# *Carbon Fiber Treatments and Sizings*

Presentation to the

1999 Electron-Beam Curing Workshop


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# *Background*

- The Interface Controls Structural Integrity and Durability of Composite Mechanical Properties
- Fiber Surface Chemistry Determines Interfacial Adhesion and Interphase Morphology
- As Produced, Glass is High Energy and Carbon is Low Energy with a Weak Boundary Layer

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# Typical Commercial Carbon Fiber Surface Treatments

1. Anodization (electrochemical oxidation) Using Acidic and Basic Electrolytes or Amine Salts
2. Dry Oxidation (hard to control)

Treatment Objectives:

- Remove poorly bonded materials and impurities
- controlled increase in surface area and roughness
- attachment of polar surface groups (oxygen)

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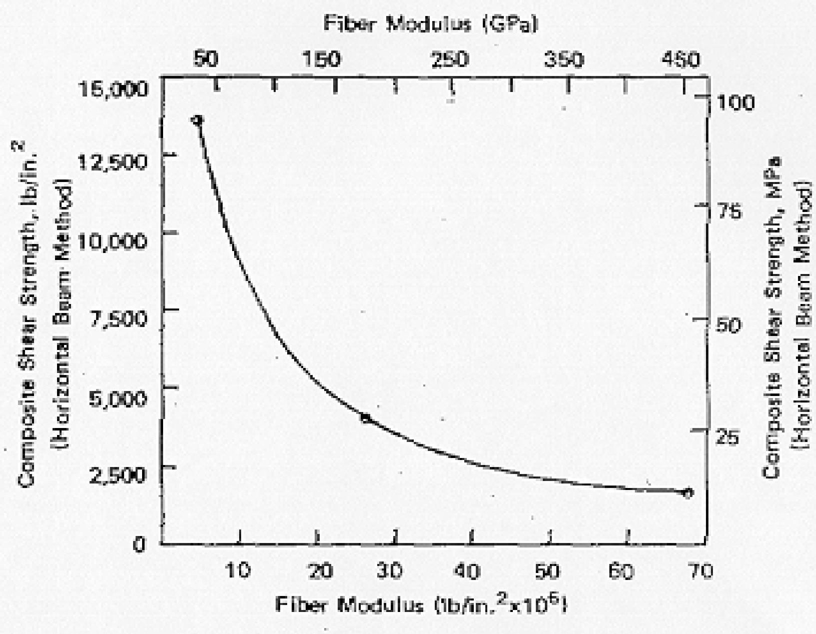
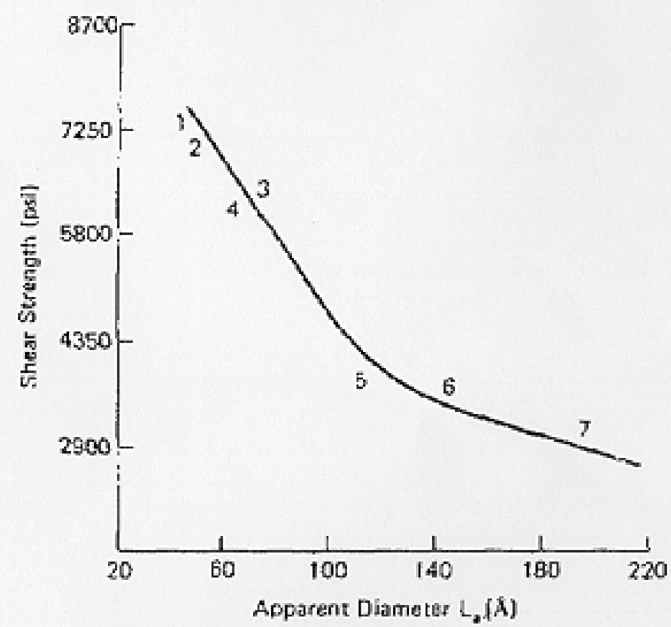
# *How Well Do Commercial Carbon Fiber Treatments Work?*




Ref. Simon et al (1967)     Ref. Tuinstra and Koenig (1970)

⇨ Higher modulus fibers and non-epoxy resins may take a different approach

# *XPS Surface Chemical Composition of Carbon Fibers (a/o)*

| Fiber (unsized) | C | O | Na |
|---|---|---|---|
| HM-type (untreated) | 97.7 | 2.3 | n.d.* |
| HMS-type (shear treated) | 92.0 | 7.3 | n.d. |
| HM-type ($CO_2$ plasma-treated) | 87.5 | 12.0 | 0.4 |

