# **IIMEC** 2012

### SUMMER SCHOOL IN ADVANCED COMPOSITE MATERIALS

International Institute for Multifunctional Materials for Energy Conversion

### Composite Materials: Mechanical Behaviour & Testing

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Technological Education Institute of Serres, Greece. July 2 – 6, 2012









# DEFINITIONS

#### • COMPOSITES:

--Multi phase materials with measurable pw fraction of every phase

- Matrix:
  - --Continuous phase

--Role:

- •Stress transfer to other reinforcing phases
- •Environmental protection
- --Classification: MMC, CMC, PMC



- Reinforcement:
- -- Discontinuous or dispersed phsae

-- Role:

MMC: increase sy, TS, creep resistance
 CMC: increase toughness
 PMC: increase E, sy, TS, creep resistance
 -- Classification : particles, fibres, structural



D. Hull and T.W. Clyne, *An Introduction to Composite Materials*, 2nd ed., Cambridge University Press, New York, 1996, Fig. 3.6, p. 47.

# Composites

particles

#### fibres

• Examples:





Structural

AI / SiC MMCs for aerospace automotive industry,

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Adapted from Fig. 16.5, *Callister 6e*. (Fig. 16.5 is courtesy Goodyear Tire and Rubber Company.)

# **Composites: FIBRES I**

particles

fibres

structural

- Continuous aligned fibres
- E.g.
  - --Metals: γ'(Ni<sub>3</sub>Al)-α(Mo) Eutectic composition.

matrix:  $\alpha$  (Mo) (ductile)



#### fibers:γ' (Ni3Al) (brittle)

From W. Funk and E. Blank, "Creep deformation of Ni3Al-Mo in-situ composites", *Metall. Trans. A* Vol. 19(4), pp. 987-998, 1988. Used with permission. --Glass w/SiC fibers E<sub>glass</sub> = 76GPa; E<sub>siC</sub> = 400GPa.



# **Composites: FIBRES II**

fibres

- Discontinuous randomly dispersed 2D fibres
- E.g: Carbon-Carbon

   -manufacturing: fibre/pitch, and pyrolysis at 2500C.
   -use: brakes, turbines, protective shells

particles

(b) (b) C fibers: very stiff very strong C matrix: less stiff view onto plane less strong fibers lie in plane

structural

- Additionally:
- -- Discontinuous randomly dispersed 3D fibres
- -- Discontinuous, 1D fibres

Adapted from F.L. Matthews and R.L. Rawlings, *Composite Materials; Engineering and Science*, Reprint ed., CRC Press, Boca Raton, FL, 2000. (a) Fig. 4.24(a), p. 151; (b) Fig. 4.24(b) p. 151. (Courtesy I.J. Davies) Reproduced with permission of CRC Press, Boca Raton, FL.



# **Composites: Benefits**



# **Composites: A hierarchical structure**



http://www.jeccomposites.com/news/composites-news/progressive-failuredynamic-analysis-composite-structures



# 1. The interface the scale of the interface





Microscopic scale





# Adhesion Mechanisms: Microstructure and Adhesion

• For carbon fibres, adhesion depends on the angel of the basal plane with the symmetry axis of the fibre. The plane edges are usually the sites of chemical reaction.

- Smaller angle means better alignment and reinforcement but worse stress transfer.
- •Oxidative treatment improves adhesion by removing exernal planes and creating edges [**Drzal, 1983**].

#### The nature of the interface[Drzal, 1990] thermal, mechanical and chemical, environments interface: - matrix a function of morphology - unreacted thermal, species 5Å to - impurities mechanical 5000Å - voids - surface and chemical chemistry - topography environment -fiber morphology ber

### **Ceramic Matrix Composites**



### Polymer Matrix Composites (Reifsnider, 1994)



### **Advanced Polymer Matrix Composites**



$$\sigma = \sigma_f V_f + \sigma_m V_m$$

*Interface*: a function of mechanical thermal and chemical enviroment/history

### **Composites: Fracture & Stress Concentration**



The matrix transfers the stress through the interface along the "ineffective length".

