# A Numerical Solution Routine for Investigating Oxidation-Induced Strength Degradation Mechanisms in SiC/SiC Composites

*Roy M. Sullivan*
*Materials and Structures Division*
*NASA Glenn Research Center*
*Cleveland, OH*

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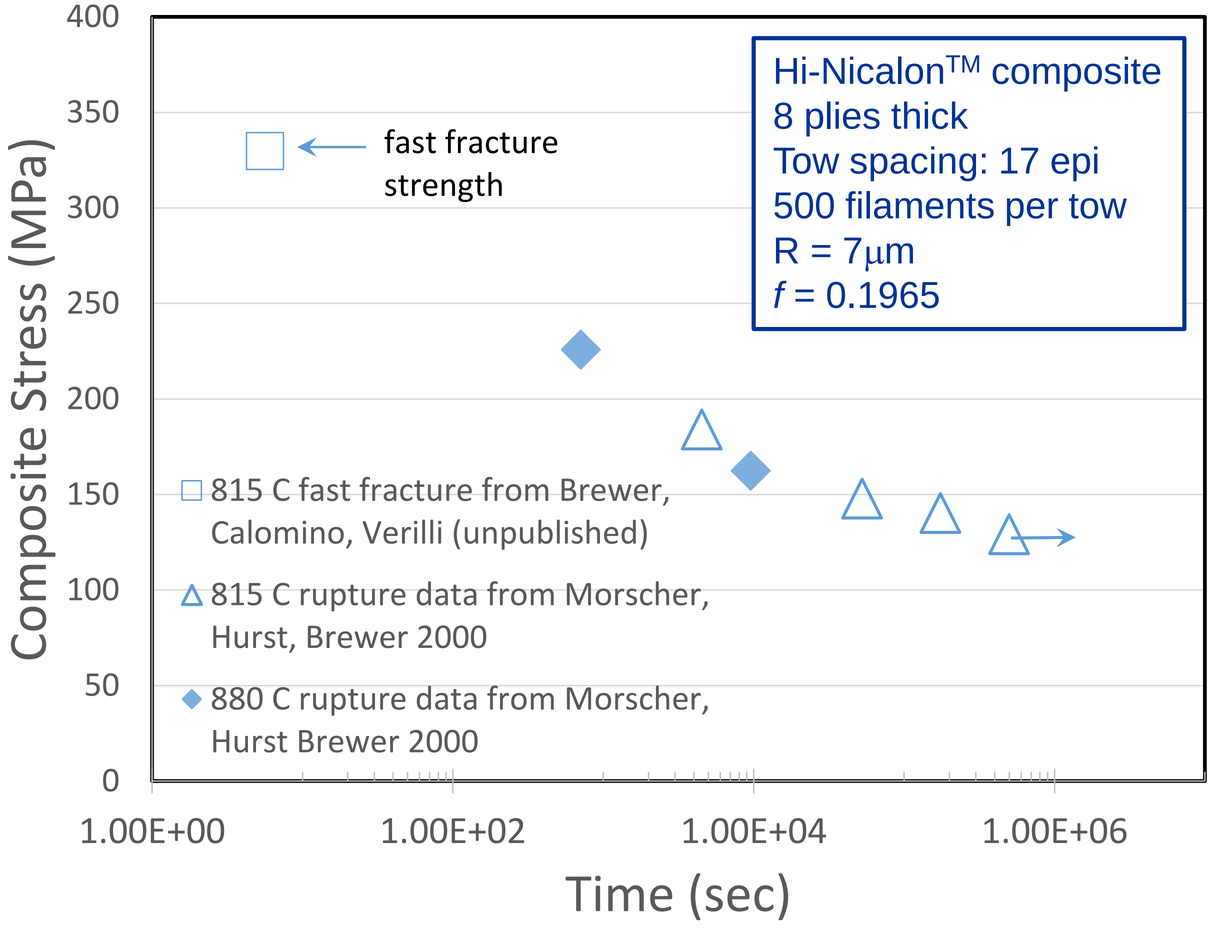
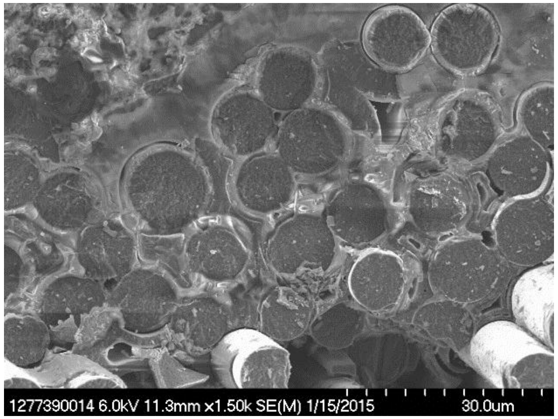
# What causes the time dependent strength degradation in SiC/SiC composites at intermediate temperatures (700 – 900 °C)?



*Stress versus time-to-failure of Hi-Nicalon$^{TM}$ composite specimens at intermediate temperatures from Morscher, Hurst and Brewer (2000).*

# Time Dependent Strength Degradation Mechanisms

Theory #1: Oxidation of BN fiber coating causes fusing of fibers to one another and to matrix resulting in embrittled composite.