*n.d. – not detected

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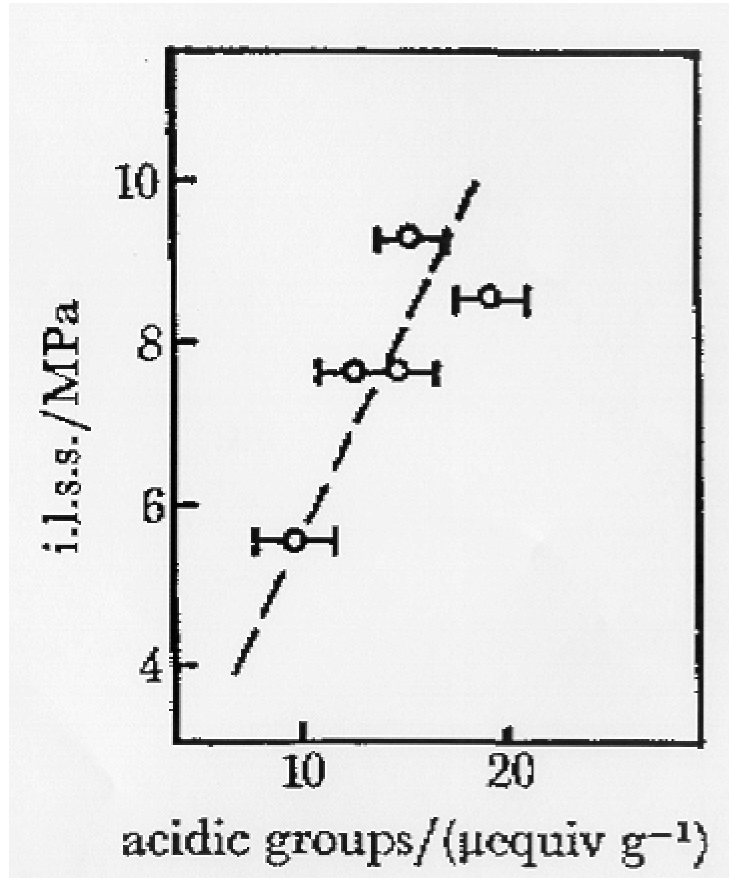
# *Effect of Surface Strong Acidic Groups on ILSS*




Ref. Ehrburger and Donnett (1980)

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# Carbon Fiber Surface Chemical Characterization

## IGC Analysis of t-Butylamine Adsorption at 30°C
## $^{\Sigma}$chemisorption (%)

| | As-Produced | Shear Treated | $CO_2$ Plasma |
|---|---|---|---|
| AS4-12K | 2.2 | 9.4 | 17.4 |
| HMS-12K | -- | 3.7 | 12.1 |
| P70-12K | 1.9 | 3.0 | 9.3 |

## Wetting Analysis with Formamide
## $W^{a/b}$, $mN/M^2$

| | | | |
|---|---|---|---|
| AS4-12K | 0 | 30.7 | 34.1 |
| HMS-12K | -- | 19.0 | 33.4 |
| P70-12K | 0 | 7.2 | 13.9 |

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# *Transverse Flexural Strengths of Unidirectional Carbon Fiber/Epoxy Laminates*

| **Fiber** | **$S_{22}$ (MPa)** | **Standard Deviation (MPa)** | **Coefficient of Variation (%)** | **Range (MPa)** | **Number of Tests** |
|---|---|---|---|---|---|
| Controls | | | | | |
| HM-type | 21.0 | 2.2 | 10 | 17.3-24.7 | 12 |
| HMS-type | 29.6 | 5.6 | 19 | 20.8-36.7 | 8 |
| UHM-type | 30.6 | 3.1 | 10 | 26.8-37.4 | 10 |
| $CO_2$-plasma-treated | | | | | |
| HM-type | 46.4 | 4.9 | 11 | 38.6-57.6 | 10 |
| UHM-type | 47.6 | 4.4 | 9 | 43.2-52.6 | 6 |

# *Conclusions - Carbon Fiber Surfaces*

- Removal of Weak Boundary Layer and Increasing Surface Energy Essential First Step
- Surface Treatments Beyond Electro-oxidative Shear Treatments Necessary for Improving Adhesion with High-Modulus Fibers
- A Suite of Characterization Techniques is Necessary for Understanding Fiber Surface Chemistry
- Voltage Contrast XPS is a Valuable Screening Technique for Fiber Surface Research

