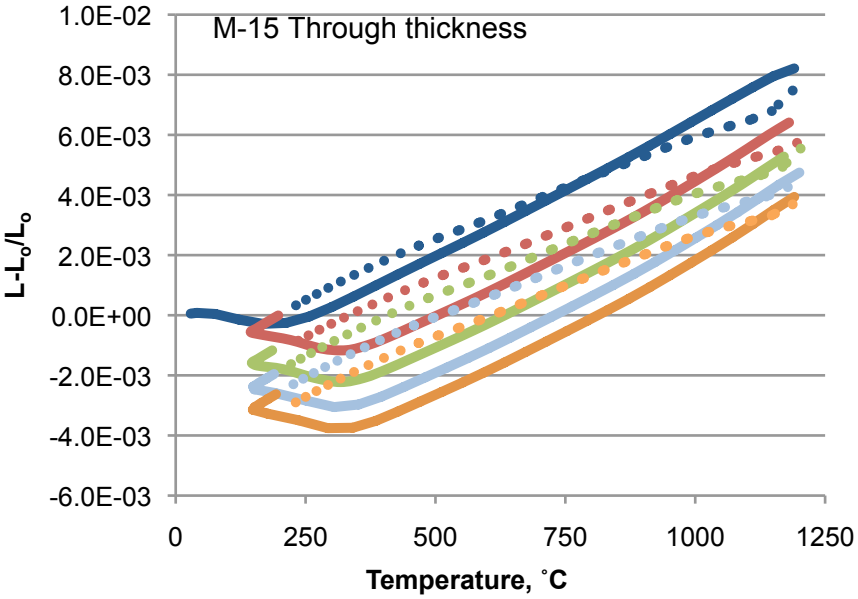


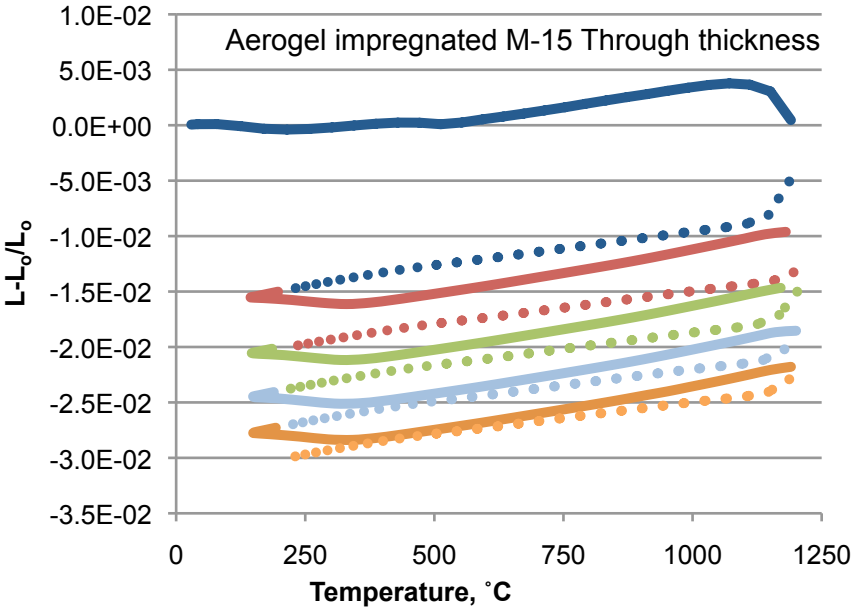


Dilatometry: M-15

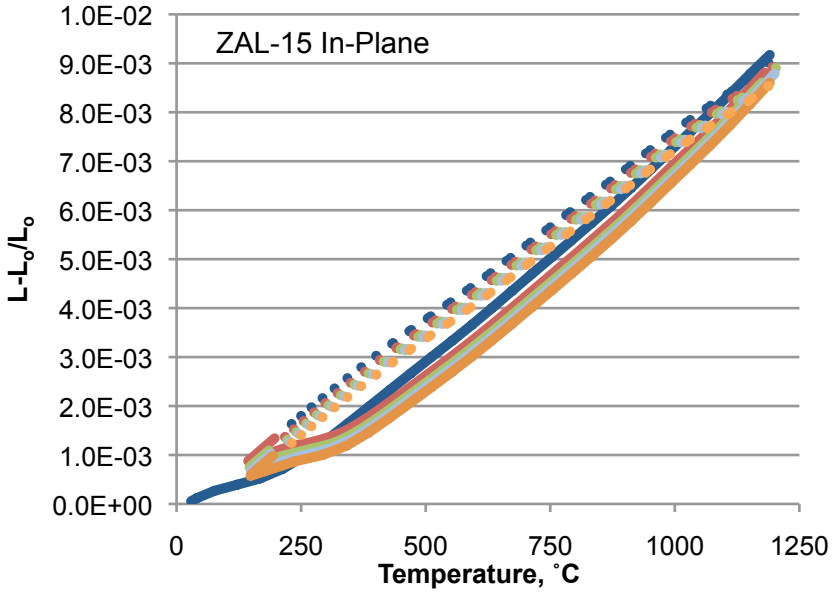