Arge "ineffective length" leads to the magnification of the volume of influence of the fracture and increases the possibility of multiple fracture interaction.

Small "ineffective length" leads to high stress concentrations and brittle failure.

# **Interface and strength**



#### (a) Strength as a function of the transfer length

### **Failure of the interface**



mode I







mixed mode





# Simple models of stress transfer

- Shear lag (Cox 1952)
- Constant shear (Kelly 1965)
- Mixed models(Piggott 1980)





# Shear lag (Cox 1952)

• Assuming that the shear force depends linearly on the difference between the actual axial translation and the one that would be if the fibre were not present:

$$S = H(w - w_{\infty})$$
$$\frac{\partial^2 \sigma_z(z)}{\partial z^2} - \beta^2 \sigma_z(z) = -\beta^2 E_f \varepsilon_{\infty}$$

where





Shear lag (Cox 1952)



Constant shear (Kelly 1965)

•Kelly & Tyson [**1965**] assumed that shear at the interface is constant. From the equilibrium equation:

$$\tau_{rz} = -\frac{R\sigma_{fu}}{l_c}$$

•In this case the axial stress coincides with the strength of the fibre which is independent of z.

• *Ic* is the critical length or the length needed to reach the strength of the fibre  $\sigma_f$  before fracture.

•The approach assumes a brittle fibre in a perfectly plastic matrix









# Mixed models (Piggot 1980)



(a) (b) (c)

### Experimental study of the stress transfer



### **Interfacial tests**







- During the pull out tests [Shiryaeva, 1962; Favre, 1972], a length of the fobre is embedded in the matrix.
- The loading of the free end leads gradually to the pull out of the fibre.
- The Force displacement curve may be recorded

- Initially, the load increases linearly with displacement
- Matrix plasticity may lead to non linearities
- After a maximum load value, there is a sudden drop which lasts until the pull out of the fibre [**Li**, **1994**].
- The interfacial strength is defined as a function of the maximum load *Pmax*.:

$$\tau = \frac{P_{max}}{2\pi R\ell}$$

• The maximum stress on the fibre  $\sigma_{max}$  should not exceed its strength  $\sigma_{fu}$  [Broutman, 1969] :

$$\sigma_{max} = \frac{P_{max}}{\pi R^2} \le \sigma_{fu}$$



Displacement



#### •ADVANTAGES [Drzal, 1993]:

- •(i) All fibre types can be tested
- •(ii) All matrix types can be tested

•(iii) Direct measurement of interfacial strength

Debonding strength (MPa)

 $120^{\circ}$ 

500 200 300 400 5 Embedded length (µm) Fig. 7. Pullout tests on T800/5250.

Pull-out test

T800(s/us)/\$250

500

#### **DISADVANTAGES** (Mostly due to the test geometry)

•The wetting of the fibre may create a meniscus that affects the stress field.

•For small fibre diameters (>10  $\mu m)$  the technique is vey difficult.

•The axial fibre alignment is very important

•The maximum load *Pmax* depends on the embedded length. For constant shear, the dependence is linear. However, it has been shown both theoretically [**Gray**, **1984**] and experimentally [**Meretz**, **1993**] **that shear is not constant**.

 The geometry does not simulate the stress field in macroscopic composites because the stresses in the entrance of the fibre may be tensile [Drzal, 1993].
 Many tests should be performed for statistical significance.

