SiC/SiC Composites: The Effect of Fiber Type and Fiber Architecture on Mechanical Properties

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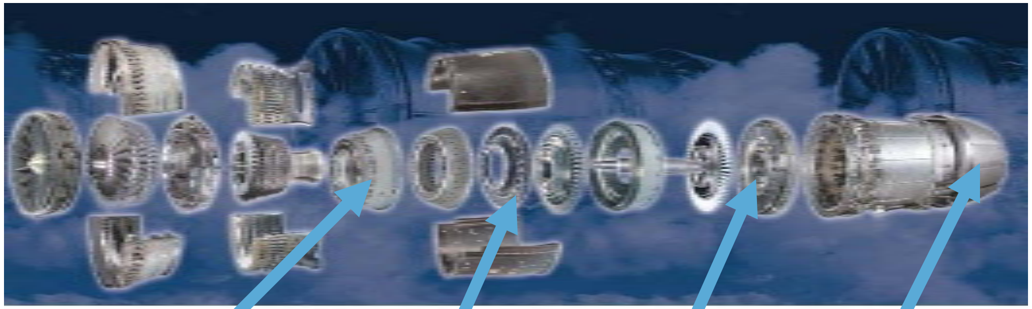
Abstract

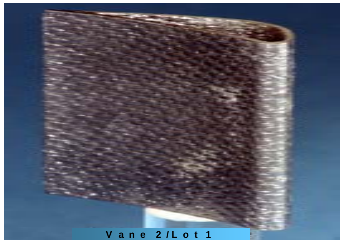
Woven SiC/SiC composites represent a broad family of composites with a broad range of properties which are of interest for many energy-based and aero-based applications. Two important features of SiC/SiC composites which one must consider are the reinforcing fibers themselves and the fiber-architecture they are formed into. The range of choices for these two features can result in a wide range of elastic, mechanical, thermal, and electrical properties. In this presentation, it will be demonstrated how the effect of fiber-type and fiber architecture effects the important property of “matrix cracking stress” for slurry-cast melt-infiltrated SiC matrix composites, which is often considered to be a critical design parameter for this system of composites.

CMC Potential Applications

- Aero hot-section parts
- Hypersonic TPS and control structures
- Auto and land-based gas turbine components
- Nuclear containment for future generation reactors

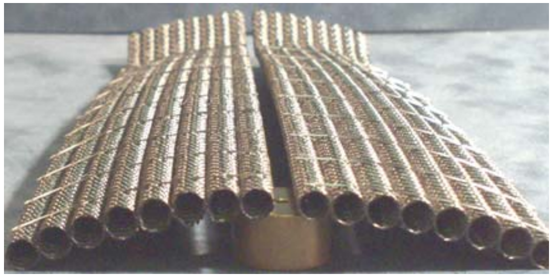


Combustor liner *Vanes* *Blades* *Flaps and Seals*



Inlet Turbine Vane

Rocket nozzles



Courtesy of David Marshall, Teledyne

Critical Issues for Composite Designer

- **The range of composites available**
  - Fiber-type
  - Fiber architecture
  - Interphase
  - Matrix
- Cost
- Performance
  - Models
  - Property database
  - Reliability
- Manufacturability

Therefore, it is essential that constituent-based performance relationships are established so that the composite designer can weigh cost vs performance vs manufacturability issues and capabilities for the range of composites available.

There is much to be done. However, much is known which should serve as a good starting point for future work.

Outline

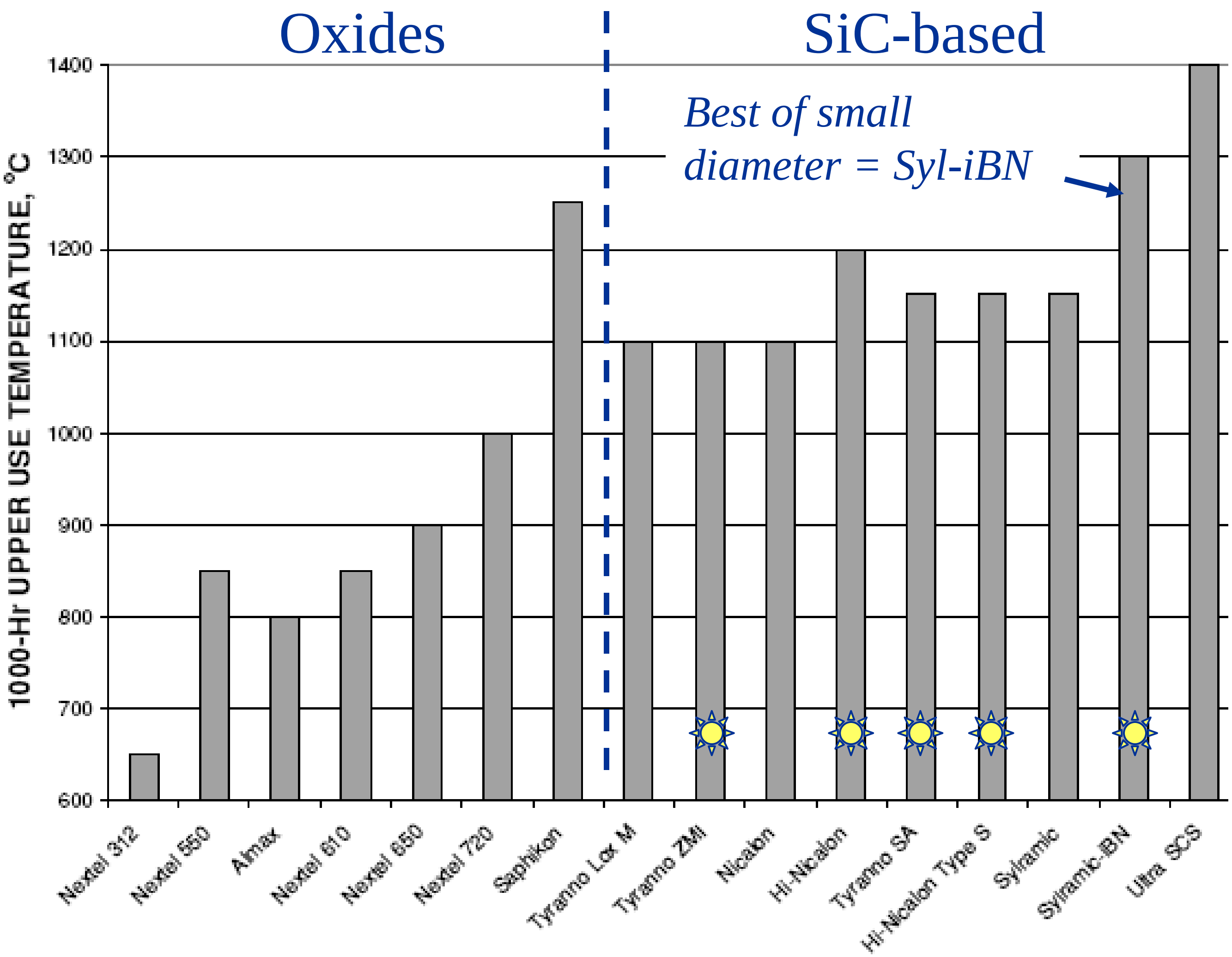
- The effect of fiber-type on woven composite mechanical properties (Slurry Cast Melt Infiltrated Matrix)
  – As the fiber goes, so goes the composite
- Fiber architectures that enable
  – Understanding the effect of fiber architecture in order to fabricate the best combination of composite properties
- Issues, Implications and Conclusions

The Effect of Fiber-Type on 2D Woven Melt-Infiltrated SiC-matrix Composites

Based on IGTI publications in 2004 and 2007 and a paper in process with *International Journal of Applied Ceramic Technology* (V. Pujar coauthor)

Fiber Comparison

1000 hr Use Temperature (σ_f = 500 MPa)



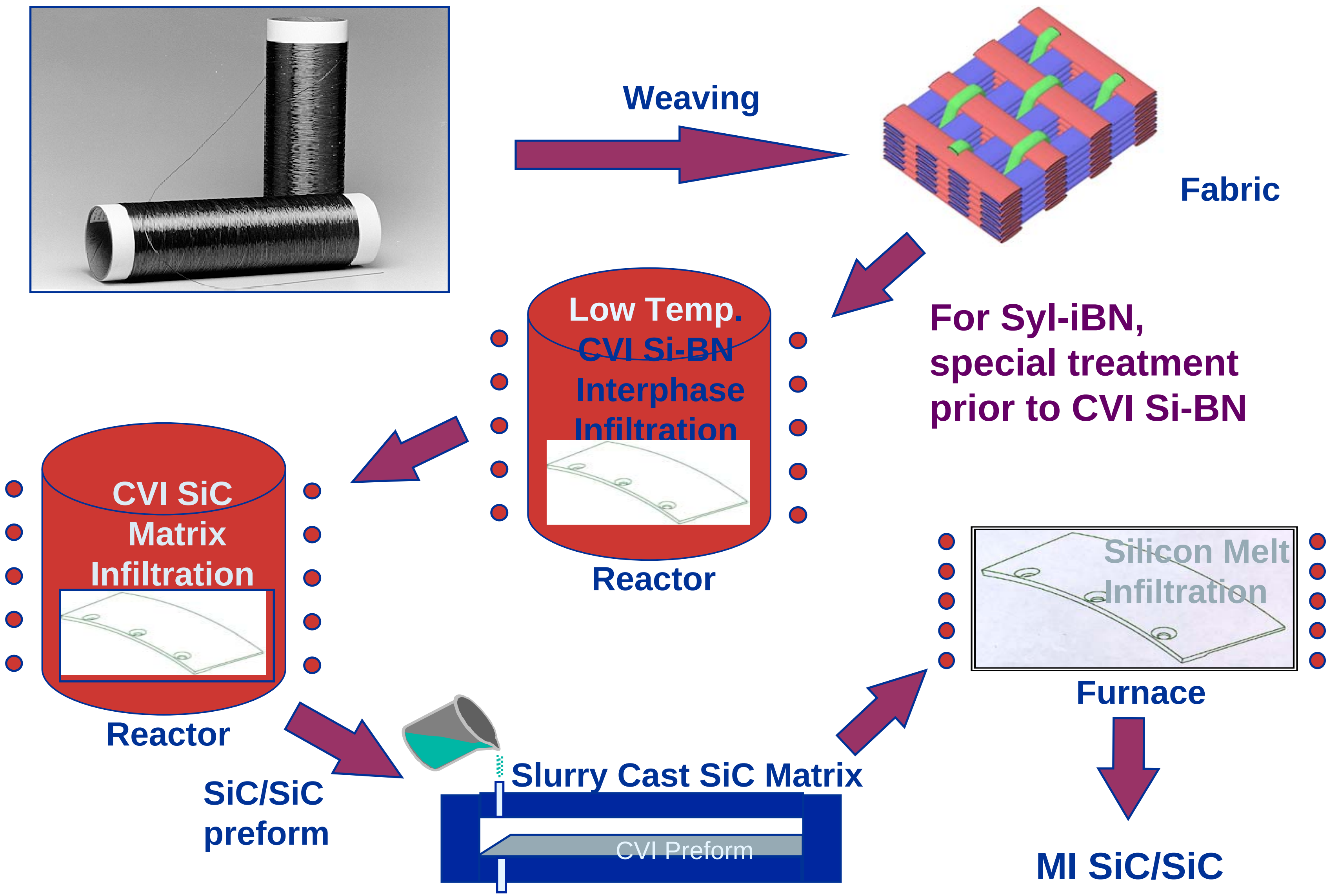
Sylramic-iBN:

Polycrystalline B-containing SiC fiber (Sylramic, processed by COIC) subjected to post-process nitrogen containing heat treatment at high temperature (> 1700^{o}C).