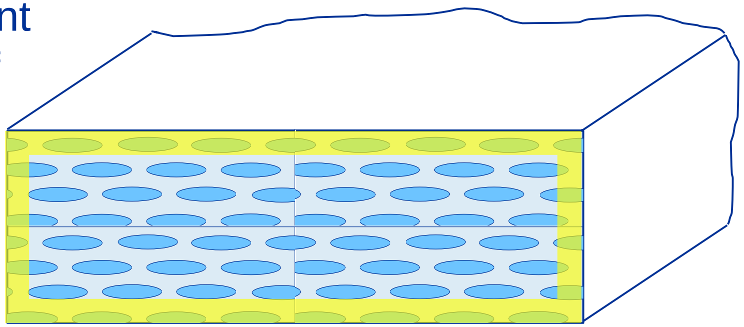
*Micrograph of SiC/SiC composite showing oxidized BN fiber coating. Courtesy of Ram Bhatt (NASA/GRC).*

Heredia, et al. (1995)
Morscher (1997)
Glime and Cawley (1998)
Morscher, Hurst and Brewer (2000)
Morscher and Cawley (2002).

$$2BN + \frac{3}{2}O_2 \rightarrow B_2O_3 + N_2$$

$$B_2O_3 + SiO_2 \rightarrow borosilicate\ glass$$

- Fusing causes local load sharing (LLS): Fibers adjacent to failed fibers are overloaded, causing a cascading of fiber failures and composite failure.
- Embrittlement is time dependent since extent of the cross-section that is fused increases with time.

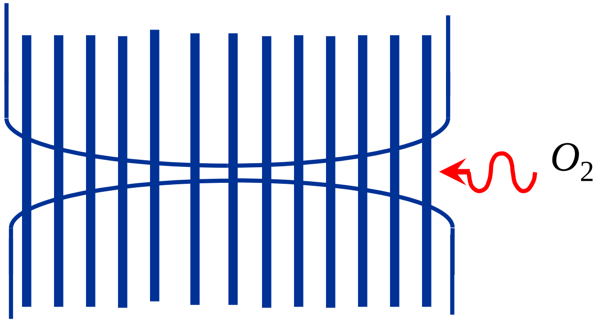


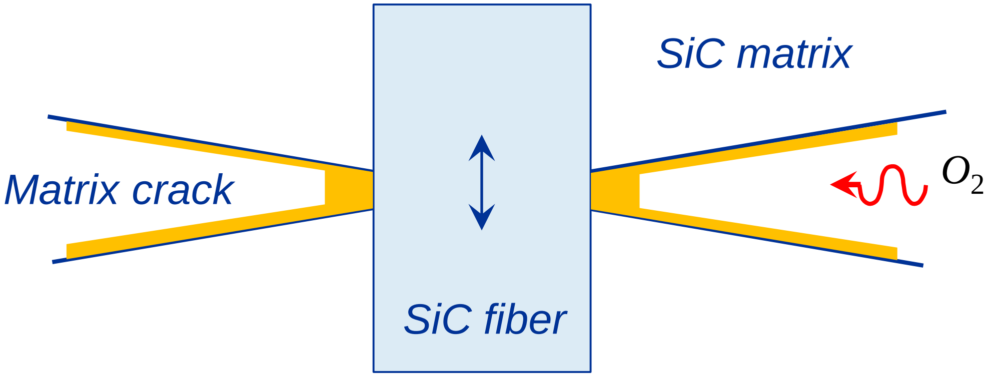
# Time Dependent Strength Degradation Mechanisms

Theory #2: Oxidation of SiC fiber results in tensile stress in fiber.

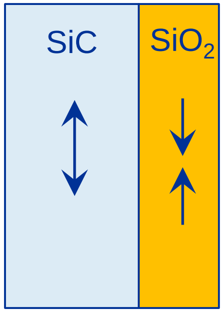
Xu, Zok and McMeeking (2014)
Hay (2012).

$$SiC + 2O_2 \rightarrow SiO_2 + CO_2$$





- Molar volume of silica is greater than SiC causing compression in oxide and tension in fiber.
- Tensile stress in fiber increases with time since oxide thickness grows with time.
- Results in an apparent loss in fiber strength over time.



*Fiber with oxide scale*

# Time Dependent Strength Degradation Mechanisms

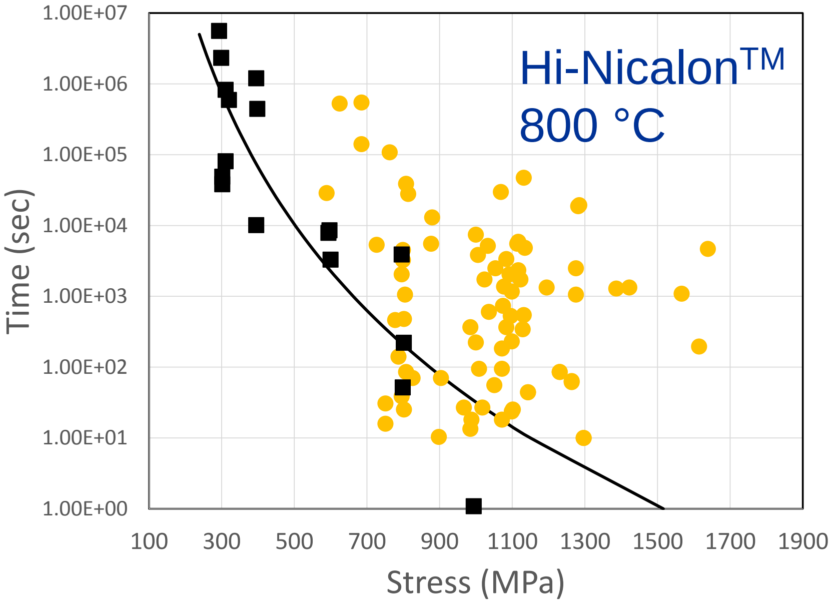
Theory #3: SiC fiber strength is intrinsically time dependent due to slow crack growth in fibers.

Forio, Lavaire and Lamon (2004)
Gauthier, Pailler, Lamon and Pailler (2009)
Gauthier and Lamon (2009).