### Pull out test: Variations



[Shiryaeva, 1962; Favre,

1972]

(b) fibre matrix

[Qiu, 1993]



[Penn, 1989]



Paul J. Hogg, NOVEL TOUGHENING CONCEPTS FOR LIQUID COMPOSITE MOULDING



## The microindentation test (MIT) [Mandel, 1986]

- MIT is essentially a microhardness test.
- It is performed on a grinded and polished surface
- The force displacement curve is recorded
- Specimen preparation is critical



# The microindentation test (MIT) [Mandel, 1986]

- The strength is assumed arbitrarily as the point when there is interfacial rupture of a percentage of the circumference, [Desaeger, 1993], the change od slope in the force displacement curve [Netravali, 1989], the sudden load drop [Pitkethly, 1993].
- Interfacial strength is derived analytically (e.g. with shear-lag) [Desaeger, 1993] or numerically [Tsai, 1990].
- The major advantage is that the test is performed in macroscopic composites but it is outweighed by the absence of a single failure criterion
- The stress concentration due to the indentor geometry may further complicate the interpretation of the data.

# Fragmentation test [Kelly, 1965]

Fragmentation Gauge Length



Distance Along the Gauge Length x


## Fragmentation test [Kelly, 1965]

•The fibre is embedded in a polymer matrix

•The coupon in loaded in tension until the fibre starts to fracture

•Fragmentation continues until there is saturation, that is no more fractures occur. It is worth noting that if the interface did not fail, the fractures would continue until macroscopic failure of the coupon.

As a result, saturation is connected with the failure of the interface

•During the fragmentation test, fractures are recorded either optically [Waterbury, 1991], or with other techniques (acoustic emission) [Favre, 1990].

• The distribution of the fragment lengths is recorded. Interfacial strength must be derived assuming a stress transfer model.



During tension, the fibre breaks when it reaches its tensile strength.
If *I<sub>c</sub>* is the required length for stress transfer then the distribution of fragment lengths *I<sub>f</sub>* is between *I<sub>d</sub>* 2 and *I<sub>c</sub>* [Narkis, 1988].
To define *I<sub>α</sub>* the strength distribution of the fibre must be known. For a normal strength distribution the transfer length *I<sub>c</sub>* is defined as:

$$l_{c} = 4/3 l_{f}$$

To derive interfacial strength, the stress field must be defined. For constant shear the problem is simplified [**Kelly**, **1965**]:

$$\tau_{rz} = -\frac{R\sigma_{fu}}{l_c}$$



## Fragmentation test [Kelly, 1965]

#### ADVANTAGES

Symmetric stress field [Drzal, 1990].
Large measurement number per test
Sensitivity in different interfacial conditions
Direct observation of the failure events
Qualitative assessment of the stress field and the failure modes
Correlation with the fibre strength
[Gulino, 1991]
Ideal geometry for advanced methods (Raman microscopy, photoelasticity, Acoustic Emission)

#### DISADVANTAGES

•Only brittle fibres in ductile matrices may be tested (at least threefold strain to failure [**Drzal, 1993**]).

•The saturation strain is much larger than the real composite strain to failure, which instigates failure mechanisms not present in real life (debonding [**Wagner**, **1995; DiBenendetto**, **1996**], shear flow [**Nath**, **1996**], or frictional sliding [**Piggot**, **1980**])

•Thermal stresses dominate the stress field [Nairn 1996]

•The test is very difficult for small fibre diametres

## Interface tests: Interlaboratory Scatter [Pitkethly et al., 1993]



# Advanced methods for interfacial testing

- Acoustic Emission
- Raman microscopy
- Acoustic microscopy
- Polarised light microscopy
- SEM



# **Acoustic Microscopy**



Measurement of the local elastic properties near the surface and correlation with the stress transfer

# Raman Frequencies: Dependence on Applied Stress



## Stress transfer for different systems



#### *Elastic Domain:* The ' $\beta$ ' parametre [Cox, 1952]



 $\sigma_z(z)$  : local stress on the fibre

 $\sigma_{\infty}$  : stress at infinity

 $\beta$  : constant

# **Polarised Microscopy**



(a)



# SEM (I)



**(a)** 



**(c)** 



**(b)** 







Fig. 11. SEM analysis of the fracture surface of M40 broken tows: distribution of lengths (in μm) of the pulled-out fibers. (a) Treated M40 at room temperature and 250°C; (b) treated M40 and epoxy-sized M40 at 250°C.