Removes B and improves creep-rupture properties

From, J.A. DiCarlo and H.M. Yun, Handbook of Ceramic Composites, Chapter 2 (Kluwer: NY, 2005)

Standard Slurry Cast Melt-Infiltrated (MI) 2D&3D Woven Composites (GEPSC, Newark Delaware)



2D Woven MI SiC/SiC Composites Evaluated

Panel	Fiber-type	Avg fiber radius, μm	# of fibers per tow	epcm	Avg specimen thickness, mm	Average f [# specimens] (scatter)	Average f_{BN}*	Average $f_{CVI\ SiC}$*
SYLiBN-1 (223)	Sylramic-iBN	5	800	7.9	2.26 [11] (+0.07/-0.19)	0.352 [11] (+0.014/-0.004)	0.114	0.286
SYLiBN-2 (224)	Sylramic-iBN	5	800	7.9	2.05 [10] (+0.14/-0.12)	0.386 [10] (+0.026/-0.022)	0.157	0.287
SYLiBN-3 (226)	Sylramic-iBN	5	800	7.9	1.93 [10] ± 0.09	0.410 [10] (+0.02/-0.018)	0.134	0.270
SA-1 (243)	Tyranno SA3	5	800	7.1	2.05 [7] (+0.06/-0.12)	0.348 [7] (+0.02/-0.01)	0.120	0.281
SA-2 (244)	Tyranno SA3	5	800	7.1	1.97 [5] (+0.04/-0.05)	0.362 [5] (±0.008)	0.126	0.281
SA-3 (246)	Tyranno SA3	5	800	7.1	2.15 [10] (+0.05/-0.08)	0.332 [10] (+0.006/-0.004)	0.098	0.274
HN (94)	Hi-Nicalon	6.85	500	7.1	3.05 [7] (+0.11/-0.13)	0.274 [7] (+0.012/-0.01)	0.039	0.227
Z-1 (132)	Tyranno ZMI	5.5	800	8.7	3.75 [9] ±0.06	0.281 [9] (+0.004/-0.006)	0.082	0.227
Z-2 (137)	Tyranno ZMI	5.5	800	8.7	3.62 [4] (+0.12/-0.14)	0.292 [4] (+0.01/-0.01)	0.072	0.198
HNS-1 [6]	Hi-Nicalon S	6.5	500	7.1	2.49 [7] (+0.04/-0.09)	0.302 [9] (+0.012/-0.004)	0.04	0.25
HNS-2 [6]	Hi-Nicalon S	6.5	500	7.1	2.17 [9] (+0.08/-0.12)	0.348 [9] (+0.020/-0.018)	0.04	0.21

All fiber fractions related to architecture and thickness

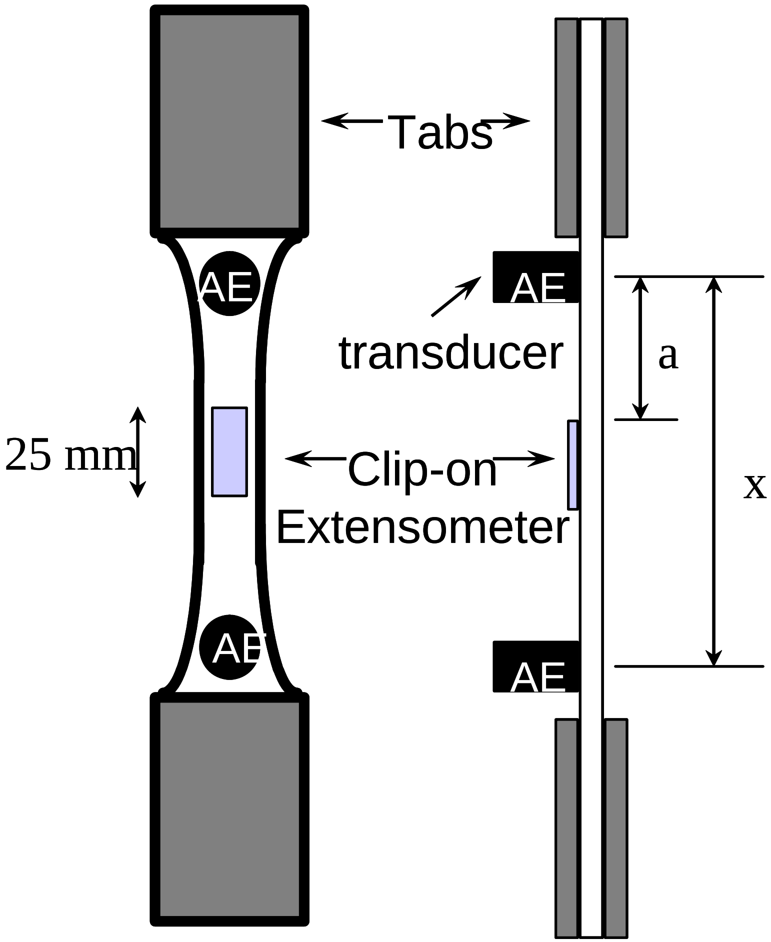
$$f = 2^*(N_{ply}\ N_f)\ (epcm/10)\ (\pi R_f^2) / t$$

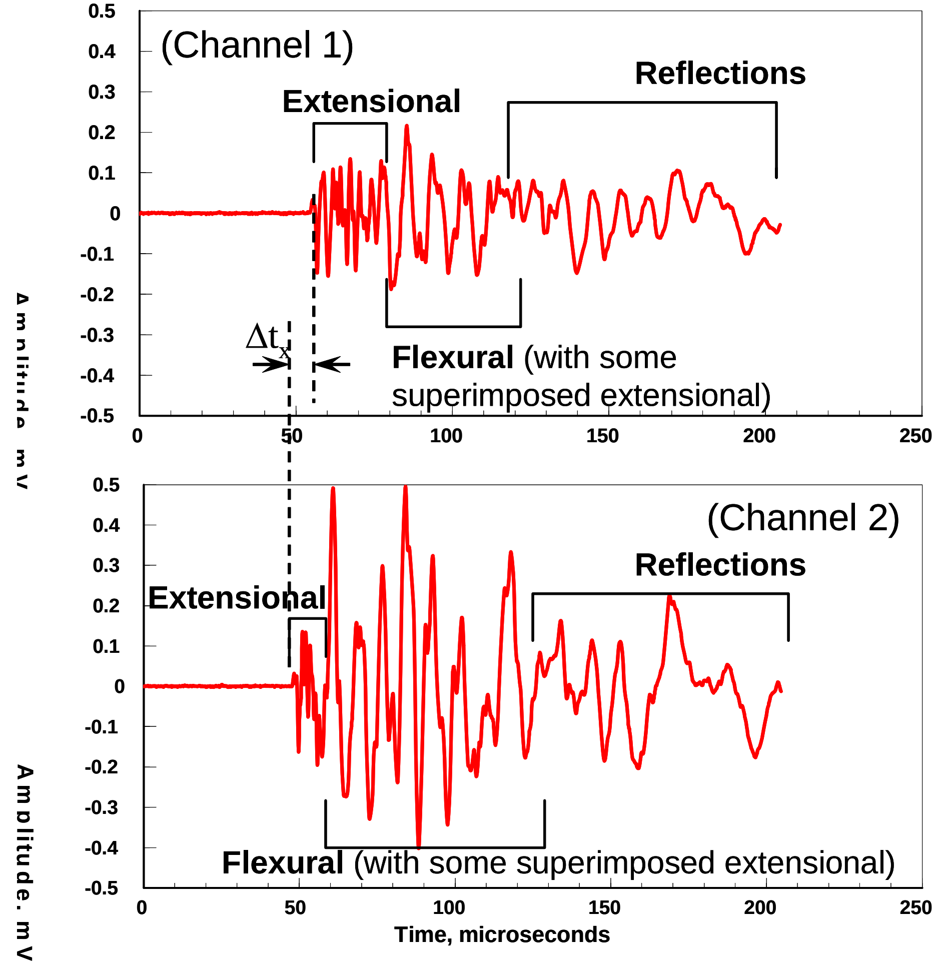
2D Woven MI SiC/SiC Composites: Properties

Panel	Avg. E, GPa [#RT spec] (scatter)	Avg. UTS, MPa [# specimens] (scatter)	Avg. ε, % [# specimens] (scatter)	Avg. Stress on Fibers, GPa [#RT spec] (scatter)	0.005% Offset Stress, MPa	1st AE Event Stress, MPa	1st Loud AE Event Stress, MPa	AE Onset Stress, MPa	Residual stress, MPa
SYLiBN-1 (223)	247 [3] (+0.007/-0.006)	361 [3] (+36/-32)	0.35 [3] (+0.04/-0.06)	1997 [2] (+ 79/-143)	194 [3] (+ 6/- 9)	150 [2] ± 3	170 [2] ± 2	192 [2] ± 2	-60 [3] ± 7
SYLiBN-2 (224)	271 [2] (± 12)	465 [2] ± 37	0.47 [2] ± 0.03	2368 [2] ± 75	181 [2] ± 4	131 [2] ± 1	142 [2] ± 12	189 [2] ± 16	-60 [2] ± 10
SYLiBN-3 (226)	238 [1]	444 [1]	0.45 [1]	2210 [1]	176 [1]	113 [1]	155 [1]	155 [1]	-45 [1]
SA-1 (243)	254 [1]	358 [1]	0.33 [1]	2000 [1]	152 [1]	117 [1]	141 [1]	145 [1]	-20 [1]
SA-2 (244)	236 [1]	372 [1]	0.34 [1]	2047 [1]	178 [1]	117 [1]	117 [1]	138 [1]	-15 [1]
SA-3 (246)	230 [1]	334 [1]	0.30 [1]	1978 [1]	178 [1]	113 [1]	125 [1]	135 [1]	-30 [1]
HN (94)	244 [7] (+43/-31)	311 [7] (+17/-10)	0.79 [7] (+0.12/-0.04)	2272 [7] (+208/-141)	126 [6] (+4/-5)	85 [6] (+5/-8)	109 [6] (+9/-5)	114 [6] (+12/-8)	-4 [6] (+7/-8)
Z-1 (132)	213 [4] (+ 5/-3)	279 [3] (+ 9/- 6)	0.95 [3] (+0.04/-0.03)	1973 [4] (+66/-35)	111 [4] (+7/-6)	60 [4] (+17/-18)	67 [4] (+14/-16)	85 [4] (+10/-15)	+12 [4] (+5/-9)
Z-2 (137)	202 [4] (+ 5/- 3)	261 [4] (+12/- 6)	0.83 [4] (+0.02/-.0.03)	1794 [4] (+49/-53)	107 [4] (+ 5/- 4)	64 [4] (+11/-9)	74 [4] (+18/-13)	83 [4] (+11/-14)	+12 [4] (+8/-7)
HNS-1 [6]	262 [1]	341 [1]	0.63 [1]	2278 [1]	154 [1]	80	134	150	-20
HNS-2 [6]	232 [1]	412 [1]	0.60 [1]	2245 [1]	147 [1]	85	115	135	-20