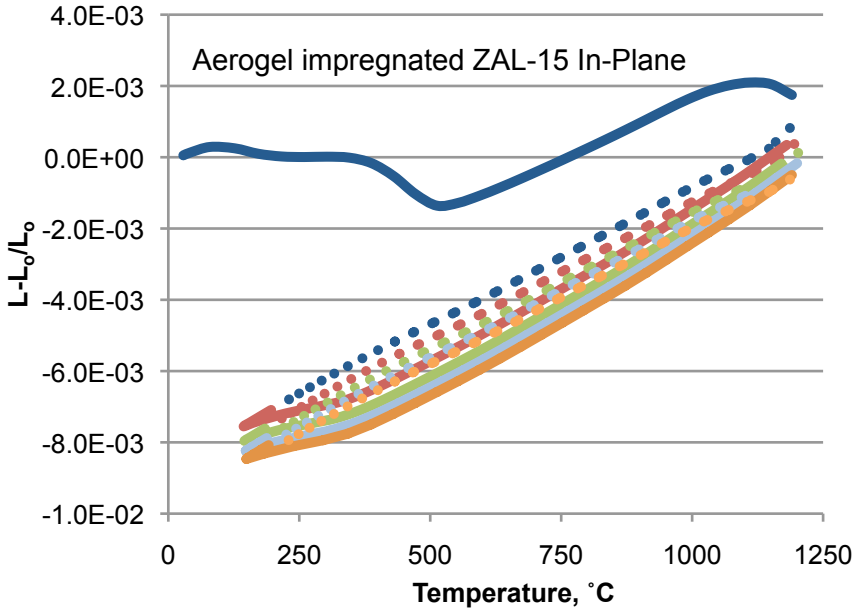


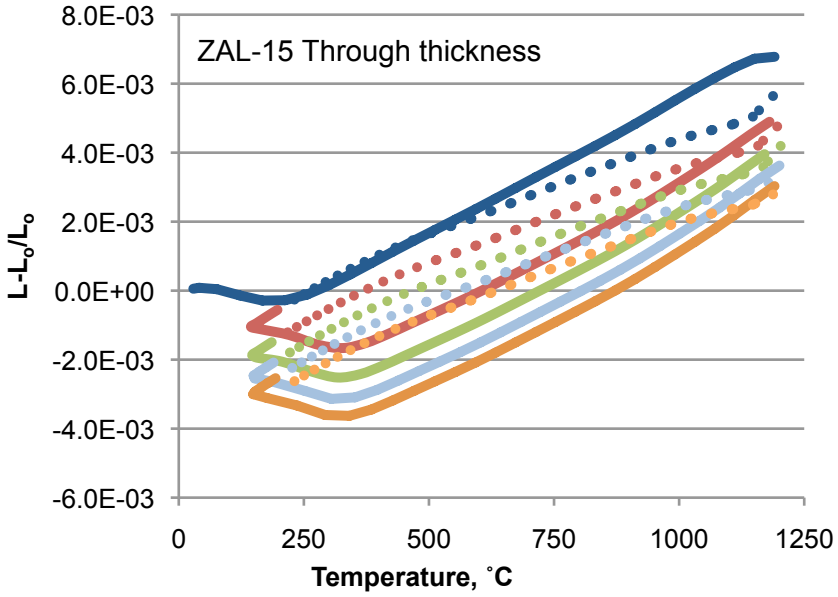


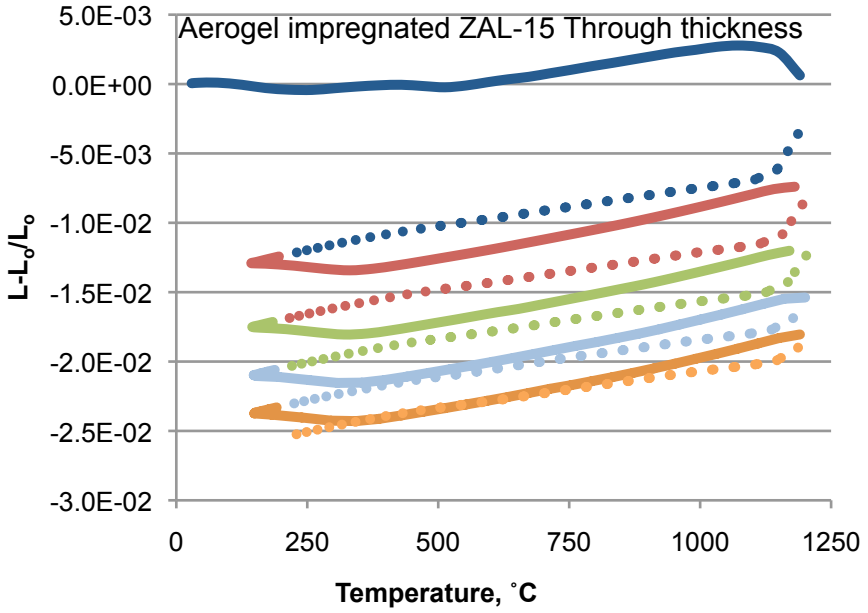


Dilatometry: ZAL-15

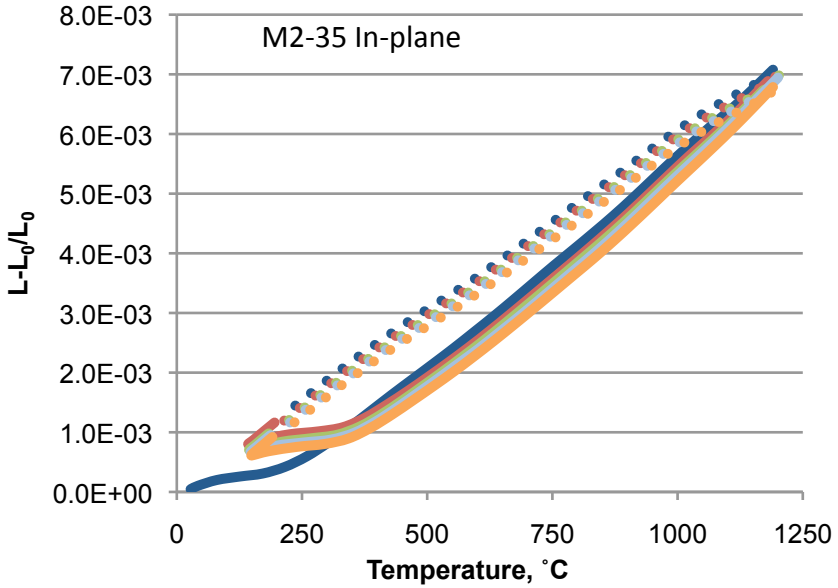