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# *Carbon Fiber Sizings and Finishes*

Purpose: To protect fiber during processing and improve compatibility with matrix

Common Approaches:

- film-forming polymers (PVA, PVAC, epoxy, starch, phenoxy, polyimide)
- adhesion promoters (silanes, titanates)
- interlayers (grafting, elastomeric)
- reactive (e.g., isocyanates)

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# *Fiber Finish or Improving Thermo-Oxidative Stability of T650-35/AMB-21 Composites*

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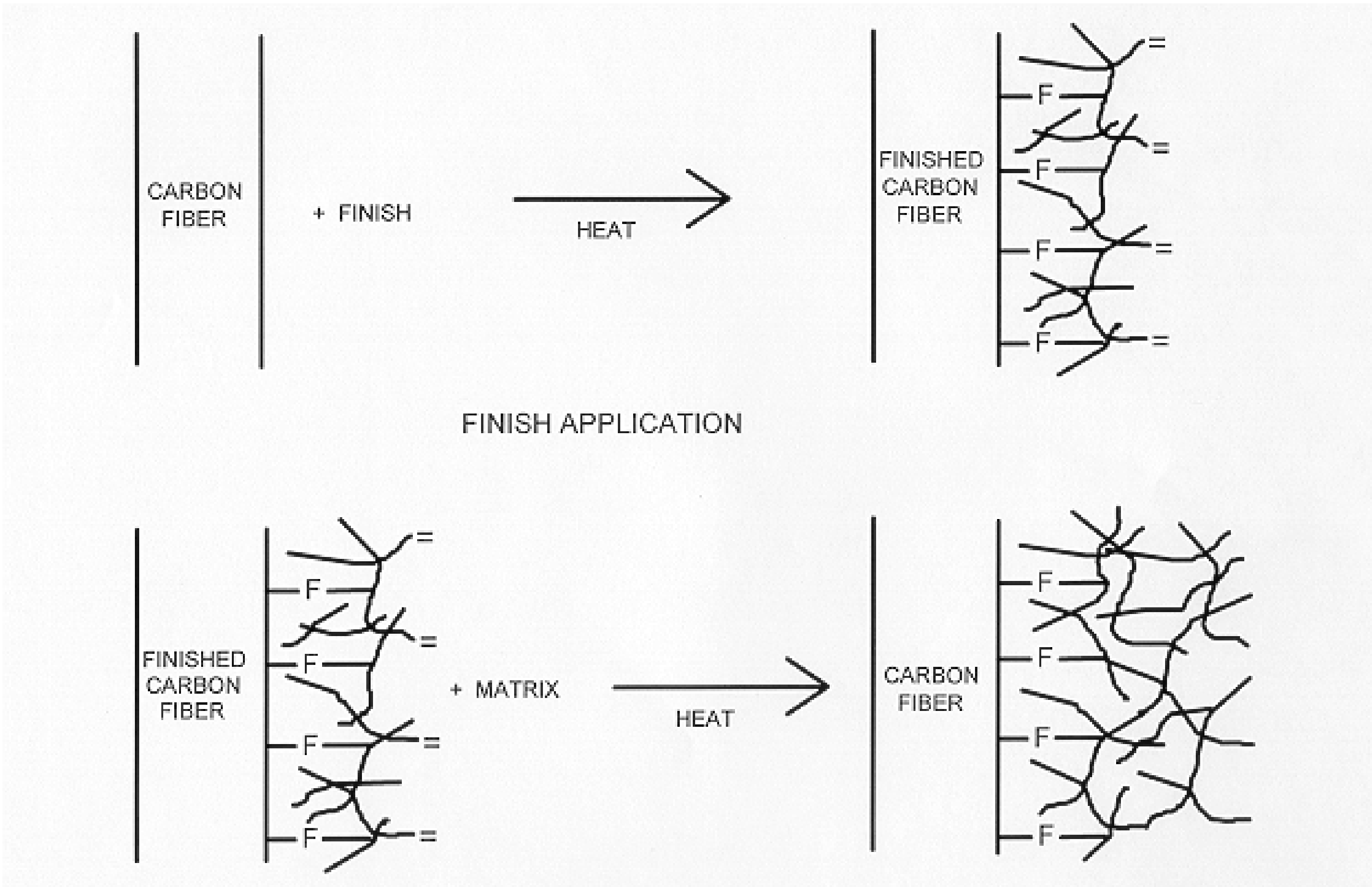
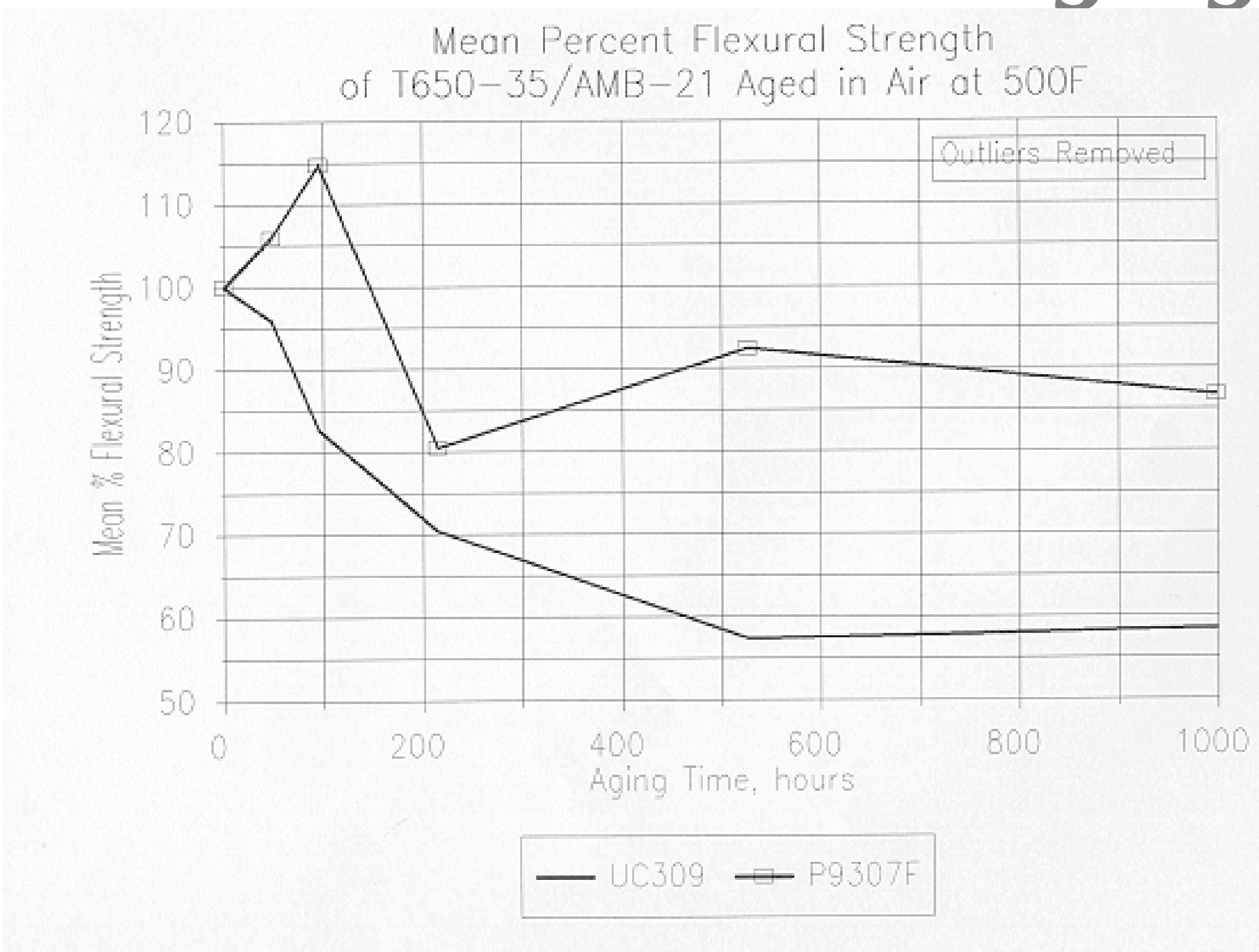
# Reactive Finish Concept




COMPOSITE FABRICATION

# Transverse Flexural Strength Retention vs 260°C Aging



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# *Summary*
# *Sizings and Finishes*

- Considered Necessary Evil
  - necessary, especially for weaving and braiding
  - evil, often compromise composite properties

- Potential Area for Controlling Off-Axis Properties in E-beam Cured Composites Depending Upon Operative Mechanisms