Focus on matrix cracking strength: strength-reduction due to oxidation ingress (interphase and fiber/matrix oxidation resulting in strong bonding of fibers)

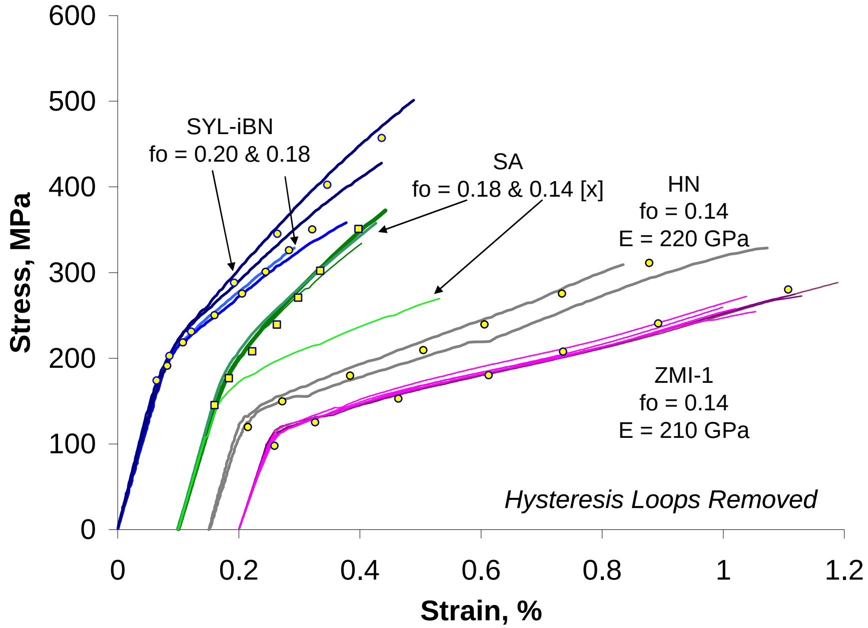
Modal Acoustic Emission of CMCs

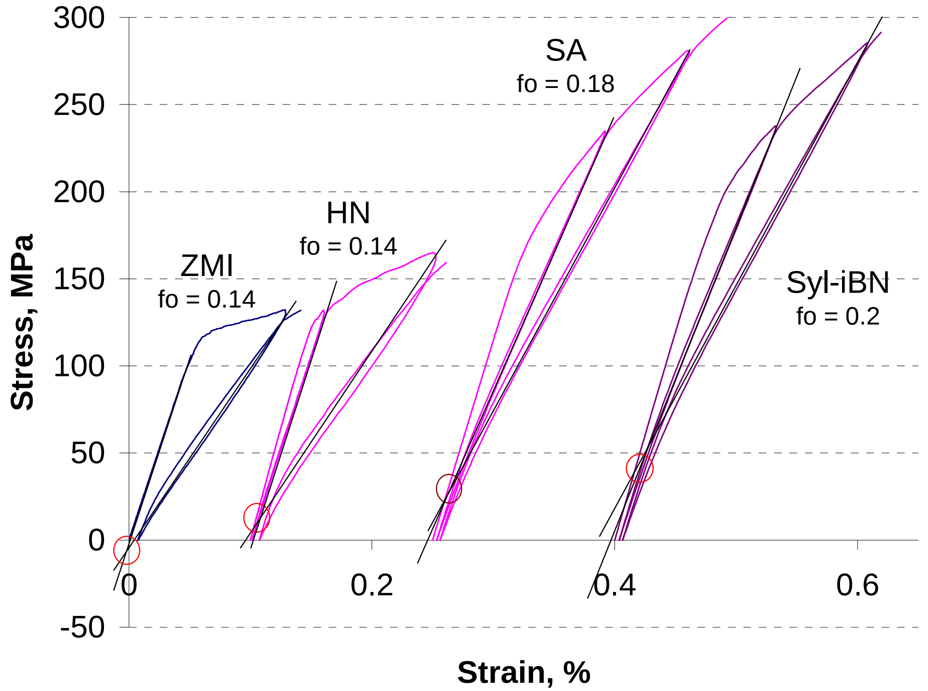




- ***Locate damage events and failure events → Δt***
- ***Monitor stress(or time)-dependent matrix cracking → Cumulative AE Energy***
- Identify damage sources, e.g. matrix cracks, fiber breaks → Frequency
- Measure stress(or time) dependent Elastic Modulus → Speed of sound