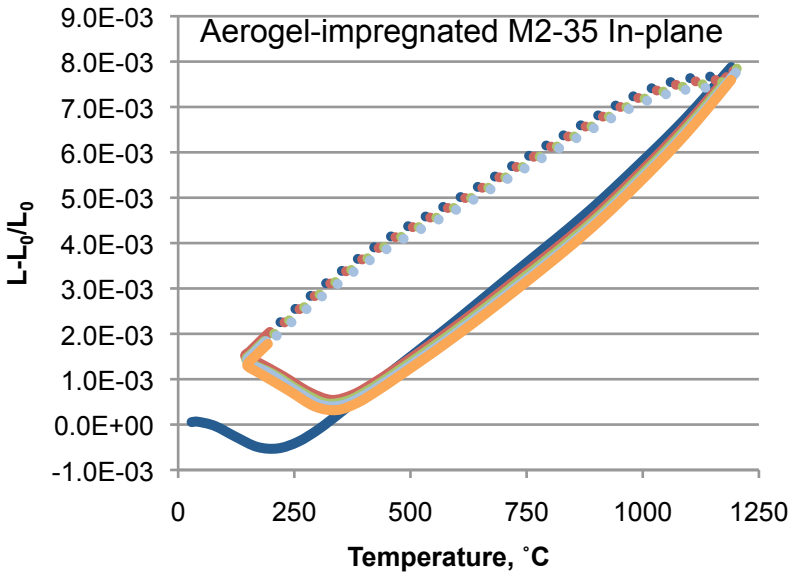


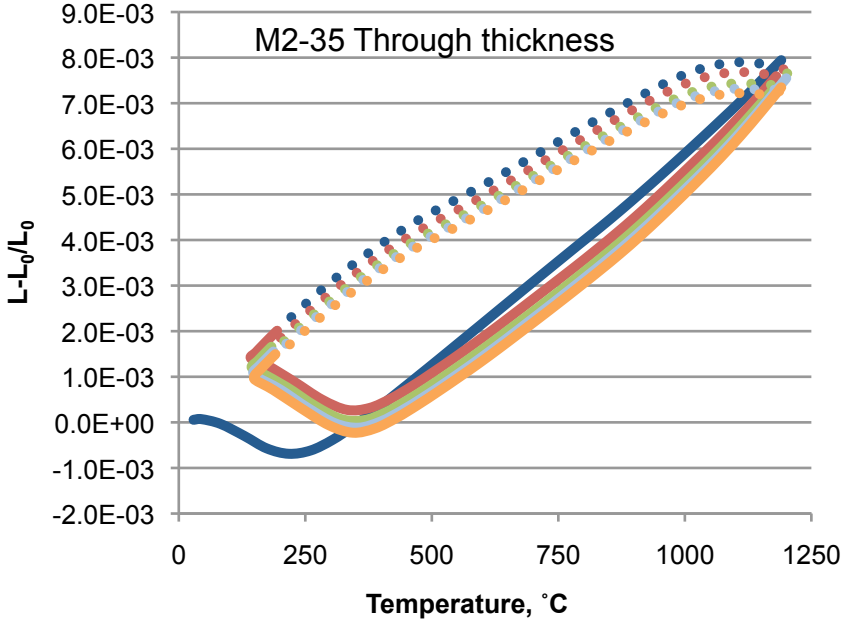


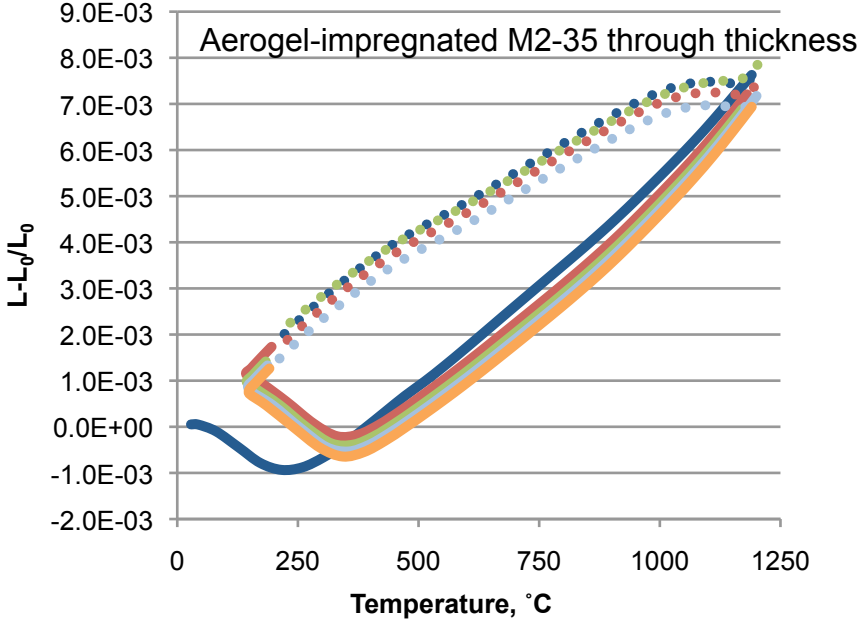


Dilatometry: M2-35