*At intermediate temperatures, tows and single fibers have a stress vs time-to-failure relationship that follows*

$$\sigma_f^n \, t = A$$

*Some evidence of slow crack growth on fiber fracture surfaces*



*Rupture time versus stress for Hi-Nicalon™ single filaments and tows at 800 °C. Data from Gauthier and Lamon (2009).*

## Objective

- Investigate the cause of the time-to-failure vs. stress relationship in SiC/SiC composites with a BN fiber coating at intermediate temperatures.

## Approach

- Develop a progressive failure analysis routine (based on Theory #3) and apply it to simulate the composite stress rupture tests that produced the results shown on the first slide. The ability to simulate the stress vs. time-to-failure behavior will judge its validity.

## Assumptions

- Composite failure initiates at a matrix crack.

- The progression of fiber failure occurs under global load sharing (GLS).

# Fiber Failure Model (Relationship between $P_f - \sigma - t$)

*Fast Fracture Strength*

$$C_i = \left( \frac{K_{Ic}}{\sigma_f^s Y} \right)^2$$

*Flaw Growth Velocity (Davidge, et al., 1973)*

$$v = \frac{dC}{dt} = \alpha_1 K_I^n$$

*Weibull FF Strength Distr.*

$$P_f = 1 - \exp\left( -\frac{L}{L_o} \left( \frac{\sigma_f}{S_o} \right)^m \right)$$

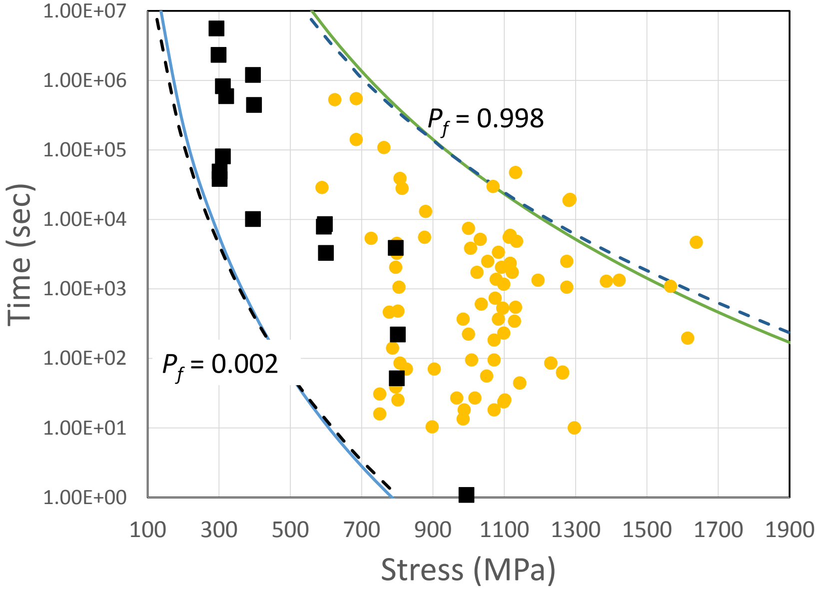
$$\sigma_f^n t = \frac{2}{\alpha_1 Y^2 (n-2)} \left( \frac{S_o}{K_{Ic}} \right)^{n-2} \left( \frac{L_o}{L} \ln \frac{1}{1-P_f} \right)^{\frac{n-2}{m}}$$

Fitting the probability of failure curves to the data, one can extract values for the constants

$n \quad \alpha_1$ *Flaw Growth Parameters*

$m \quad S_o$ *Weibull Statistical Parameters*

$K_{Ic}$ *Fracture Toughness*



*Time-to-failure versus applied stress for Hi-Nicalon$^{TM}$ single fibers (gold circles) and tows (black squares) at 800 °C from Gauthier and Lamon (2009).*

# Rearrange the previous expression to get an expression for the Probability of Failure

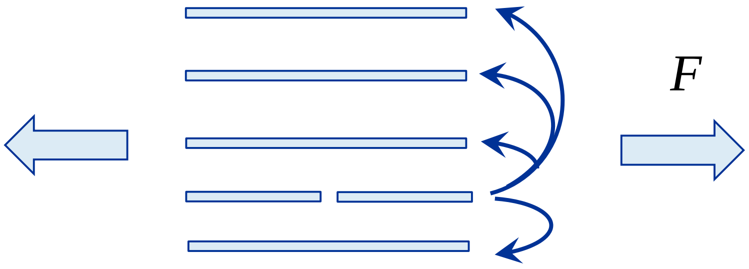
$$\sigma_f^n t = \frac{2}{\alpha_1 Y^2 (n-2)} \left(\frac{S_o}{K_{Ic}}\right)^{n-2} \left(\frac{L_o}{L} \ln \frac{1}{1-P_f}\right)^{\frac{n-2}{m}}$$

$$P_f = 1 - \exp\left\{ -\frac{L}{L_o} \left(\frac{1}{2} Y^2 \alpha_1 (n-2) t\right)^{\frac{m}{n-2}} \left(\frac{K_{IC}}{S_o}\right)^m \sigma_f^{\frac{nm}{n-2}} \right\}$$