Macroscopic mechanical behavior of the composite lamina



Fabrics carbon, aramid, κτλ.



Plain weave (1 up, 1 down) glass fabric



Eight-harness satin weave (1 up, 7 down)

(2) Filling yarn, running the width of a woven fabric at right angles to the warp

weft direction<sup>(2)</sup>

warp direction<sup>(1)</sup>

(1) In the fabric industry, those fibers or threads in a woven fabric which run lengthwise, or which are parallel to the selvedge

#### **Composite Laminates**



SEM photograph of a typical composite after exposure to water at 333 K for one day (c=0.59%) subjected to 45% of its UTS [O. Gillat, L.J. Broutman, STP 658 (1978)]



For a UD lamina, the composite inhomogeneity (at the fibre matrix level) dominates the micro-failure mechanisms





Typical microstructures of fractured specimens [A.G.Miller, A.L.Wingert, STP 696 (1979)]

#### Macroscopic behaviour:

The mean apparent mechanical properties of the orthotropic lamina or the laminate

The lamina is considered as *a homogeneous anisotropic material* 

(experimentally acceptable for mechanical properties such as *technical elastic constants or strengths*)

The anisotropic composite is usually regarded as a *linear elastic medium until* 



#### Robert Hooke (1635-1703)

•"De Potentia restitutivâ" or "Of Spring" (1678)

# CEIIINOSSSTTUV

### "UT TENSIO SIC VIS"

The present form of Hooke's Law the stress tensor formulation and the equilibrium equations are expressed by:

Augustin Cauchy (1789-1875)



#### **Composite Materials: Symmetries Orthotropic medium**

The elastic anisotropic medium with two mutually perpendicular planes of elastic symmetry. It can be proved that there is a third symmetry plane perpendicular to the other two..



#### Typical orthotropic medium : woven fabric



#### Transversely isotropic medium

It posesses an axis of *elastic symmetry*:

All directions perpendicular to that axis are elastically equivalent i.e all planes perpendicular to that axis are isotropic



#### Typical transversely isotropic medium: Fibre tow



#### **Isotropic medium**

All directions are elastically equivalent



24/25

#### Typical isotropic medium: Particulate reinforced composite



# Elastic properties of a lamina

- Loading parallel to the reinforcement
- Loading perpendicular to the reinforcement
- Loading in an angle to the reinforcement





# Loading parallel to the reinforcement

For a tensile stress parallel to the reinforcement assuming that:

- Interfacial bond is perfect,
- The strain  $\varepsilon_1$  of the matrix equals that on the fibre
- The matrix and the fibre are linear elastic solids:

Which phase undertakes the maximum stress?

$$\sigma_{_f} = E_{_f} arepsilon_{_1}$$
 and  $\sigma_{_m} = E_{_m} arepsilon_{_1}$ 

$$P = P_f + P_m$$
 and  $P_m = \sigma_m A_m$ 

$$\sigma_1 = \frac{P}{A}$$
 and  $P_f = \sigma_f A_f$ 

### **Rule of Mixtures**

$$E_{1} \equiv E_{//} = E_{f}V_{f} + E_{m}(1 - V_{f})$$



# Loading perpendicular to the reinforcement

For a tensile stress parallel to the reinforcement assuming that:

- Interfacial bond is perfect,
- The strain  $\varepsilon_1$  of the matrix equals that on the fibre
- The matrix and the fibre are linear elastic solids:

Which phase undertakes the maximum stress?

$$\sigma = E_{_f} arepsilon_{_f}$$
 and  $\sigma = E_{_m} arepsilon_{_m}$ 







## Corrections



Where M is the composite property and  $\boldsymbol{\xi}$  a parameter depending on reinforcement attributes:



## Stress concentration and strain magnification (Kies)



 $\pi$ 

2+2

 $-\frac{E_m}{E_{\star}}$ 

S

 $E_m$ 

 $E_{f}$ 

 $\mathcal{E}_{x}$ 

 $\overline{\overline{\mathcal{E}}_x}$ 

 $\pi$ 

2 +

 $\frac{\mathcal{E}_x}{\overline{\mathcal{E}}_x}$ 



# Strain Magnification: Glass polyester





# Long fibre composites with random orientation (Nielsen και Chen 1968)

$$\overline{E} = 2\pi \int_{0}^{\pi/2} E(\theta) d\theta$$

 $E(\theta)$ : Stiffness of UD lamina as a function of Theta for constant Vf.

$$\frac{1}{E(\theta)} = \frac{1}{E_1} \cdot C^4 + \left(\frac{1}{G_{12}} - \frac{2\nu_{12}}{E_1}\right) \cdot C^2 \cdot S^2 + \frac{1}{E_2} \cdot S^4$$

where:  $C = cos\theta$ ,  $S = sin\theta$ 

## Long fibre composites with random orientation

• Empirical relationships:

$$\overline{E} = \frac{3}{8}E_1 + \frac{5}{8}E_2 \qquad \overline{G} = \frac{1}{8}E_1 + \frac{1}{4}E_2$$

The effect of  $V_f$  comes through  $E_1$  and  $E_2$ .

## Long fibre composites

Material	E <sub>1</sub> (GPa)	E <sub>2</sub> (GPa)	<b>G</b> <sub>12</sub> ( <b>GPa</b> )	v <sub>12</sub>
Glass-polyester	35 - 40	8-12	3,5-5,5	0,26
Type I carbon-epoxy	190-240	5-8	3 - 6	0,26
Kevlar 49 - epoxy	65 - 75	4 - 5	2 - 3	0,35

Typical  $E_1$ ,  $E_2$ ,  $G_{12}$  & $v_{12}$ For composite types


glass fibre- polyester resin with Vf=0.30 [D. Hull, 1981]

### **Elastic properties of short fibre composite**

 Ineffective length correction (shear Lag)

$$E_1 \equiv E_{//} = \eta_l E_f V_f + E_m (1 - V_f)$$



Shoor	Υλικό	l (mm)	G <sub>m</sub> / E <sub>f</sub>	r (μm)	$\mathbf{V_{f}}$	η
lag	Carbon-epoxy	0,1 1,0	0,005 0,005	8	0,3 0,3	0,20 0,89
	Glass-nylon	10,0 0,1	0,005	8	0,3 0,3	0,99
		1,0 10,0	0,010 0,010	11 11	0,3 0,3	0,89 0,99

### Elastic properties [Dingle 1974]

Fibre length 1 (mm)	V <sub>f</sub>	E <sub>//</sub> Theoretical (GPa)	E <sub>//</sub> Experimental (GPa)	η <sub>ι</sub>
1	0,49	194	155	0,80
4	0,32	128	112	0,87
6	0,42	167	141	0,84

### Tensile strength of long fibre composites

Typical strength of UD laminates (V<sub>f</sub> 0.50)

Material	σ*//T (MPa)	σ*// C (MPa)	σ*⊥T (MPa)	σ*⊥C (MPa)	τ*# (MPa)
Glass-polyester	650-750	600-900	20-25	90-120	45-60
Type I carbon-epoxy	850-1100	700-900	35-40	130-190	60-75
Kevlar 49-epoxy	1100-1250	240-290	20-30	110-140	40-60

T: Tension, C: Compression

### **Deterministic fibre strength**

From the rule of mixtures:

$$\sigma_{\parallel} = \sigma_f V_f + \sigma_m (1 - V_f)$$

$$\sigma_{||} = E_f \varepsilon_{||} V_f + E_m \varepsilon_{\perp} (1 - V_f)$$

#### **Failure Possibilities:**

1. 
$$\epsilon_{f}^{*} > \epsilon_{m}^{*}$$
  
2.  $\epsilon_{f}^{*} < \epsilon_{m}^{*}$ 

### **Uniform fibre strength**

#### For small V<sub>f</sub>:

σ\*// depends on σ\*<sub>m</sub>.
The matrix fails first
The fibres take over but cannot take the load and fail

$$\sigma_{\prime\prime}^{*} = \sigma_{f}^{'} V_{f} + \sigma_{m}^{*} (1 - V_{f})$$