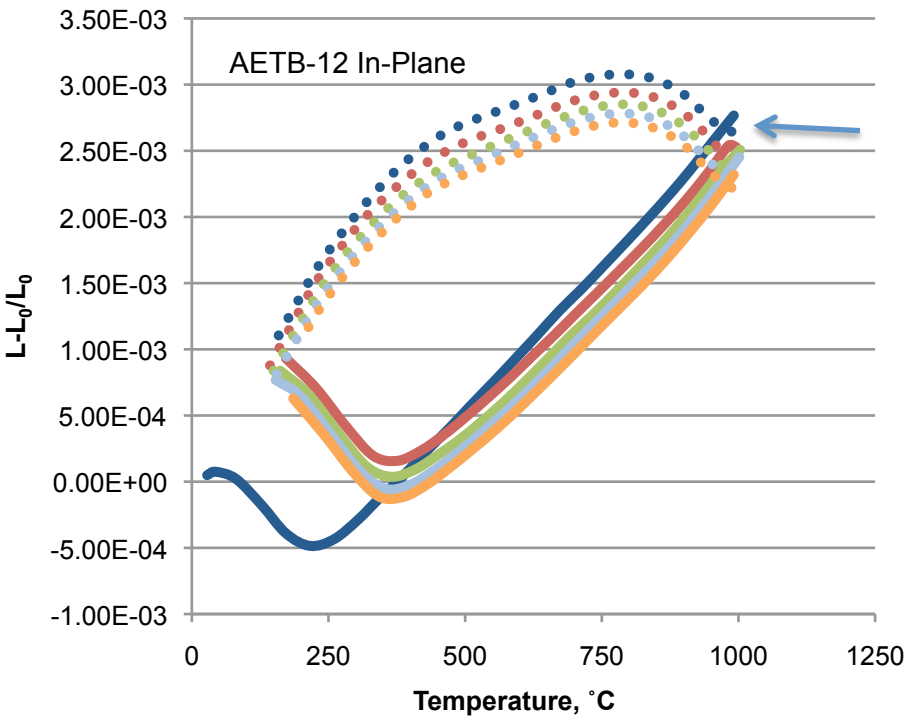


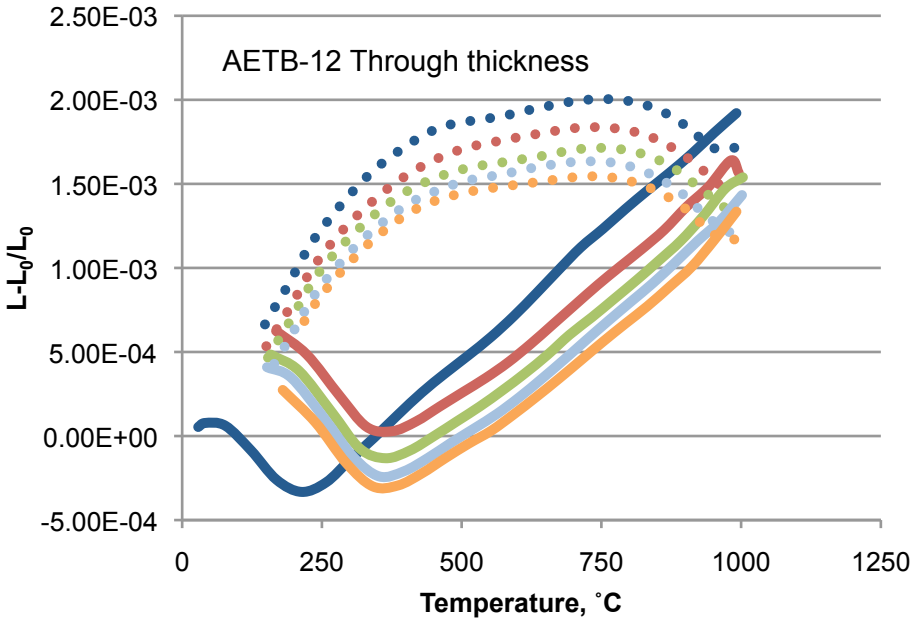




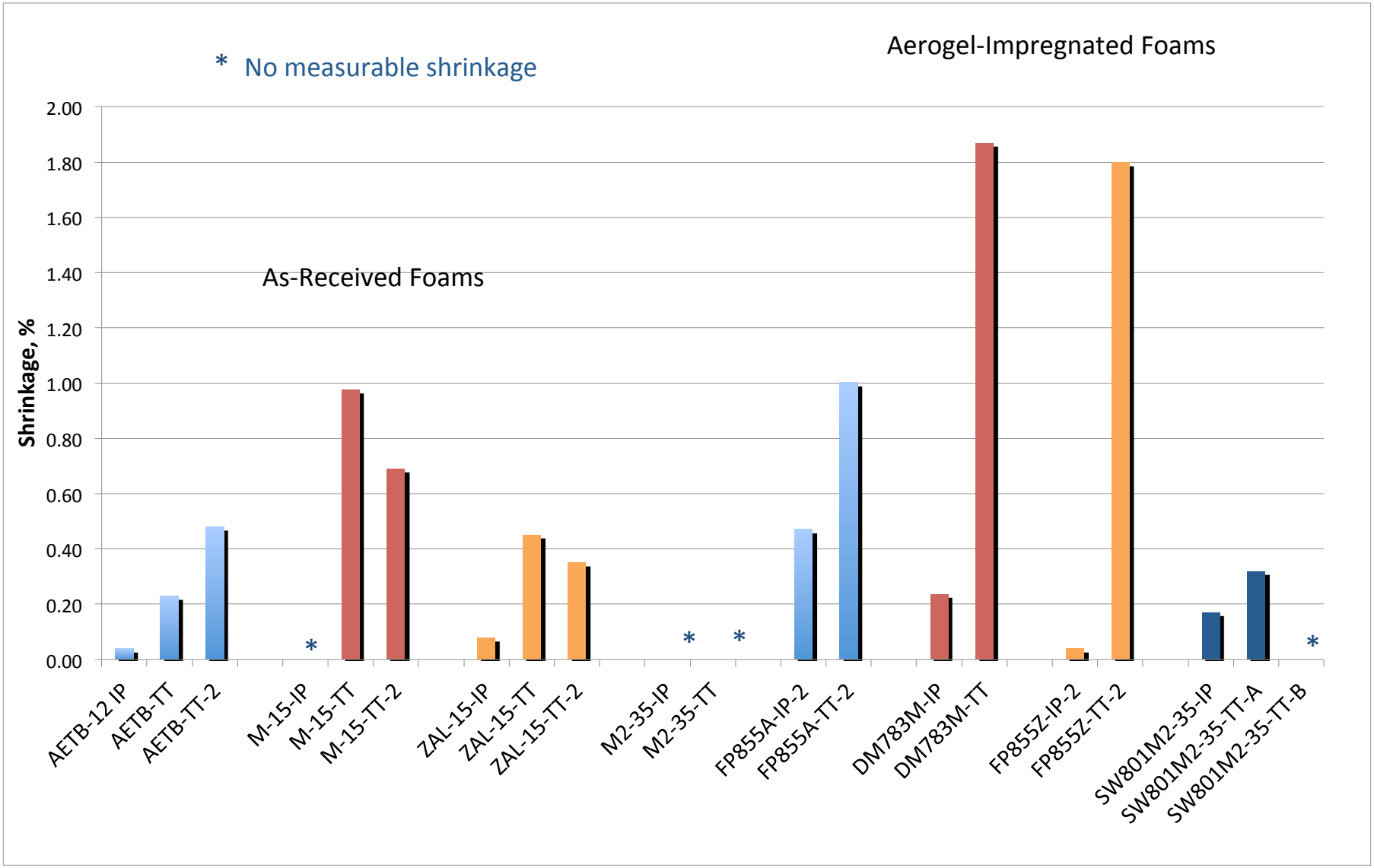


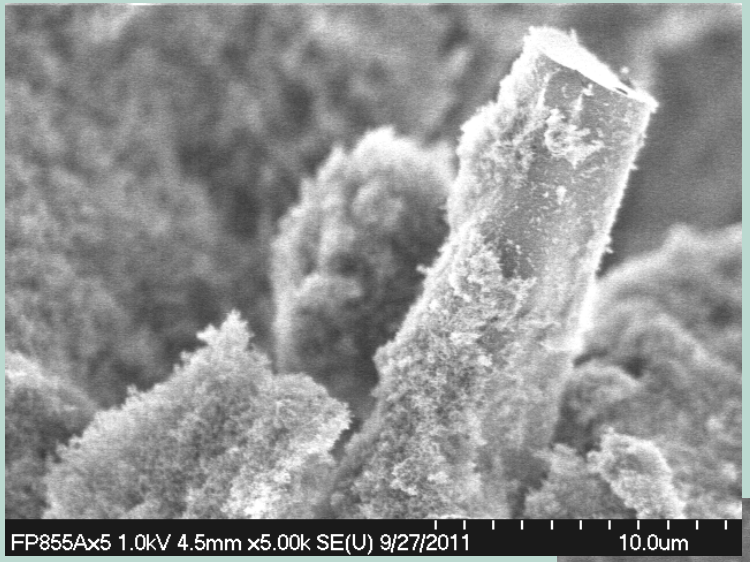