Length effect

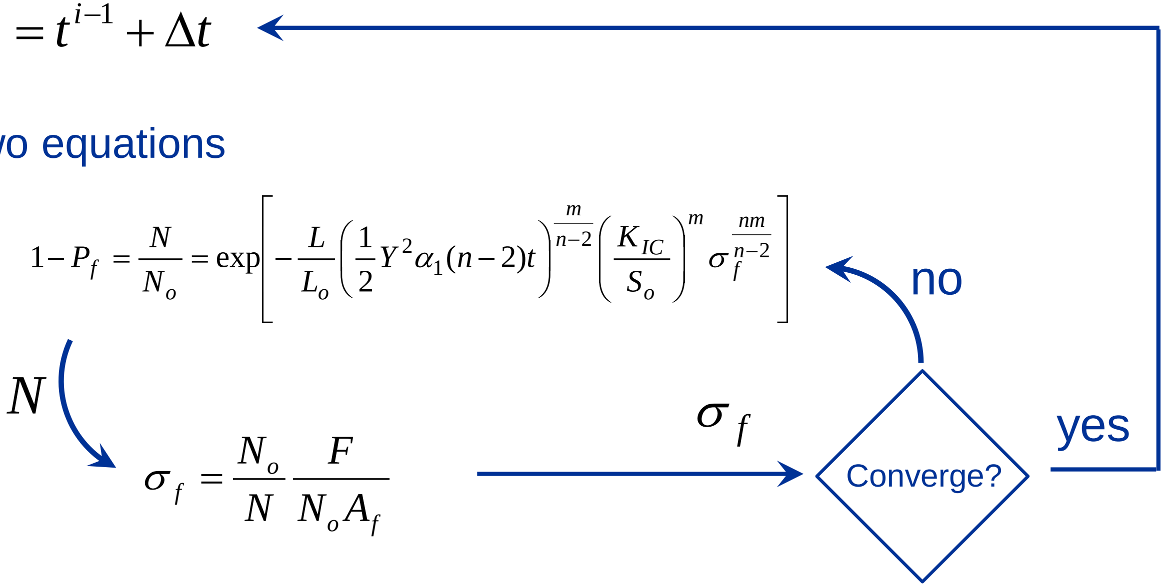
# Progressive Failure Analysis (PFA) Routine

- Numerically similar to Lara Curzio (1997)
- Based on Global Load Sharing (GLS) Model



*Global Load Sharing (GLS) Model*

## Flowchart



- Add time step, $t^i = t^{i-1} + \Delta t$
- Iterate between two equations

*Prob. of Survival*

$$1-P_f = \frac{N}{N_o} = \exp\left[-\frac{L}{L_o}\left(\frac{1}{2}Y^2\alpha_1(n-2)t\right)^{\frac{m}{n-2}}\left(\frac{K_{IC}}{S_o}\right)^m \sigma_f^{\frac{nm}{n-2}}\right]$$

$N$

*Force Equilibrium*

$$\sigma_f = \frac{N_o}{N}\frac{F}{N_o A_f}$$

$\sigma_f$

Converge?