1. 
$$\varepsilon_{f}^{*} > \varepsilon_{m}^{*}$$

#### For largeV<sub>f</sub>:

Since  $E_f >> E_m \Rightarrow$ 

- The matrix undertakes a small load fraction
- The matrix fails
- The load is transferred to the fibres until they fail

$$\sigma_{\prime\prime}^* = \sigma_f^* V_f$$





### **Uniform fibre strength**

#### For small V<sub>f</sub>:

- •The fibres break.
- •The matrix takes over the additional load
- •The efficient cross section is reduced by the fibre breaks

1. 
$$\varepsilon_{f}^{*} < \varepsilon_{m}^{*}$$

#### For largeV<sub>f</sub>:

Since  $E_f >> E_m \Rightarrow$ 

- The fibres break.
- The matrix cannot take over the additional load
- The composite fails

$$\boldsymbol{\varepsilon^*}_{\mathbf{f}} < \boldsymbol{\varepsilon^*}_{\mathbf{m}} : \text{For equal } V_{\mathbf{f}}: \quad V'_f = \frac{\left(\sigma_m^* - \sigma_m'\right)}{\left(\sigma_f^* + \sigma_m^* - \sigma_m'\right)}$$



# Variable fibre strength

### • The fibre is brittle

- Fracture occurs at the flaw sites where strength is reduced
- The strength reduction is stochastic
- How does the strength depend on the fibre size? (volume or length for constant cross section);
- Experimental campaign: strength as a function of length:
- definitions:
  - $\sigma_{f}^{*}$  fibre strength
  - 2r diametre,
  - I length
  - $-\sigma_1$  minimum fibre strength
  - $-\sigma_{u}^{-}$  maximum fibre strength

### **Weibul distribution**



# For a fibre bundle (Coleman 1958)

- Assumptions:
  - $-\alpha$ ) the fibres are distinct and have equal cross sections
  - $\beta)$  For stress  $\sigma_{i<}\sigma_{l}$  the fibre deform equally and do not break
  - $-\gamma$ ) as the load increases the weaker fibres break and the intact fibres take up the load

# Fibre bundle strength(Coleman 1958)

- The maximum fracture load occurs when the developing stress on the remaining fibres reaches  $\sigma_{\rm u}$  and the bundle fails
  - The strength,  $\sigma_{\rm b},$  of the bundle is less than the mean fibre strength
  - The reductions depends on the spread of the fibre strength of individual fibres:



# Cumulative weakening (Rosen)



The statistical distribution of fibre strength leads to global weakening and failure:

$$\frac{\sigma_{cum}^*}{\overline{\sigma}} = \left(\frac{1}{l_c m e}\right)^{1/m} = \frac{1}{\Gamma\left(1 + \frac{1}{m}\right)}$$

 $\sigma_{cum}$ : fibre strength  $I_c$ : critical length

# Statistical strength (Carbon / Epoxy)



# Crack propagation in UD laminates



Stress
 concentration
 leads to
 transverse
 cracking

Εικόνα 6.19 Θραύση γειτονικών ινών λόγω συγκέντρωσης τάσεων στο άκρο της πρώτης ρωγμής.

### Crack-fibre interaction

- Possibilities
  - a) The crack propagates in the matrix.
  - b) The matrix around the crack yields creating a plastic zone along the fibre.
  - c) The interface fails and the fibre retracts in the matrix.