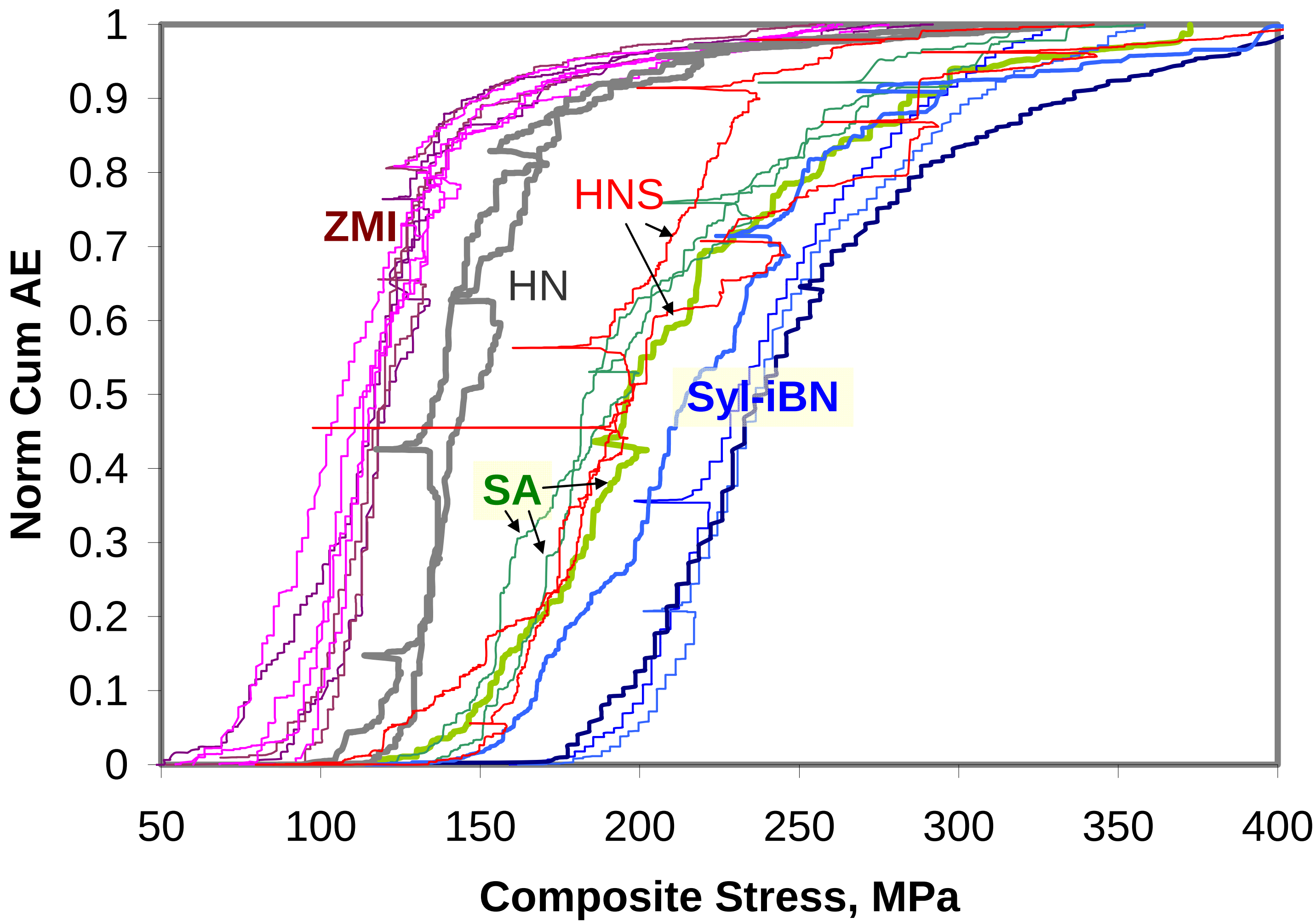
Room Temperature Stress Strain Behavior



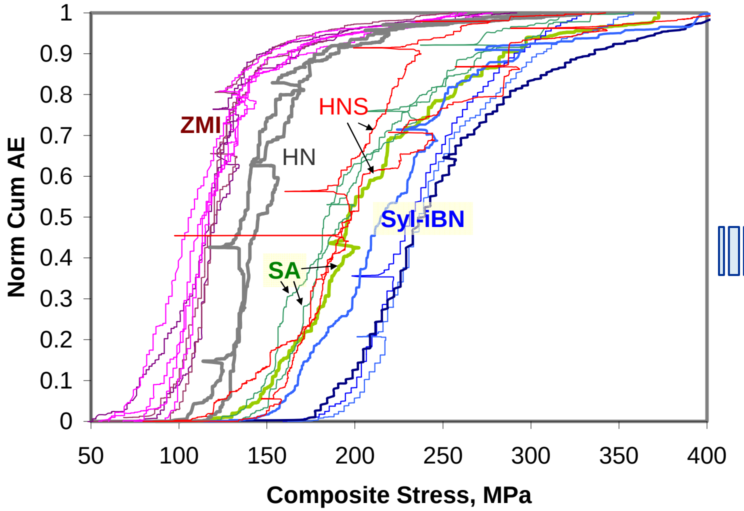


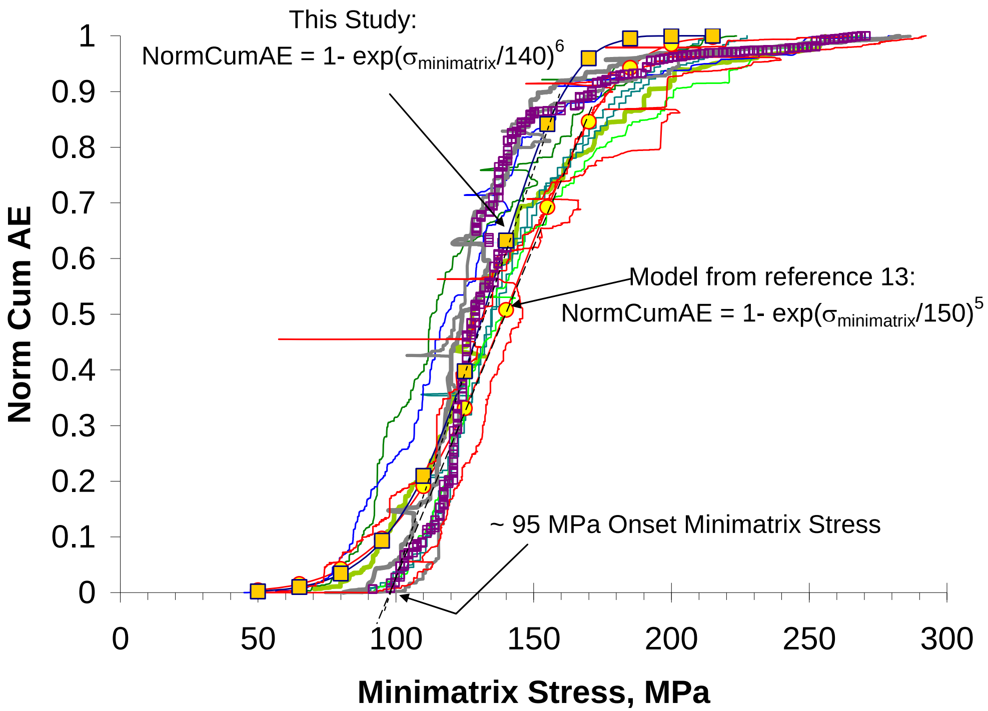
- Polycrystalline SiC fibers have higher residual compressive stress, higher E, and higher nonlinear stress
- Lower E SiC-based fibers (HN and ZMI) have larger strains to failure

Acoustic Emission Activity



Convert composite stress to the stress in the composite “outside” the load-bearing minicomposite





$$\sigma_{\min imatrix} = \frac{(\sigma_c + \sigma_{th})}{E_c}\left(\frac{E_c - f_{\min i} E_{\min i}}{1 - f_{\min i}}\right)$$

$f_{mini} = f_f + f_{BN} + f_{CVI\text{-}SiC}$

E_{mini} = R.O.M.

From, G.N. Morscher, *Composites Science and Technology* (2004)

Benefits of "minimatrix" Approach

1 Can model stress-strain behavior of most 2D woven MI composites (w/similar tow size)

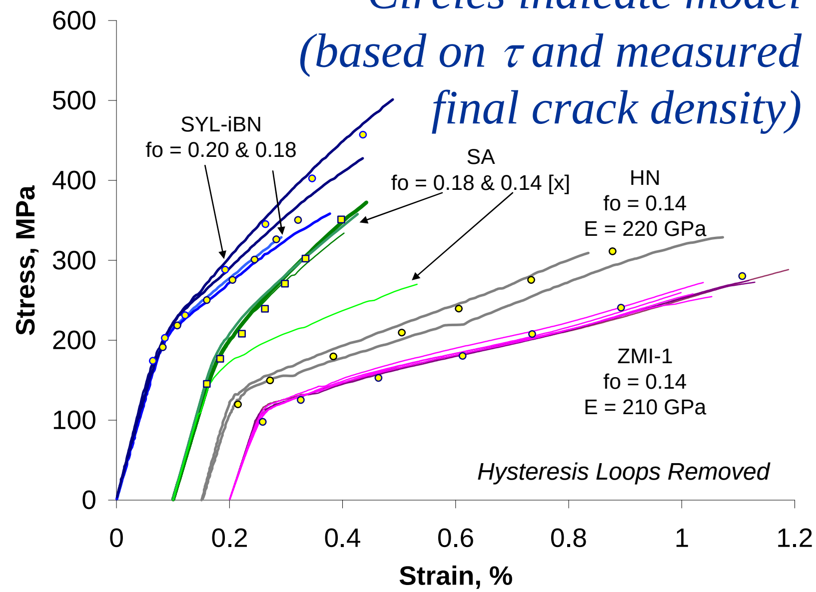
$$\varepsilon = \boldsymbol{\sigma/E_c + \alpha\delta\rho_c/E_f \; (\sigma + \sigma_{th})}$$

after Pryce and Smith; Curtin et al.

$$\delta = \alpha \, r \, (\sigma + \sigma_{th}) / 2\tau$$

$$\alpha = (1\text{-}f) \, E_m / f \, E_c$$

Circles indicate model (based on τ and measured final crack density)

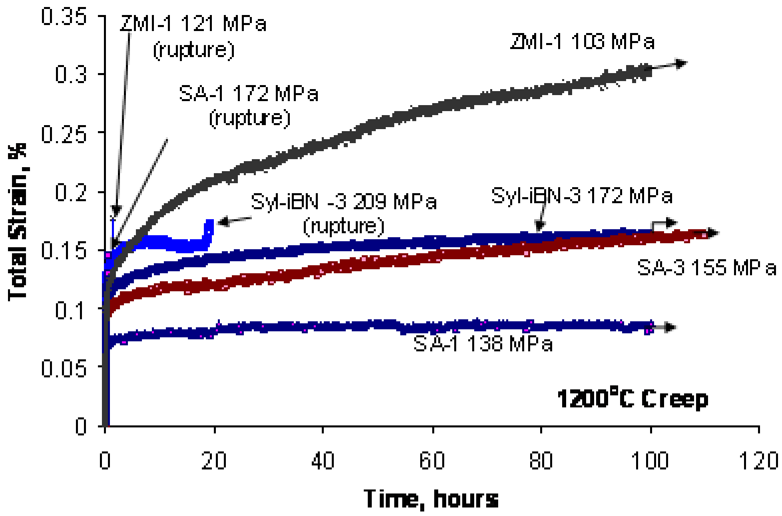


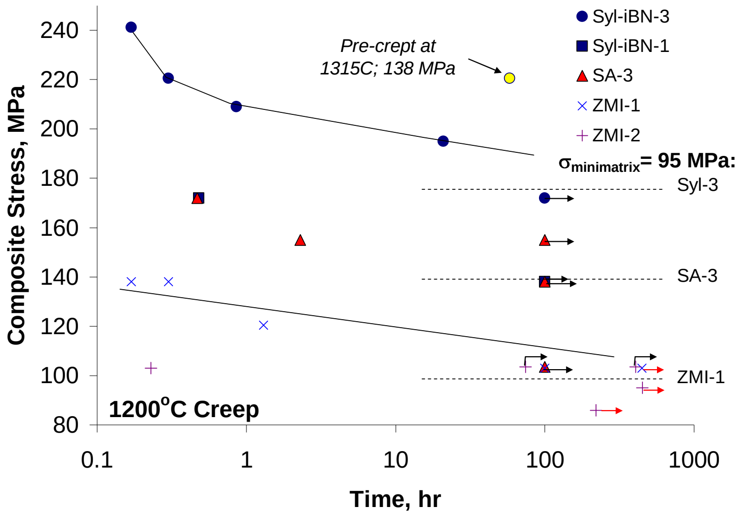
2 Can establish a simple design stress: AE onset stress

$$\sigma_{c-MatrixCracking} = \frac{(95MPa \bullet E_c)}{E_c - f_{\min i} E_{\min i}} (1 - f_{\min i}) - \sigma_{th}$$

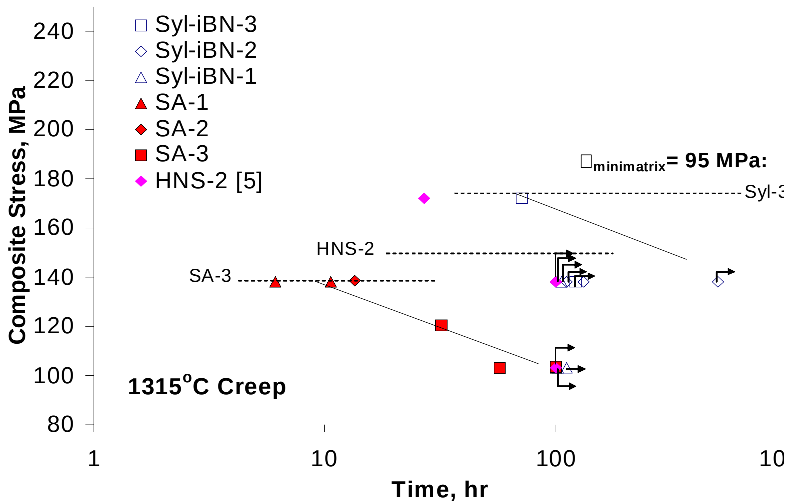
Minimatrix parameter compared to creep run-out at 1200 and 1315^{o}C

<u>1200^{o}C</u>: Good correlation between $\sigma_{c\text{-Matrix-Cracking}}$ and run-out





<u>1315^{o}C</u>: $\sigma_{c\text{-Matrix-Cracking}}$ overestimates run-out condition *(creep effects become dominant)*