Dilatometry: AETB-12

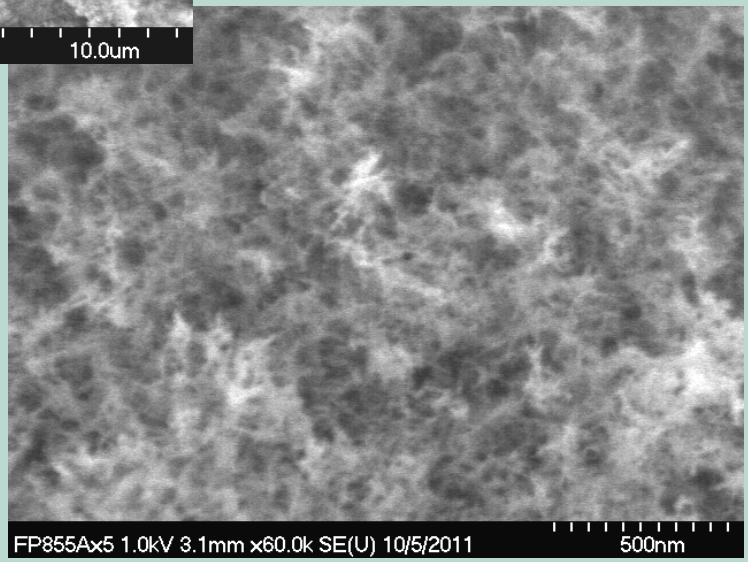




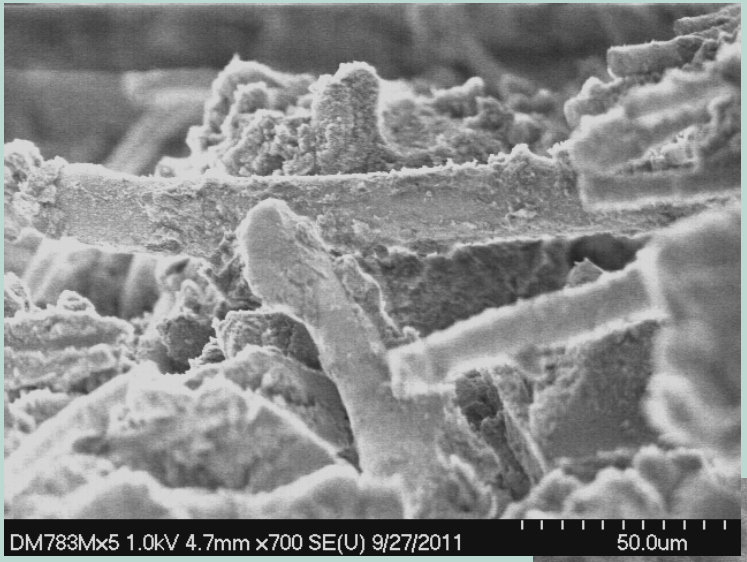
Dilatometry: Dimensional changes after 5 cycles to 1200˚C