no

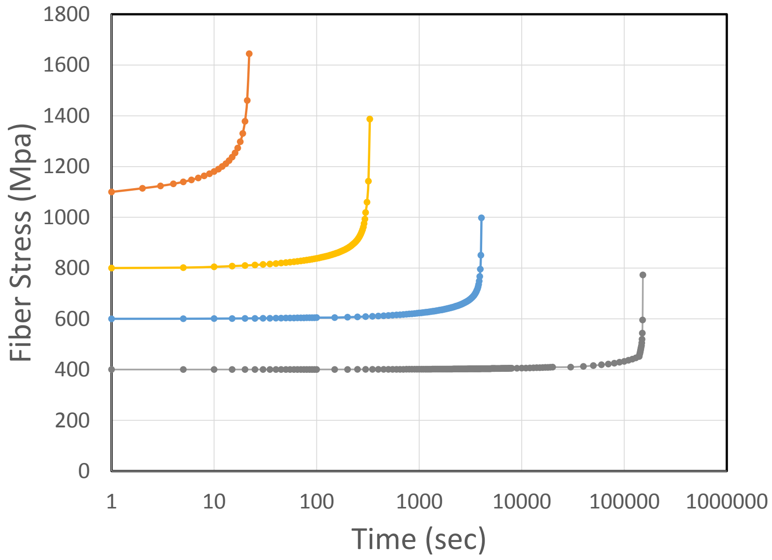
yes

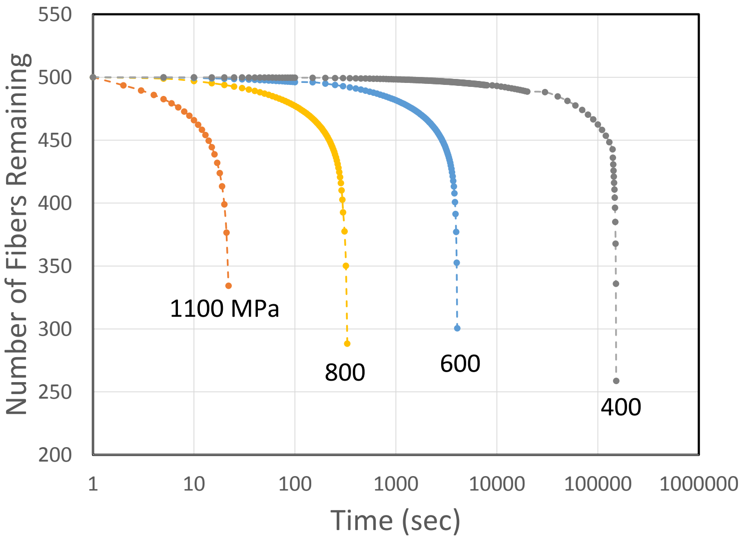
$N_o$ *number of fibers initially*

$N$ *number of fibers that are not failed*

$A_f$ *Cross-sectional area of fibers*

# PFA Simulation of Tow Tensile Tests from Gauthier and Lamon (2009)

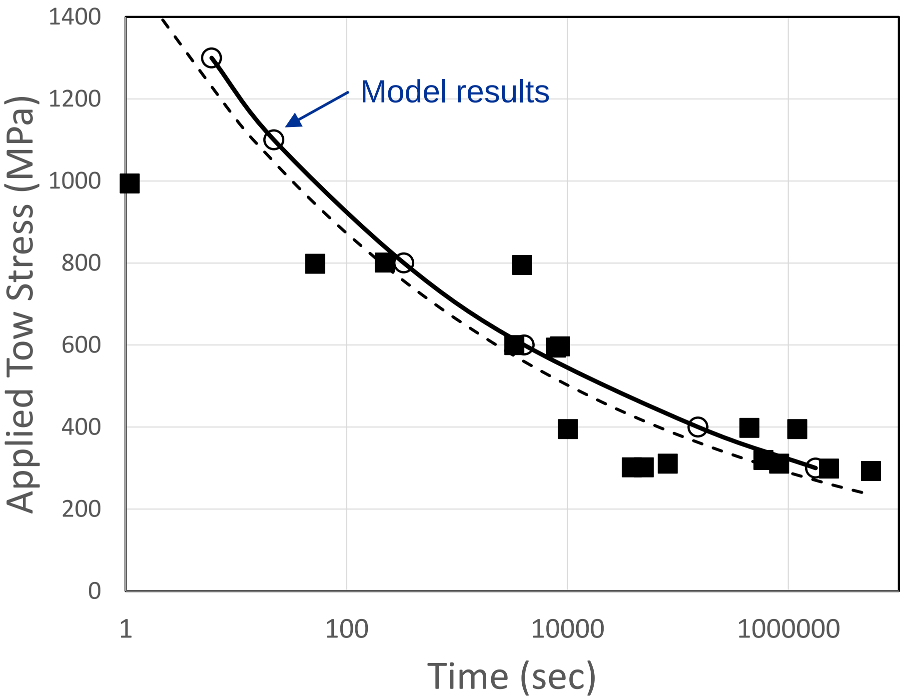




$$\sigma_f = \frac{F}{NA_f}$$

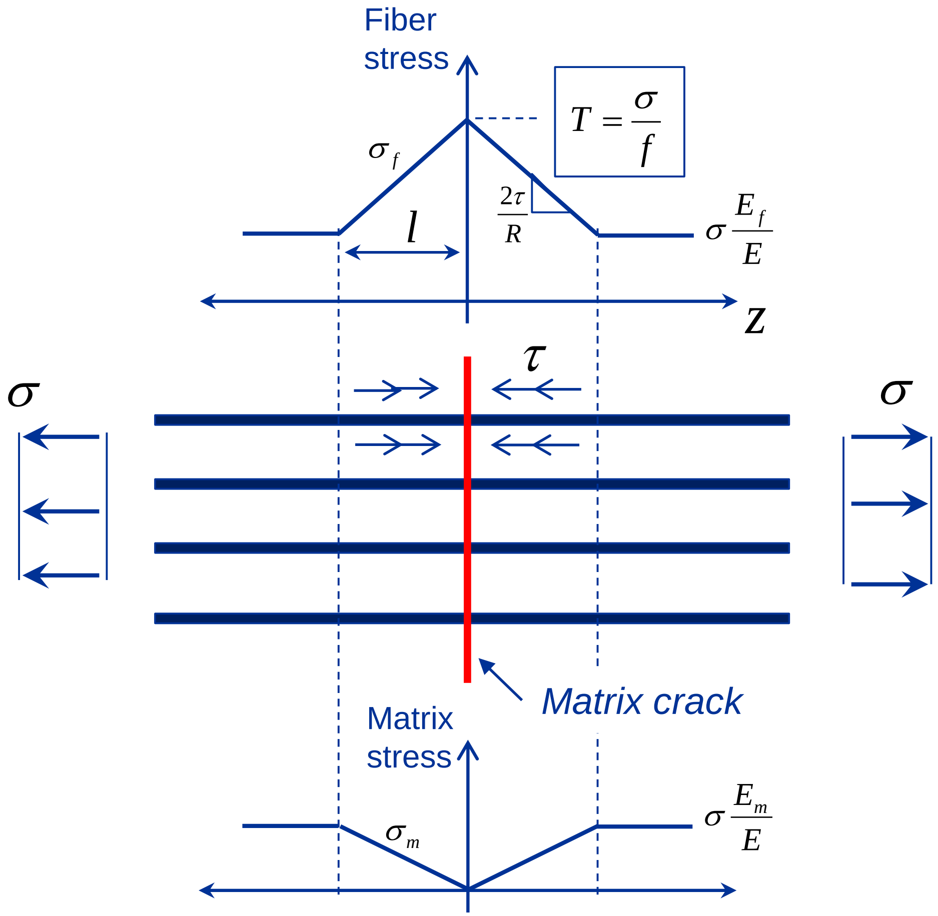
$$N_o = 500$$

$$L_o = 25\,mm$$



# Analysis of Composite Systems

Assumption: Composite failure initiates at a matrix crack.



Aveston, Cooper and Kelly (1971)
Curtin (1991)
Curtin (1994)
Curtin, Ahn and Takeda (1998)
Thouless and Evans (1988)

*Slip length*

$$l = \frac{R}{2\tau}\left(\frac{\sigma}{f}\right)\frac{(1-f)E_m}{E}$$

| | |
|---|---|
| $R$ | the fiber radius |
| $f$ | the fiber volume fraction in loading direction |
| $\tau$ | the sliding resistance shear stress |