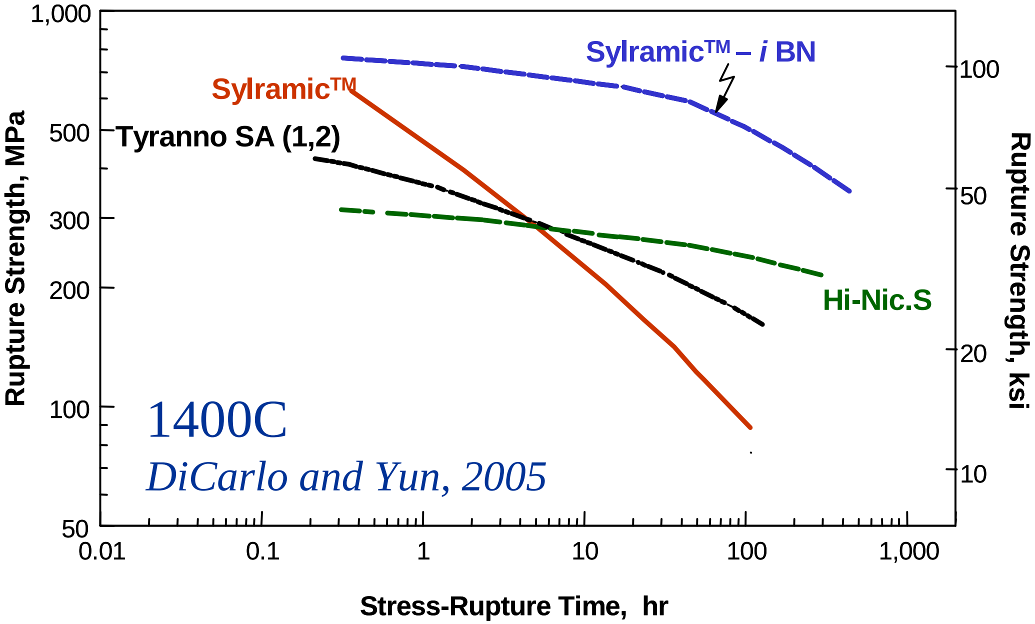
Fiber Architectures that Enable Processing and Properties for Desired Components

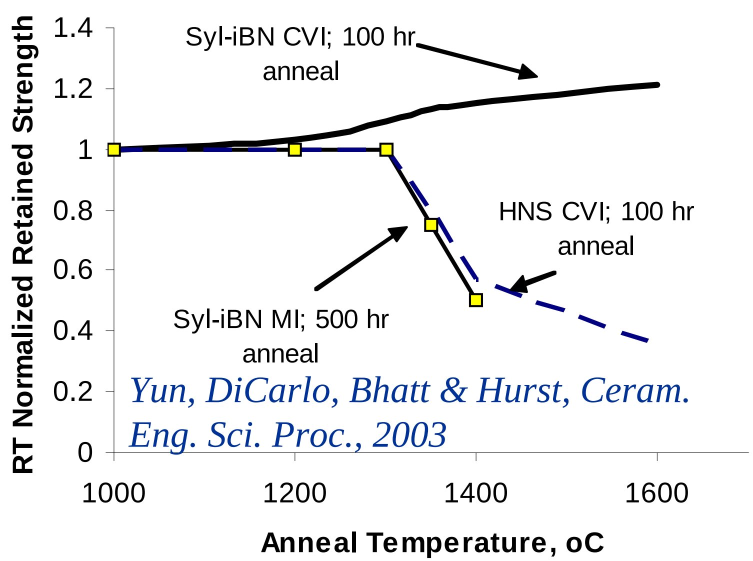
Approach → Process a wide variety of fiber-architectures in order to (1) determine the effect of architecture on composite properties for the purpose of tailoring properties in desired directions and (2) determine if these architectures could be successfully fabricated in order to anticipate processing further architecture modifications.

Based on paper in process with *Journal of the American Ceramic Society* (J.A. DiCarlo, J.D. Kiser, and H.M. Yun co-authors)

Sylramic-iBN Based Composites for Applications ≥ 1300ºC

- Sylramic-iBN = NASA derived heat treatments of Sylramic fiber
- Excellent creep resistance and thermal stability (up to 1800ºC)
  - Best mechanical performance at high temperatures
  - In-situ grown (tailorable) BN-based interphase composition
  - Enables high temp processing routes not possible with other fiber-types, usually at temperatures well above the application use temperature!

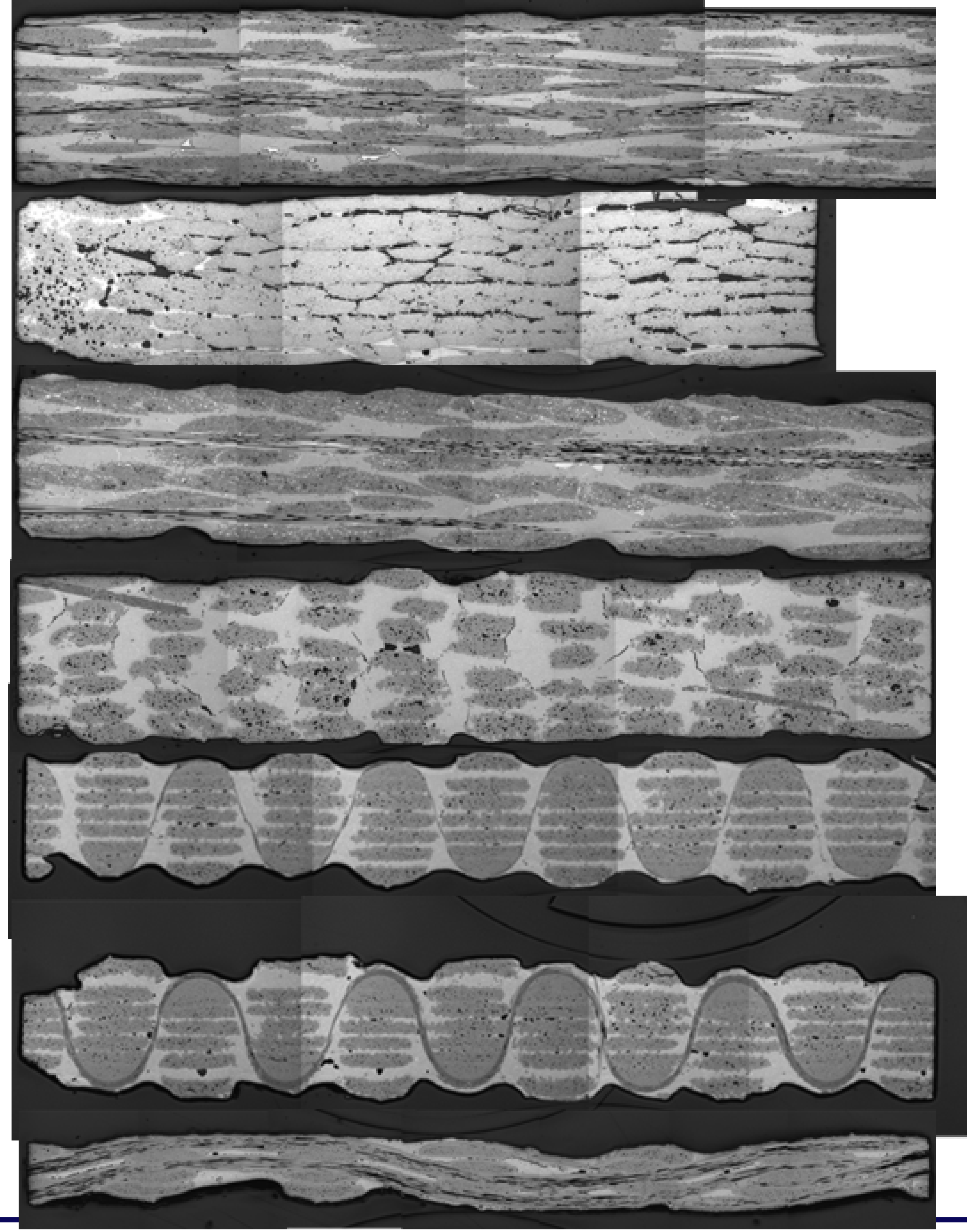




Tailoring Cracking Behavior with Fiber Architecture (Syl-BN MI Composites)

- A variety of architectures are being studied for the Syl-iBN MI system to determine effect of fiber architecture and fiber content on matrix cracking
  - 2D five harness satin with different tow ends per inch
    - Standard composite (N24A) = 8 layers of balanced 7.9 epcm (20 epi)
  - 2D five harness satin with different tow sizes
  - 3D orthogonal with different Z fibers – balanced and unbalanced in X and Y direction
  - Layer to layer angle interlock
  - Through the thickness angle interlock (with low Y fiber content) ≅ ***Unidirectional composite***
  - 2D five harness satin with high tow ends per inch in X direction and rayon in Y direction ≅ ***Unidirectional composite***

Some Cross-Sections



2D 5HS
N24A

5HS UNI

Braid

AI UNI

3DO-R

3DO-Z

LTL AI

Determination of Fiber Volume Fraction

f_o = fraction of fibers that bridge a matrix crack (0 = loading direction), including fibers at an angle, e.g., a braided architecture

$$f_o = \frac{N_f A_f}{A_c} = \frac{N_{ply} N_{f/tow} N_{tows/ply} \pi R_f^2}{tw}$$

$$N_{tows/ply} = \frac{epcm}{10} w$$

$$f_o = \frac{N_{ply} N_{f/tow} epcm \pi R_f^2}{10t}$$

N_f = total number of fibers in the cross-section of the tensile specimen,

A_f = area of a fiber

A_c = cross-sectional area of the tensile specimen (tw)