# **Crack propagation**







And meets the fibre



The matrix debonds from the fibre

- The stress
   concentration
   is proportional
   to (c/ρ)<sup>1/2</sup>
  - ρ is the radius of curvature at the crack tip
  - 2c is the crack length

# Stress field at the crack



The maximum tensile stress
 σ<sub>1max</sub> perpendicular to the
 crack propagation and the
 maximum tensile stress σ<sub>2max</sub>
 parallel to the crack path
 develop simultaneously at
 the crack front

# Stress field at the crack



Η ρωγμή συναντά την ίνα

For isotropic materials,

 $-\sigma_{1\max}/\sigma_{2\max} \sim 5$ 

- For anisotropic materials the ratio depends on the crack orientation and the degree of anisotropy.
  - For carbon fibre-epoxy with  $V_f = 0.5$
  - $-\sigma_{1\max}/\sigma_{2\max} \sim 48$

$$-\sigma_{1\max}/\tau_{\max}=11$$

$$-\tau_{\rm max}/\sigma_{\rm 2max}$$
 =4.4.



# Failure at the vicinity of the crack

- The process depends on the values of  $\sigma//*$ ,  $\sigma \perp *$ ,  $\tau_{\#}^*$ . :
  - $\alpha$ )  $\sigma//*/\sigma \perp * > \sigma_{1max}/\sigma_{2max}$ : tensile failure parallel to the interface will precede fibre fracture
  - β) σ//\* / τ<sub>#</sub>\* > σ<sub>1max</sub> / τ<sub>max</sub> : shear failure will precede fibre fracture,
  - γ) τ<sub>#</sub>\* / σ ⊥ \* > τ<sub>max</sub> / σ<sub>2max</sub> : tensile failure at the interface is more probale than shear failure.

# Typical values for laminates (Vf~50%)

 $p_{1} \in r$ 

Υλικό	σ* <sub>//</sub> Τ (MPa)	σ*// C (MPa)	σ*⊥T (MPa)	σ*⊥C (MPa)	τ*# (MPa)
Glass-polyester	650-750	600-900	20-25	90-120	45-60
Type I carbon-epoxy	850-1100	700-900	35-40	130-190	60-75
Kevlar 49-epoxy	1100-1250	240-290	20-30	110-140	40-60

# Transverse tensile strength

- Often, transverse strength is less than the matrix strength
  - Assumptions:
    - Zero interfacial strength at the transverse direction
    - Tough matrix (resisting crack propagation)
  - The strength is that of the matrix with reduced effective cross section



# For square distribution...



Derek Hull, 1981

### Transverse tensile strength



Εικόνα 6.36: Διάδοση εγκάρσιας ρωγμής σε μία στρώση glass fibre-polyester resin. Η ρωγμή έχει συναντήσει μία περιοχή πλούσια σε ρητίνη οπότε παρατηρείται διαπλάτυνση του άκρου της και ταυτόχρονα διαρροή της ρητίνης. ΠΗΓΗ: D. Hull, An Introduction to Composite Materials, Cambridge Univ. Press 1981



#### BRITISH STANDARD

# Plastics — Determination of tensile properties

Part 5. Test conditions for unidirectional fibre-reinforced plastic composites

BS EN ISO 527-5 : 1997 BS 2782 : Part 3 : Method 326G : 1997



#### Geometry of test specimens



#### **CRP** materials

MATERIAL	CODE NAME	LAYUP
	GOBU	[0] <sub>T</sub>
GOB	GOBM	$[90_2]_{\rm T}$
	GOBR	[±45] <sub>s</sub>
	HEXU	$[0_2]_{T}$
HEX	HEXM	[90 <sub>3</sub> ] <sub>T</sub>
	HEXR	[±45] <sub>s</sub>

b



Axial stress vs. axial strain UD laminate



#### Transverse strain vs. axial strain UD laminate













Failed HEXM coupons



#### Coupons D1, failed in tension



#### Coupons D2, failed in tension



### Longitudinal Compression.

Difficult to assess because it depends : •On the compressive properties of both fibre and matrix,

✓On the interface✓On the void content.

The failure mode depens on ✓The lateral fibre support, ✓The volume fraction ✓The matrix properties.