# Analysis of Composite Systems

Length of fiber loading is now 2*l* and probability of failure now involves fiber stress at crack plane *T*

$$P_f = 1 - \exp\left\{ -\frac{\boxed{2l}}{L_o}\left(\frac{1}{2}Y^2\alpha_1(n-2)t\right)^{\frac{m}{n-2}}\left(\frac{K_{IC}}{S_o}\right)^m \frac{\boxed{T}^{\frac{mn}{n-2}}}{\left(\frac{mn}{n-2}+1\right)} \right\}$$

$$l = \frac{R}{2\tau}\left(\frac{\sigma}{f}\right)\frac{(1-f)E_m}{E}$$

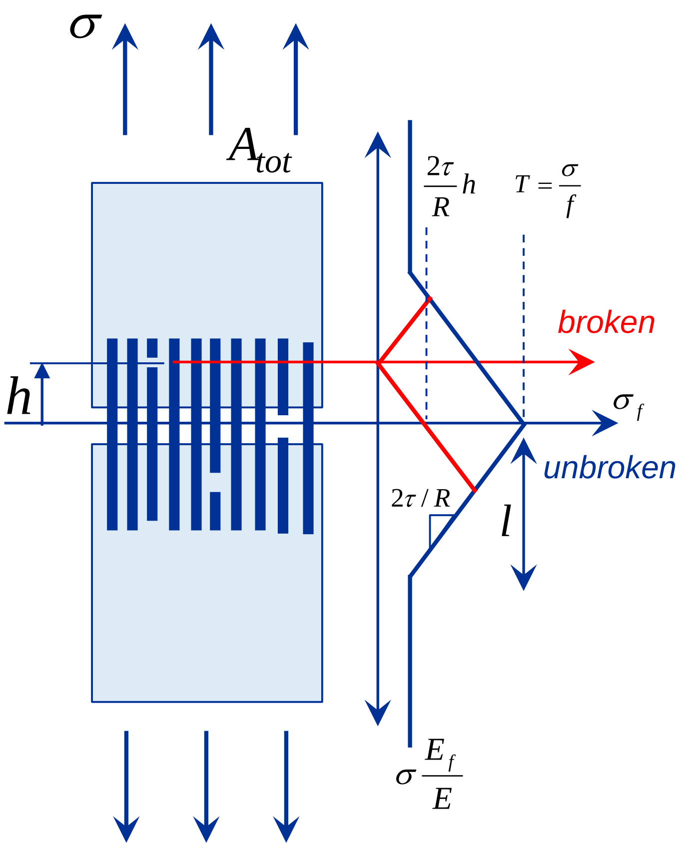
# Analysis of Composite Systems

For a single matrix crack, force equilibrium at crack plane requires

$$F = \sigma A_{tot} = \underbrace{TNA_f}_{\text{Force carried by unbroken fibers}} + \underbrace{(N_o - N)\langle \sigma_p \rangle A_f}_{\text{Force carried by broken fibers via pullout stress}}$$

$$\langle \sigma_p \rangle = \frac{2\tau}{R} \langle h \rangle$$ *Average Pull-out stress*

$\langle h \rangle$ *Average Pull-out length**



Rearranging

$$T = \frac{N_o}{N} \left[ \frac{F}{N_o A_f} - \left( 1 - \frac{N}{N_o} \right) \langle \sigma_p \rangle \right]$$

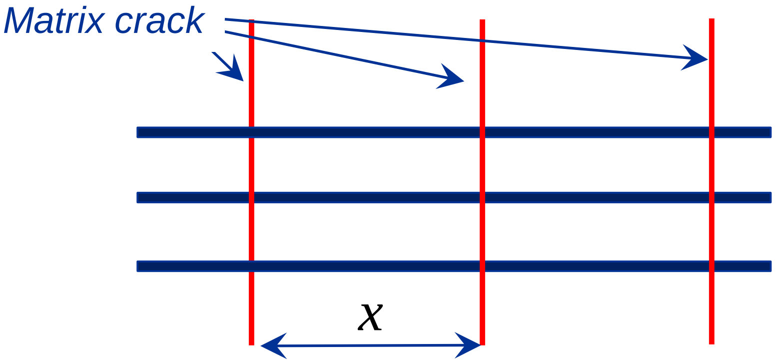
** Expression for average pull-out length obtained from Thouless and Evans (1988)*

# Crack Spacing and Shear Stress Calculations

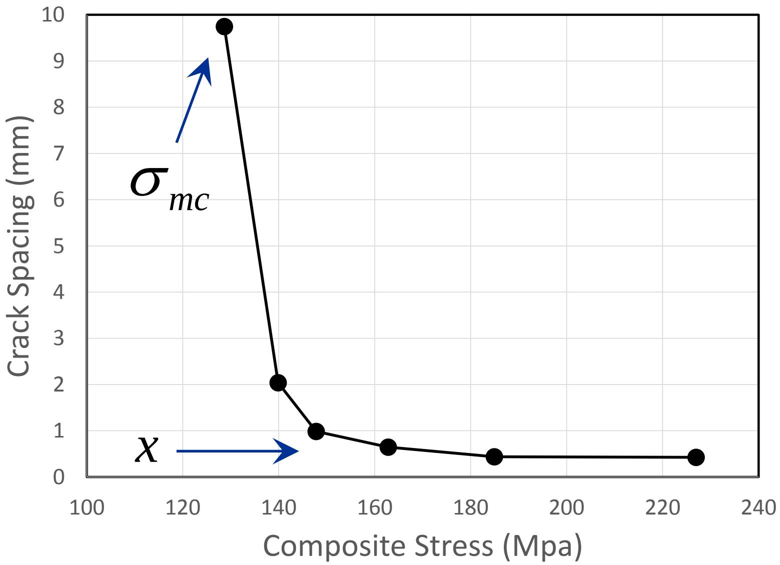
*Shear stress can be estimated from crack density measurements when cracks are saturated.*