N_{ply} = # of plys or layers through the thickness,

$N_{f/tow}$ = # of fibers per tow (800 for Syl-iBN),

$N_{tows/ply}$ = number of tows per ply or layer

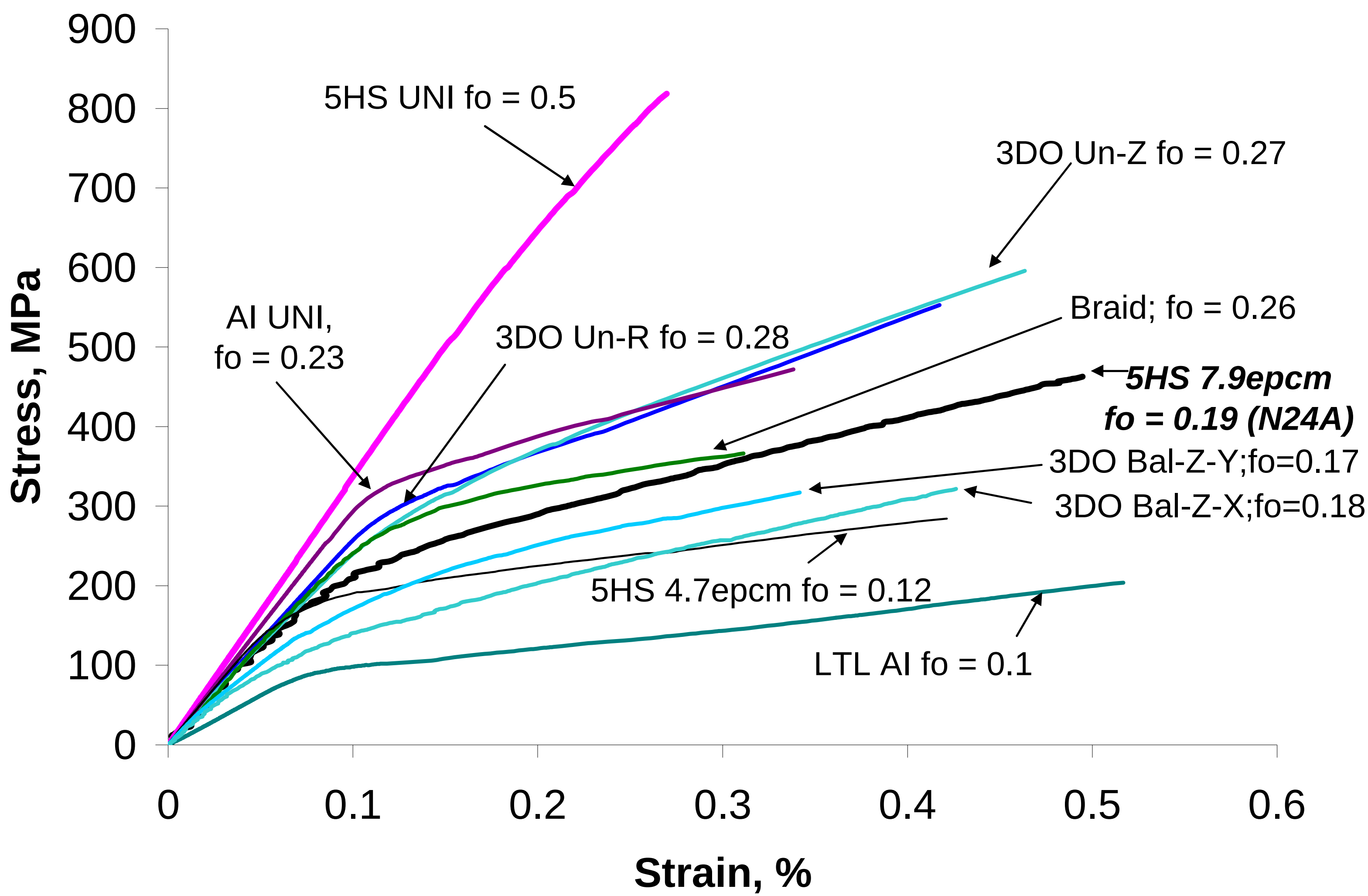
R_f is the fiber radius (5 mm or 0.005 mm for Syl-iBN).

epcm = tow ends per cm

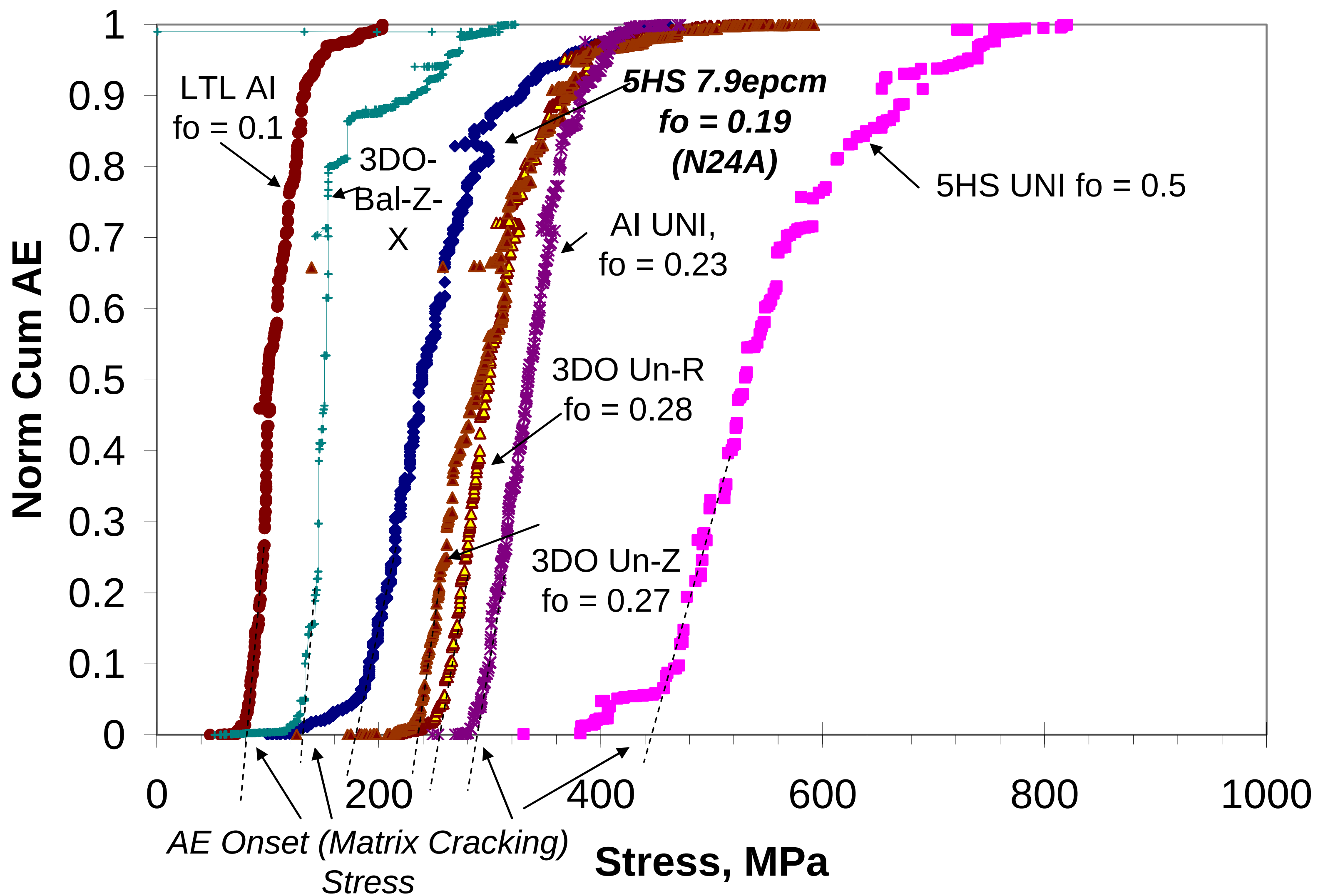
Description of Different Architecture Composites

Composite	Description	Thickness (mm)	Fiber fraction, $f_{o,}$ in load direction	E (GPa)	UTS (MPa)
5HS UNI (1)	Unbalanced five-harness satin; fill direction = Sylramic at 17 epcm; warp direction = low epcm rayon	**2.17**	**0.50**	**335**	**>818**
AI UNI (2)	Unbalanced through-the-thickness angle interlock; fill direction = Sylramic at 11 epcm, 7 layers; warp direction = low epcm ZMI and rayon	2.0	**0.23**	305 ± 4	>472
3DO-Un-R (2)	Unbalanced 3D orthogonal; Y (loading) direction = Sylramic at 9.8 epcm, 7 layers; X direction = Sylramic at 3.9 epcm; Z direction = Rayon	1.53	**0.28**	275 ± 9	>575
3DO-Un-Z (2)	Unbalanced 3D orthogonal; Y (loading) direction = Sylramic at 9.8 epcm, 7 layers; X direction = Sylramic at 3.9 epcm; Z direction = ZMI	1.58	**0.27**	262 ± 9	596
LTLAI (1)	Layer-to-layer angle interlock; 5.5 epcm, 3 layers	**0.96**	**0.10**	**125**	**204**
2D 5HS [6]	Standard balanced 2D five-harness satin; ply lay up; number of plys varied from 4 to 8; epcm varied from 4.9 to 8.7.	1.5 to 2.2	**0.12 to 0.2**	220 to 290	See [6]
2D 5HS [6] (double tow)	Balanced 2D five-harness satin ply lay up; two tows woven together at 3.9 epcm, 8 plys.	2.1	**0.19**	197	480
Braid [8]	Triaxial braid; double tow; -67/0/67 – tested in hoop orientation so fibers are oriented ± 23° to testing axis, 4 layers		**0.26**	250	352
3DO-Bal-R-Y [7]	Nearly balanced 3D orthogonal; Y (loading) direction = Sylramic single tow at 7.9 epcm,8 layer; X direction = Sylramic double tow at 3.9 epcm; Z fiber = Rayon	1.95	**0.20**	238	336
3DO-Bal-Z-Y [7]	Nearly balanced 3D orthogonal; Y (loading) direction = Sylramic single tow at 7.1 epcm,8 layer; X direction = Sylramic double tow at 3.9 epcm; Z fiber = ZMI	2.05	**0.17**	248	317
3DO-Bal-Z-X [7]	Same as 3DO-Bal-Z except oriented in the X (fill) direction (7 layer)	2	**0.18**	205	322