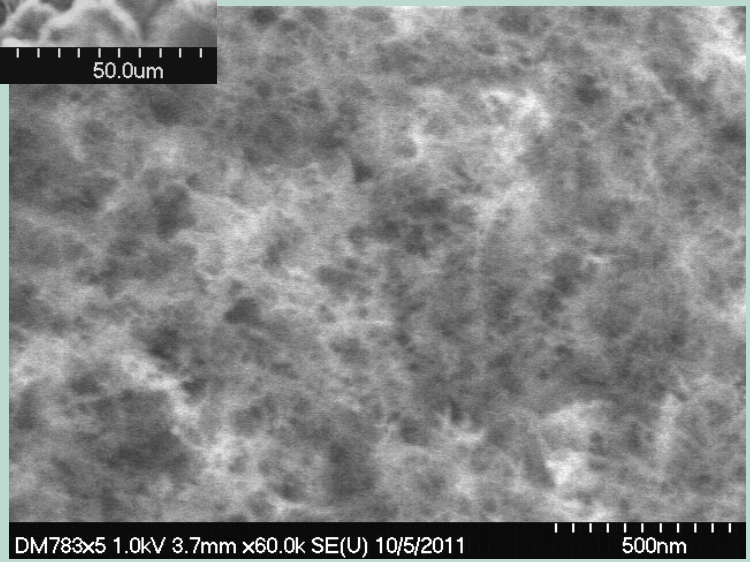




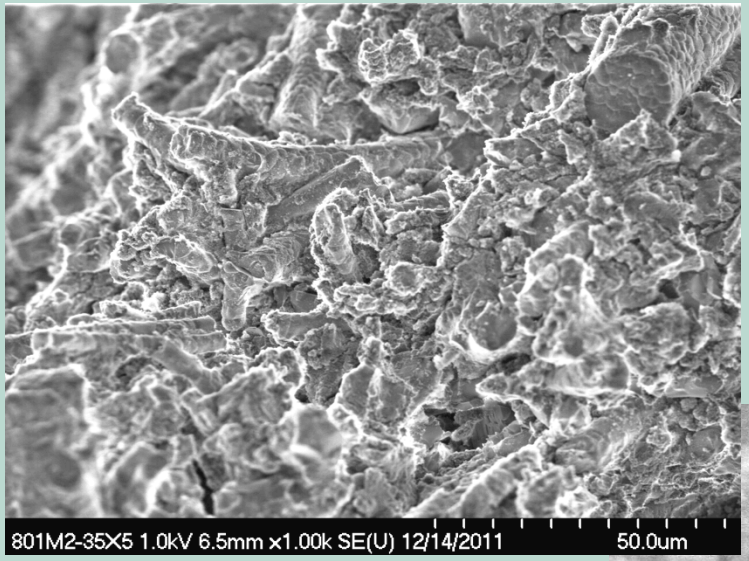


Aerogel-infiltrated AETB-12
Post 5 cycles to 1200˚C

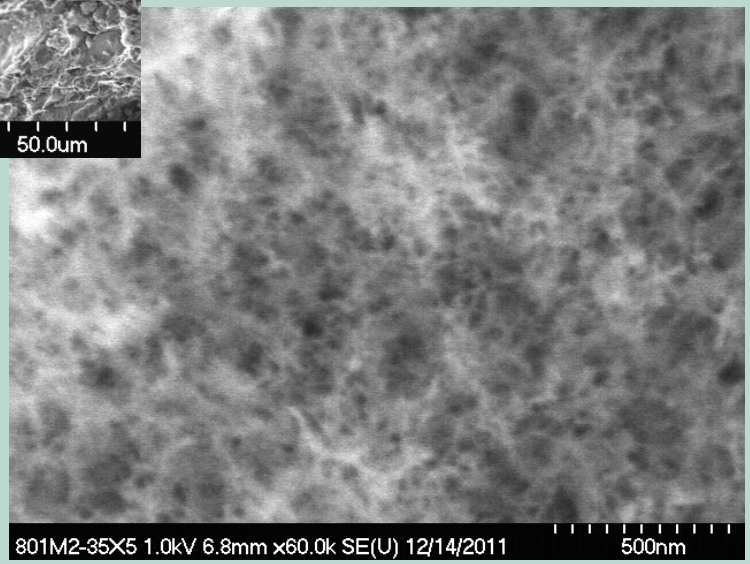




Aerogel-infiltrated M-15
Post 5 cycles to 1200˚C



Aerogel-infiltrated M2-35
Post 5 cycles to 1200˚C



X-ray Diffraction: As-received foams

AETB, as received

Chemical Formula	Compound Name	Crystal System	Ref. Code	SemiQuant [%]
Al2 O3	Aluminum Oxide	Rhombohedral	04-003-5819	53
Al4.44 Si1.56 O9.78	Aluminum Silicate	Orthorhombic	01-074-4143	35
Si O2	Silicon Oxide	Tetragonal	04-008-7636	1
? Al2.5 B0.5 O4.5	Aluminum Boron Oxide	Orthorhombic	04-012-8917	11

Significant glassy phase

M-15, as received

Chemical Formula	Compound Name	Crystal System	Ref. Code	SemiQuant [%]
Al2 O3	Alpha alumina	Rhombohedral	01-088-0826	71
Al4.52 Si1.48 O9.74	mullite	Orthorhombic	01-074-4144	21
Al2 O3	Theta alumina	Monoclinic	01-086-1410	8

ZAL-15, as received

Chemical Formula	Compound Name	Crystal System	Ref. Code	SemiQuant [%]
Al2 O3	Aluminum Oxide	Rhombohedral	01-089-7717	61
Al2 (Al2.544 Si1.456) O9.728	Aluminum Silicon Oxide	Orthorhombic	01-074-8556	18
Al2 O3	Aluminum Oxide	Monoclinic	04-008-4095	12
? B6 O0.787	Boron Oxide	Rhombohedral	01-087-2286	10

Samples after 5 cycles to 1000˚C are very similar, with possible very small SiC phase present in AETB.