Aveston, et al. (1971)
Kimber and Keer (1982)
Curtin (1991)

$$\bar{x} = 1.337 \frac{R}{2\tau}\left(\frac{\sigma_{mc}}{f}\right)\frac{(1-f)E_m}{E}$$



$$\left.\begin{array}{l} \bar{x} = 1.337 \frac{R}{2\tau}\left(\frac{\sigma_{mc}}{f}\right)\frac{(1-f)E_m}{E} \\ x = 0.4\,mm \qquad R = 7\,\mu m \\ \sigma_{mc} = 130\,MPa \qquad f = 0.1965 \end{array}\right\} \quad \tau \approx 5\,MPa$$



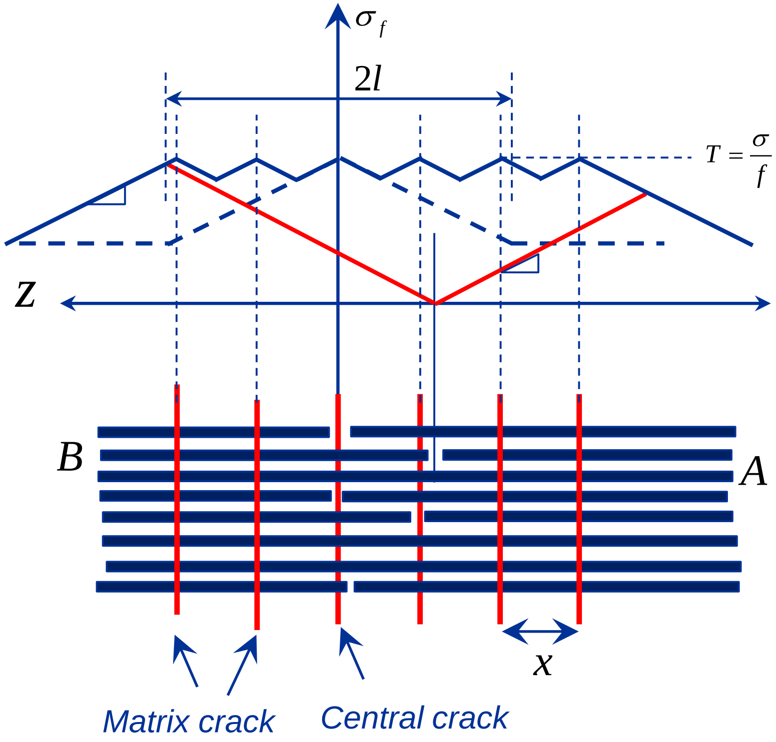
*Crack density versus composite stress for Hi-Nicalon$^{TM}$ composite from Morscher and Cawley (2002)*

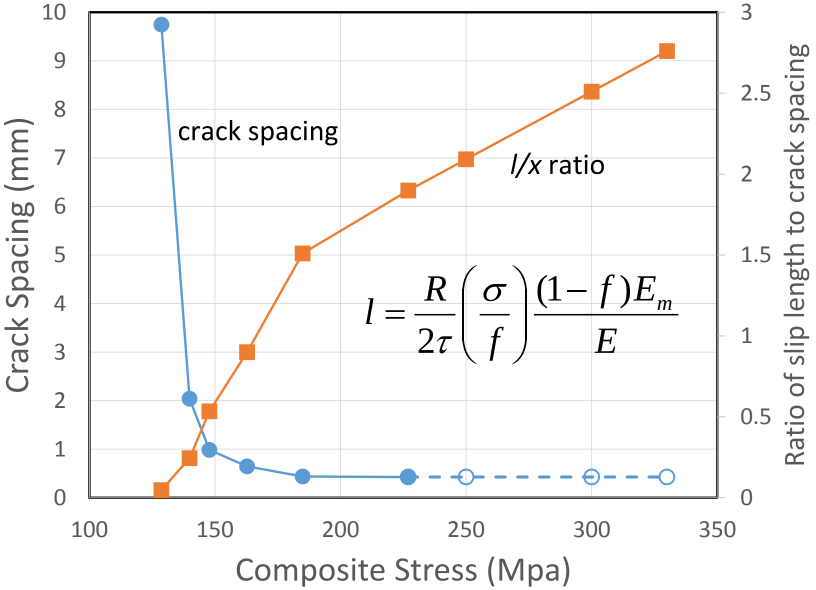
# Multiple Matrix Cracks within the Slip Length of One Another

If matrix cracks are close enough, fiber failures in nearby matrix cracks affect the force equilibrium equation in a central matrix crack plane

Number of matrix cracks within the slip length of any one matrix crack

$$\left(1+\frac{2l}{x}\right)$$





*Crack spacing and the ratio l/x versus composite stress. Crack spacing from crack density data from Morscher and Cawley (2002).*

## Force Equilibrium at Central Crack Plane (Curtin, et al. (1998))

$$T=\frac{F}{N_oA_f\left[1-\left(1-\frac{N}{N_o}\right)\left(1+\frac{l}{x}\right)\right]}$$

# Analysis of Composite Systems

## Progressive Failure Analysis routine now involves:

*Prob. of survival*

$$1-P_f = \frac{N}{N_o} = \exp\left\{-\frac{2l}{L_o}\left(\frac{1}{2}Y^2\alpha_1(n-2)t\right)^{\frac{m}{n-2}}\left(\frac{K_{IC}}{S_o}\right)^m \frac{T^{\frac{mn}{n-2}}}{\left(\frac{mn}{n-2}+1\right)}\right\}$$

*Iteration*

*Iteration*

*Force equilibrium*

$$T = \frac{N_o}{N}\left[\frac{F}{N_o A_f} - \left(1-\frac{N}{N_o}\right)\langle\sigma_p\rangle\right]$$