RT 0º σ/ε of Different Architecture Syl-iBN MI Composites

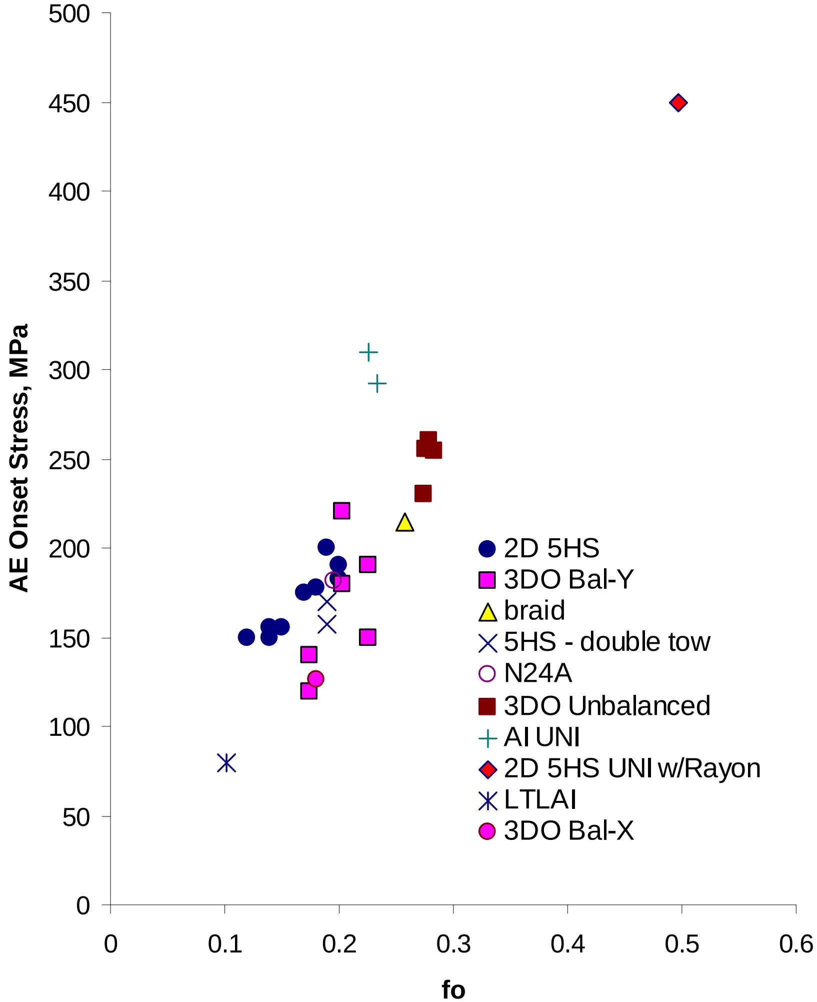


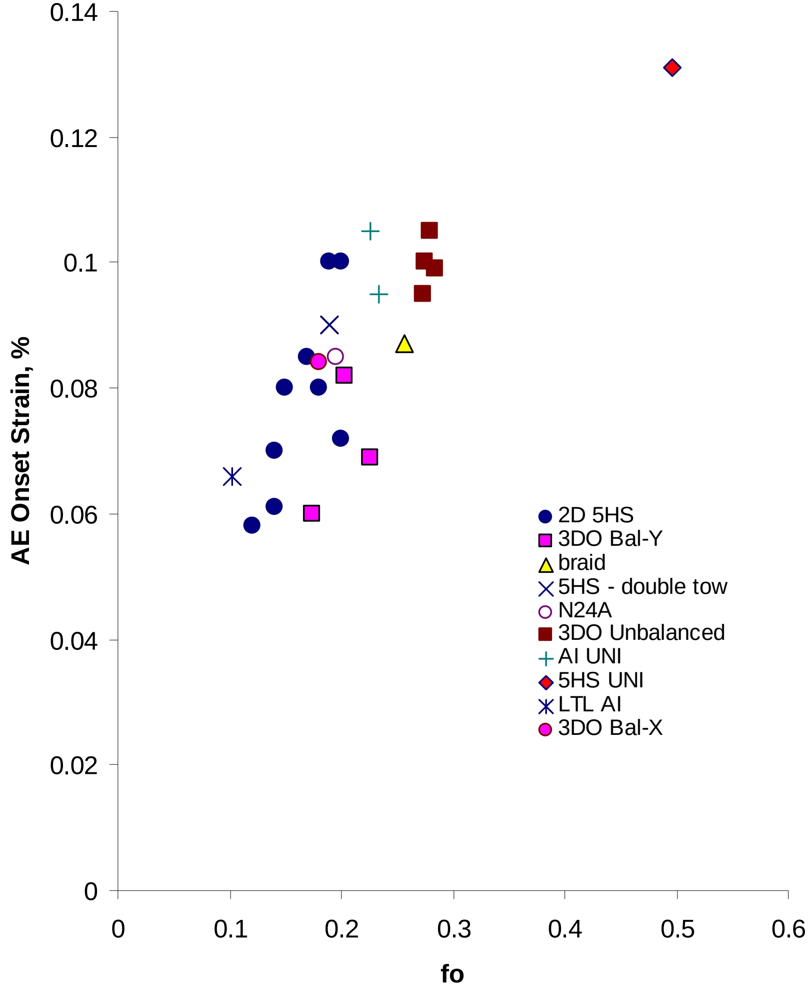
0º AE of Different Architecture Syl-iBN MI Composites



Effect of f_o on Matrix Cracking Stress

Primary factor affecting matrix cracking = fiber volume fraction





Calculating the unbridged ⊥ tow area

$$A_{\perp} = Length_{\perp Minicomposite} \cdot h_{\perp Minicomposite}$$

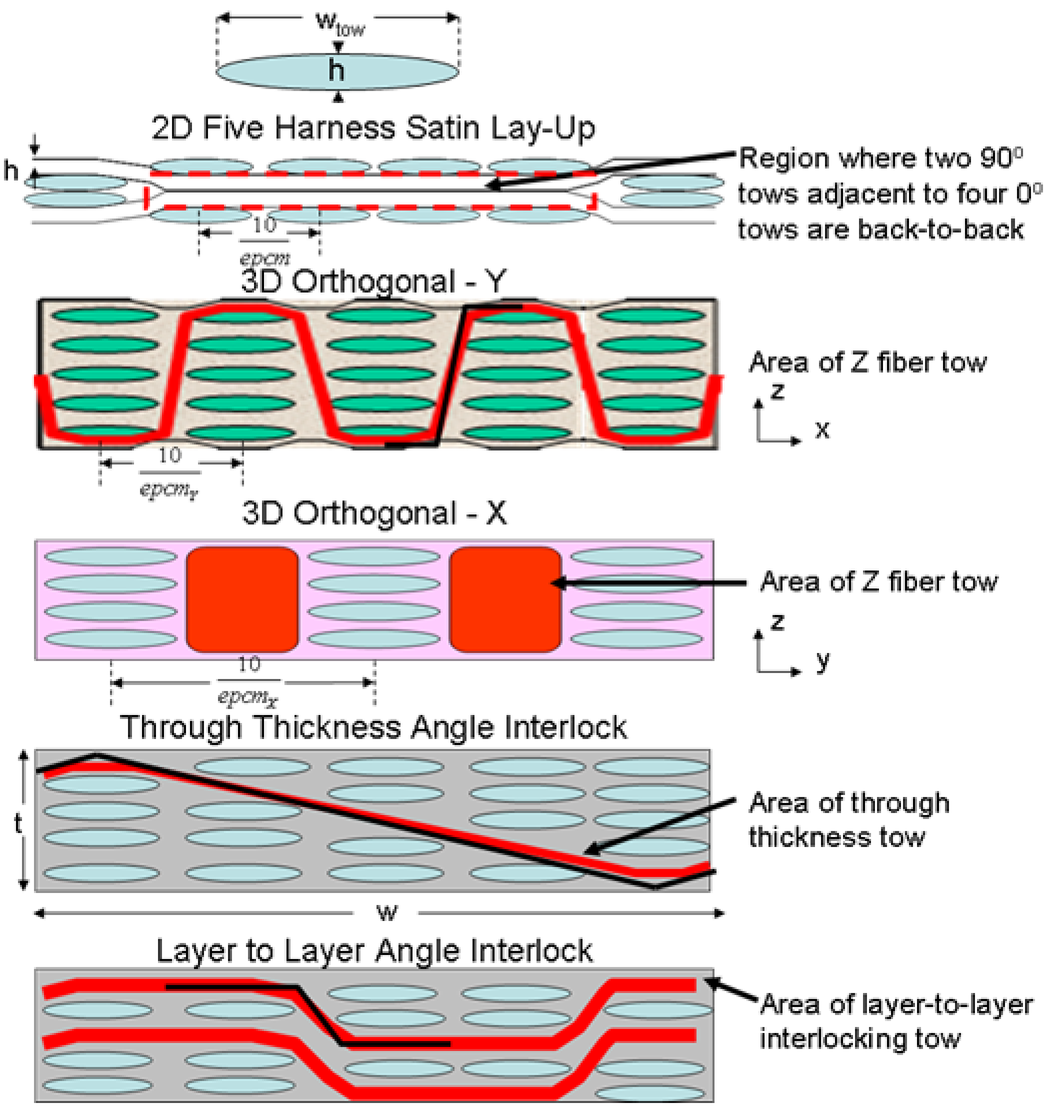
$$A_{\perp} = \frac{N_{hs} - 1}{epcm} 20 \cdot h_{90}$$

$$A_{\perp} = \frac{epcm \cdot w}{10}\left[\left\{\left(\frac{10}{epcm} - w_{tow-Y}\right)^{2} + (t - h_{z})^{2}\right\}^{1/2} + w_{tow-Y}\right] \cdot h_{z}$$

$$A_{\perp} = \left(\frac{10}{epcm_{x}} - w_{tow-X}\right) \cdot t$$

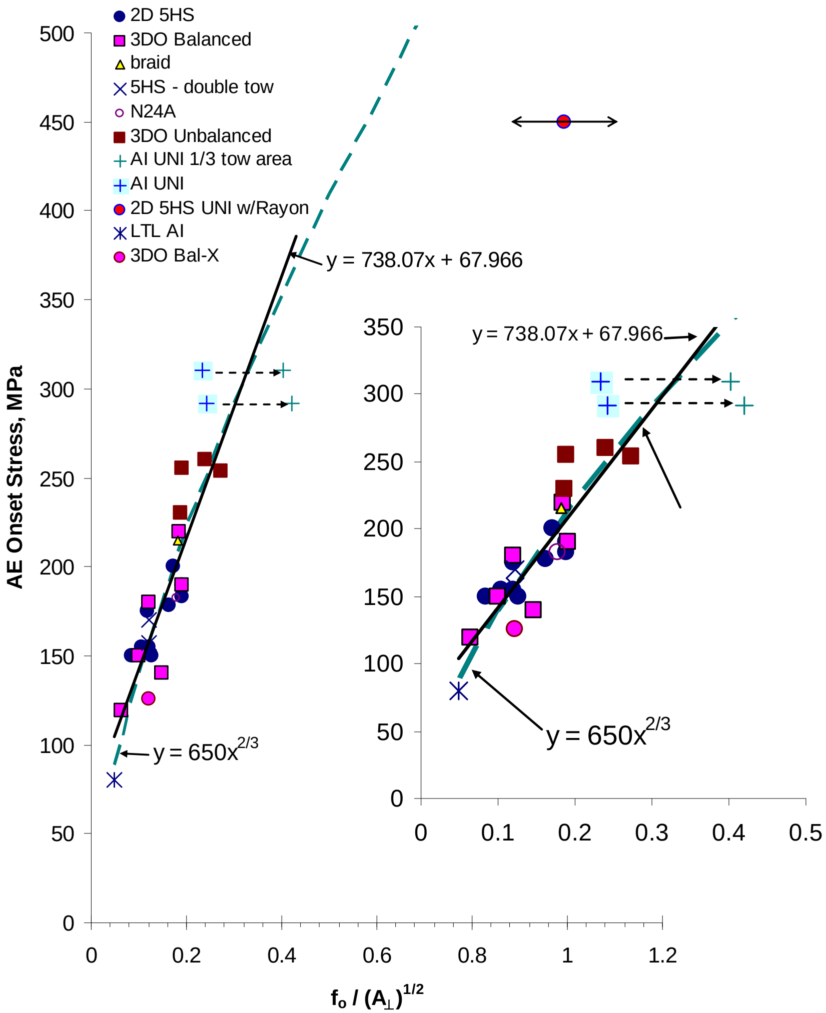
$$A_{\perp} = \frac{10 N_{ply}}{epcm} t \frac{epcm}{10} w \cdot h_{z} = N_{ply} t w \cdot h_{z}$$

$$A_{\perp} = \frac{epcm \cdot w}{10}\left[\frac{20}{epcm} + \frac{1}{2}\left\{t^{2} + \left(\frac{10}{epcm} - w_{tow-0}\right)^{2}\right\}^{1/2}\right] \cdot h_{z}$$