X-ray Diffraction: Thermally cycled composites

Aerogel impregnated AETB, 1200˚C x 5

Chemical Formula	Compound Name	Crystal System	Ref. Code	SemiQuant [%]
Al2 O3	alpha alumina	Rhombohedral	01-089-7717	53
Al4.44 Si1.56 O9.78	mullite	Orthorhombic	01-074-4143	40
? Si C	beta SiC	Cubic	01-075-0254	2
?Si O2	cristobalite	Tetragonal	04-005-4875	4

Significant glassy phase (SiO_2)

Aerogel impregnated M-15, 1200˚C x 5

Chemical Formula	Compound Name	Crystal System	Ref. Code	SemiQuant [%]
Al2 O3	alpha alumina	Rhombohedral	01-088-0826	69
Al4.52 Si1.48 O9.74	mullite	Orthorhombic	01-074-4144	21
Al2 O3	Theta alumina	Monoclinic	01-086-1410	10

Aerogel impregnated ZAL-15, 1200˚C x 5

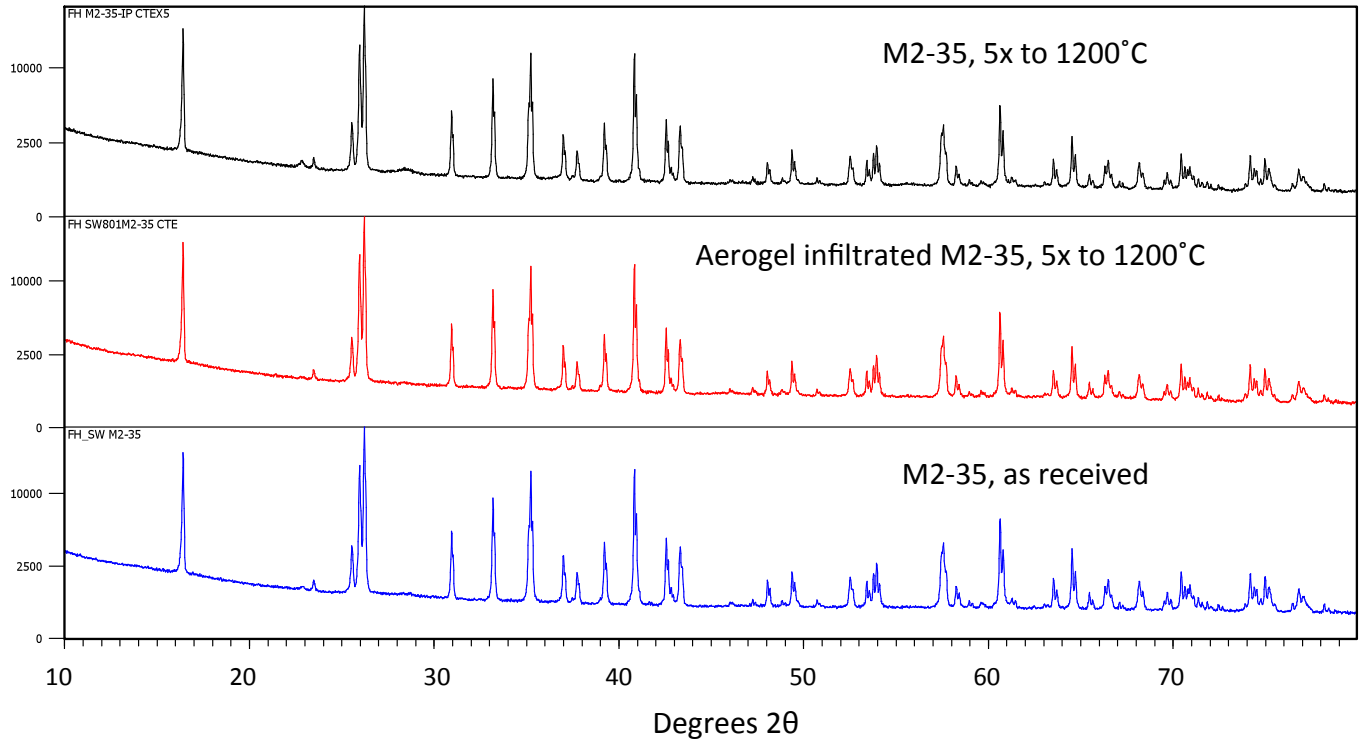
Chemical Formula	Compound Name	Crystal System	Ref. Code	SemiQuant [%]
Al2 O3	alpha alumina	Rhombohedral	01-076-8056	64
Al2 (Al2.556 Si1.444) O9.722	mullite	Orthorhombic	01-074-8552	19
Al2 O3	Theta alumina	Monoclinic	01-086-1410	16
?Si O2	cristobalite	Tetragonal	04-008-7641	1

Note1: there is a small glassy component to this sample.