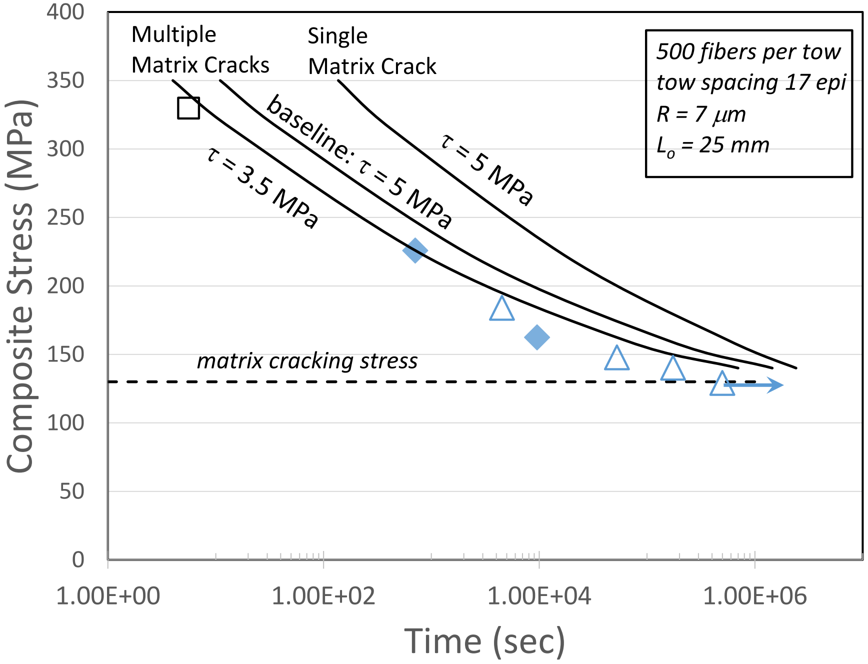
$$T = \frac{F}{N_o A_f\left[1-\left(1-\frac{N}{N_o}\right)\left(1+\frac{l}{x}\right)\right]}$$

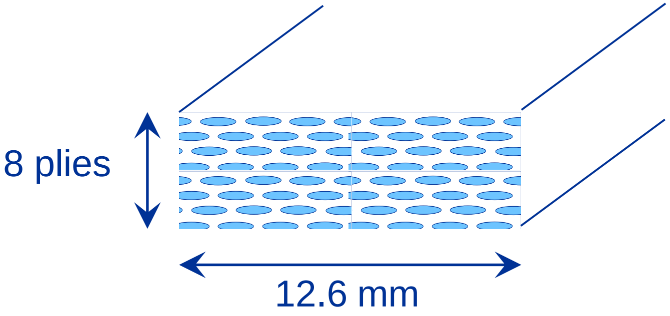
Single Matrix Cracks

Multiple Matrix Cracks

# Analysis of Composite Systems

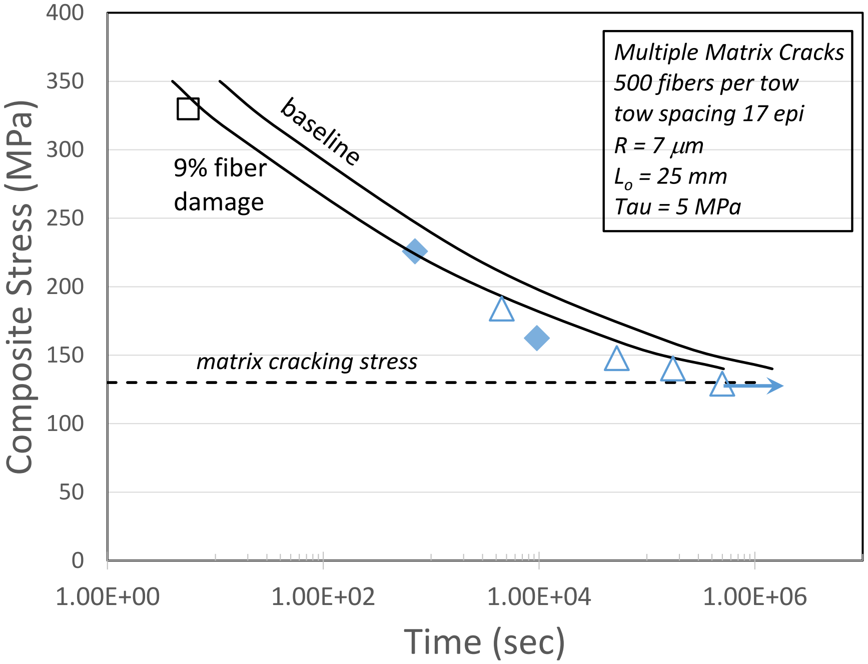
## Results: Each line represents a series of PFA solutions





$$\left(\frac{12.6mm}{25.4}\right)(17epi)(8\,plies) \approx 67.5\,tows$$

$$N_o = 67.5(500) = 33{,}750$$



Note: Marshall and Evans (1985) measured a shear stress value in other SiC fiber-reinforced ceramic matrix composites in the range of 2 - 2.5 MPa.

# Discussion and Conclusions

- The time dependent strength of Hi-Nicalon$^{TM}$ fiber reinforced composites has been shown to be largely due to the intrinsic time dependent strength of the fibers. Other mechanisms (e.g. fusing and embrittlement) may have a small effect at later times.

- Best agreement with the measured time-to-failure versus composite stress was obtained with progressive failure analyses solutions using multiple matrix crack formulation and with a combination of shear stress values between 3.5 – 5 MPa and fiber damage values of < 9%.

- If slow crack growth in fibers requires oxidation of inter-granular interface, what is the source of oxygen? Does it flow from the surrounding atmosphere down a matrix crack or is there enough present in the constituents? SiC fibers? BN fiber coating?

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