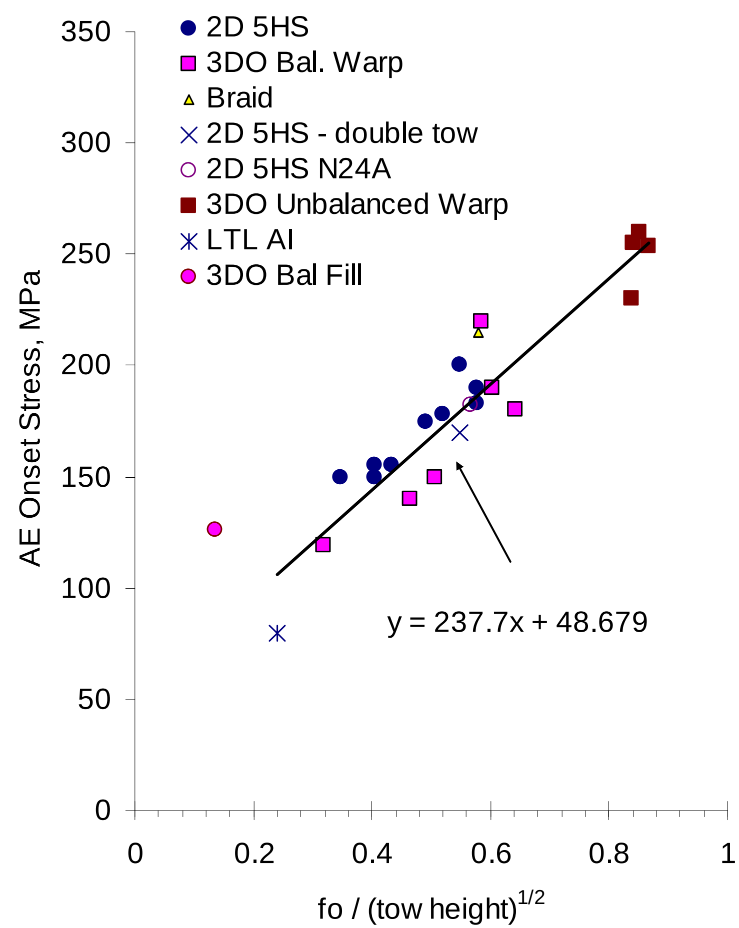


Effect of f_o and max ⊥ tow size on Matrix Cracking Stress

$f_o / (A_\perp)^{1/2}$

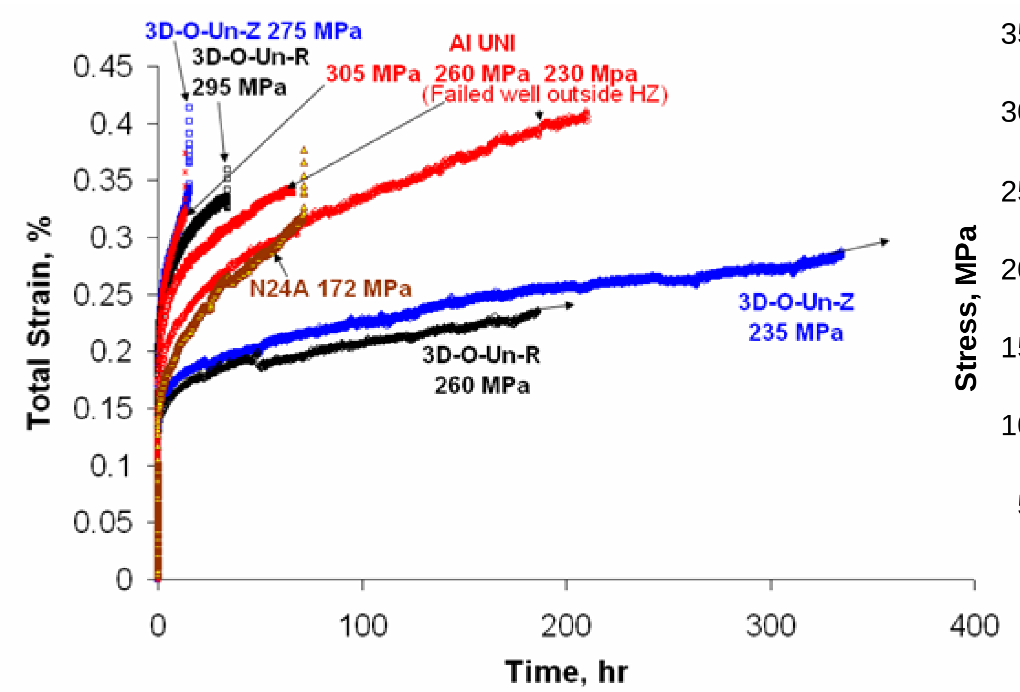


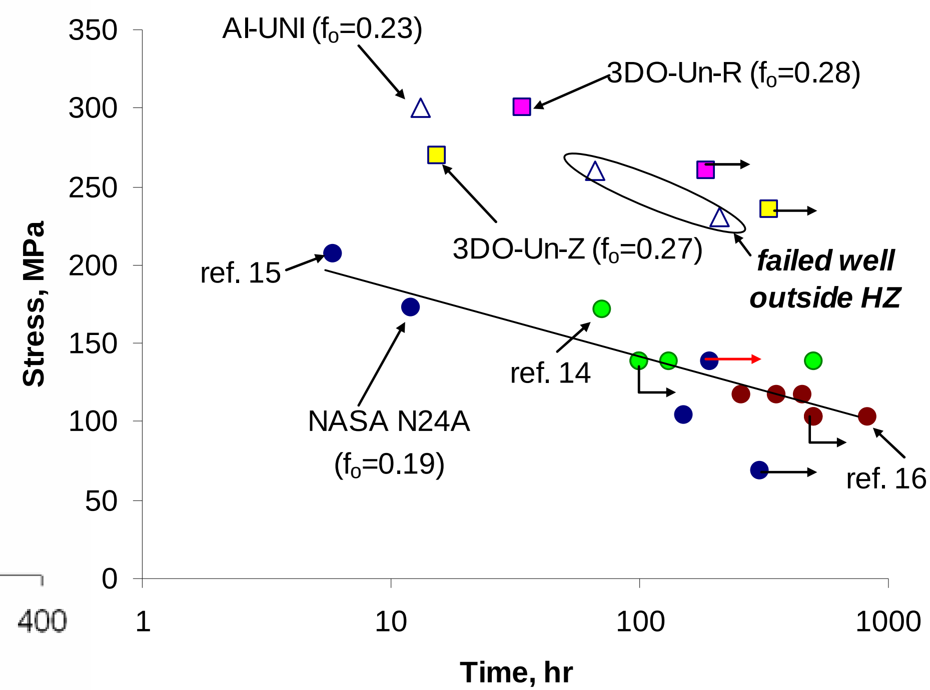
f_o / (tow height)$^{1/2}$



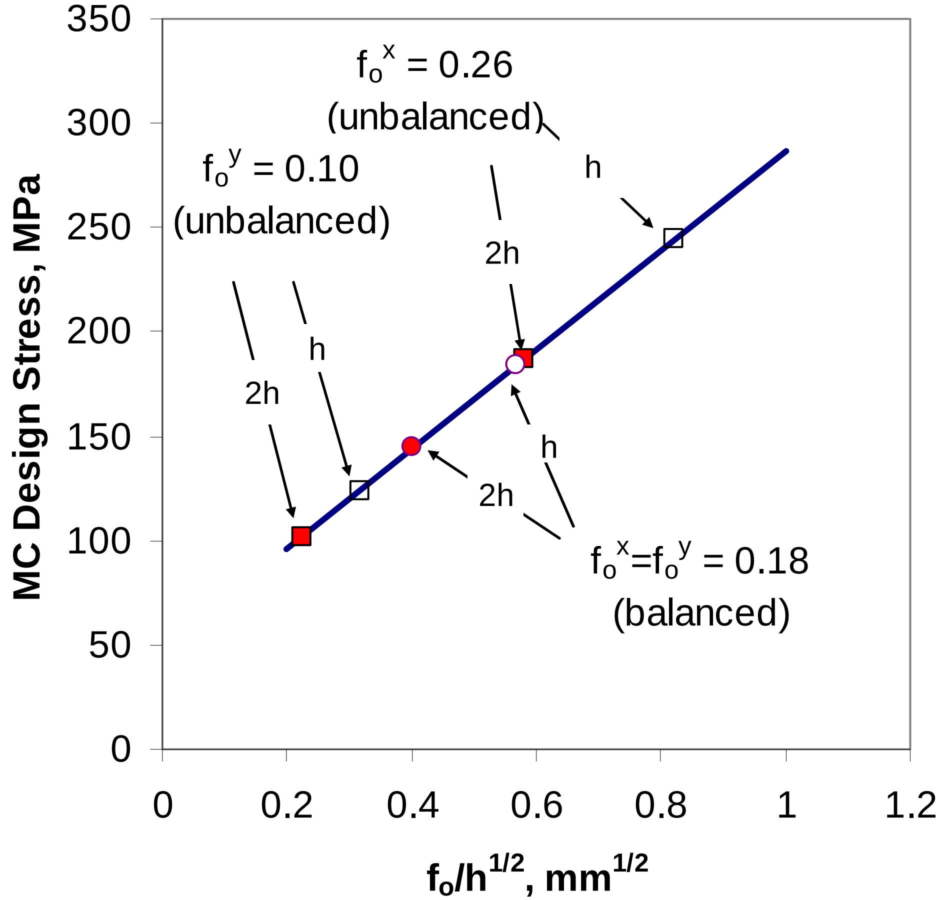
1315ºC Creep-Rupture of Different Architecture Composites

- Significant improvement (~ 100 MPa) in creep-rupture properties for unbalanced fiber architectures with high fiber fraction in loading direction over standard 2D five-harness composites





Design Stress Maps Can Be Constructed for Different Architectures and Fiber-Content



2D harness or 3D angle interlock architecture with single tow (h) or double tow (2h) weave

From paper in Proceedings to TEXCOMP9, (2008)

Implications and Conclusions

- Simple, yet robust relationships for stress-strain behavior and elevated temperature life based on general acoustic-emission derived matrix cracking relationship
  - Appears to be representative at least up to 1200^{o}C
- High temperature creep rupture properties controlled by fiber creep rupture properties
- Fiber architecture can be engineered to maximize stress carrying ability in desired direction(s)
  - Matrix cracking stress dictated by fiber volume fraction and the size of the largest perpendicular-to-stress minicomposite
  - Simple empirical relationship derived to account for effect of architecture on matrix cracking strength