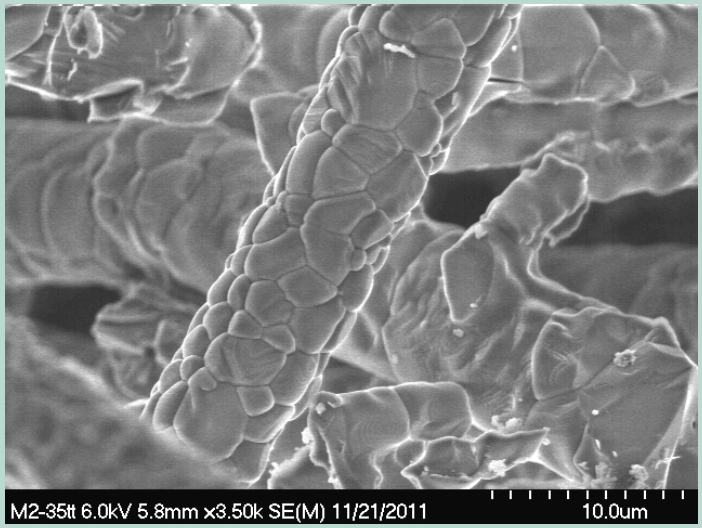
X-ray Diffraction: M2-35

M2-35 As received

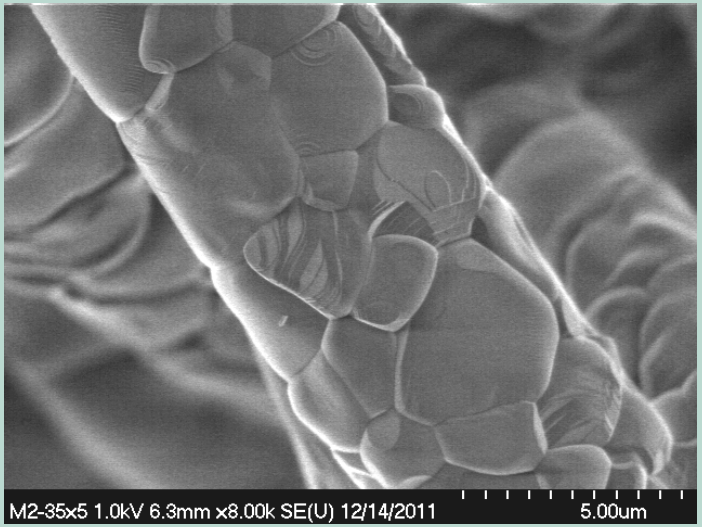
Chemical Formula	Compound Name	Crystal System	Ref. Code	SemiQuant [%]
(Al2.34 Si0.66) O4.83	Aluminum Silicate	Orthorhombic	01-076-2579	84
Al2 O3	Aluminum Oxide	Rhombohedral	01-073-6190	11
?Ni (P4 O11)	Nickel Phosphorus Oxide	Anorthic	01-073-5532	6

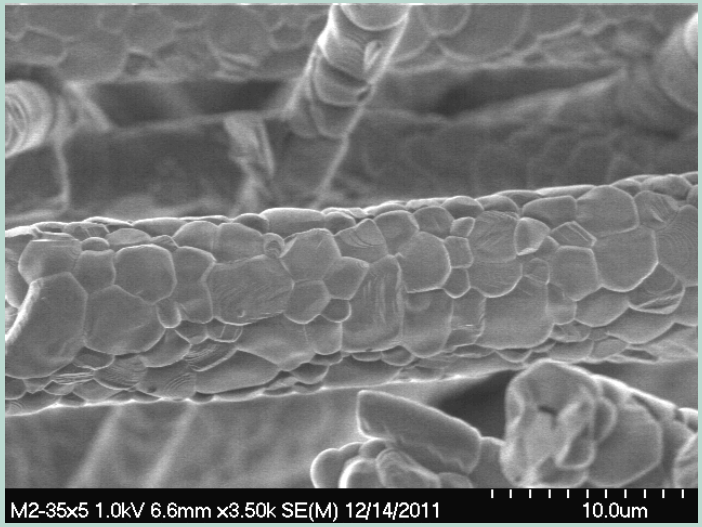


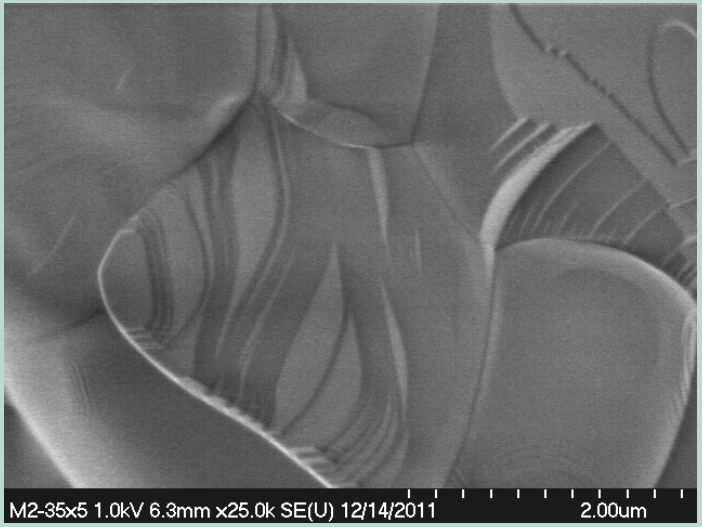
M2-35 microstructure



M2-35 As received







Post CTE 5 cycles 1200˚C

Thermal conductivity and heat capacity measurements of as-received and aerogel impregnated oxide foams in progress.

- Laser flash method being evaluated
- Samples being prepared comparative rod measurements

Ongoing work:
Additional Oxide Foams

Material	Density (g/cc)	Thermal Conductivity (W/m-K)	Specific Heat (J/kg-K)	Al_2O_3	SiO_2	Binder	Source
A-15	0.240	-	1050	97+%	-	Alumina	Zircar Zirconia
M2-15	0.240	-	1050	85%	15%	Mullite	Zircar Zirconia

All Al_2O_3 aerogels

CONCLUSIONS:

- Oxide foams (AETB-12, ZAL-15, M-15) containing *silica binders* and *glassy phases* undergo shrinkage on heating above 1100˚C. Foams continue to shrink with repeated thermal cycling.
- Incorporation of aluminosilicate aerogels exacerbates shrinkage in AETB-12, ZAL-15, M-15, particularly in through thickness dimension.
- Mullite foams (M2-35) offer considerable improvement in dimensional stability, including samples incorporating aluminosilicate aerogels. Commercially available M2-35 carries a weight penalty; however, trial fabrication of lower density M2-15, and a lower density all Al_2O_3 foam, is in progress.
- Thermal conductivity measurements of the foams, with and without aluminosilicate incorporation, are underway.
- A Boehmite-derived, all Al_2O_3 aerogel, will be compared with aluminosilicates.

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