# Prediction of compressive strength

- The fibres are regarded as Euler columns.
- The matrix prevents buckling and increases the critical buckling load
- The elastic properties of the matrix determine the critical buckling load.
- The theoretical models are based on fibre buckling or shear matrix failure

$$EIy'' = -M$$

$$\frac{d^2 y}{dx^2} = -\frac{M}{EI} = -\frac{P(y+e)}{EI}$$
Euler Buckling
$$y+e = C_1 \sin Kx + C_2 \cos Kx \quad O\piov: \quad K = \sqrt{P/EI}$$
From the boundary conditions on A & B:
$$C_2 = e \qquad C_1 = \frac{e(1-\cos KL)}{\sin KL}$$

$$y+e = \frac{e(1-\cos KL)}{\sin KL} \sin Kx + e\cos Kx$$

To maximise y, the denominatior must be infinite or

sinKL=0, or KL=0,  $\pi$ ,  $2\pi$  ...

$$KL = L\sqrt{P/EI} = \pi \Longrightarrow P = \frac{\pi^2 EI}{L^2}$$

**Critical buckling load.** 

The critical buckling load is less for shorter columns, stiffer materials and open cross sections!


# Out of phase failure

- The fibres buckle out of phase
- The matrix is subjected to tension and compression in the transverse direction
- Failure depends on critical buckling load
- Assumptions:
  - Strain in the y direction is independent of y
  - The matrix is essentially unloaded in comparison to the fibres



# In phase failure

- The fibres buckle in phase
- The matrix is subjected to shear
- Failure depends on the shear strength of the matrix
- Assumptions:
  - Strain in the y direction is independent of y
  - Gf >> Gm or the fibres essentially remain undeformed





$$\sigma_{f_{cr}} = 2 \sqrt{\frac{V_f E_m E_f}{3 (1 - V_f)}} (2.2)$$

# In phase failure









Compressive strength of unidirectional glass/epoxy composites  $\sigma_{\rm c}$  as a function of V<sub>f</sub> Out of phase failure is valid for low V<sub>f</sub>. For typical  $0.6 < V_f < 0.7$ the strength is calculated between 450 & 600 Ksi (3100 & 4150 MPa), at a strain to failure > 5%!

fibre volume fraction

# **Compressive failure**

- Other affecting parameters:
  - Local inhomogeneities in the fibre Vf,
  - Void and defect content.,
  - Fibre misalignment and curvature,
  - Weak or bad interface which instigates debonding and decreases critical buckling load,
  - Viscoelastic matrix behavior, or reduced Gm,
  - Anisotropic fibres with weak transverse properties (carbon or Kevlar) or non linear compressive behavior.

# **Compressive Failure** Modified models $\sigma_{\rm c}$ as a function of V<sub>f</sub>

- Gm is a linear function of compressive strain (Dow & Rosen)

• The matrix is perfectly plastic (Lager & June)  $\sigma_c = c \frac{G_m}{1 - V_f} (2.5)$ • Shear controlled model (M.R.Wisnom)  $\sigma_c = \frac{G_m \gamma_m}{V_m (\gamma + \alpha)} (2.6)$ 

Curvature and misalignment (Hahn & Williams)

$$\sigma_{c} = V_{f} G_{LT} \frac{\gamma_{LT}}{\gamma_{LT} + \frac{\pi f_{o}}{e}} (2.7)$$

- $\sigma_c$  : compressive strength.
- $V_f$ : volume fraction.
- $G_{LT}$ : shear modulus of the composite.
- $\gamma_{LT}$  : shear strain.
- $f_0/e$ : initial fibre deflection to wavelength.

Effect of fibre misalignment on predicted compressive strength of unidirectional XAS/914, [7].



misalignment angle (º)

# Compressive failure (mechanisms)

- Fibre buckling leads to their failure.
  - Buckled fibres are both subjected to tension and compression
  - Brittle fibres (e.g. carbon fibres) fail when their strength is reached locally creating a characteristic failure zone
  - Viscoelastic or plastic fibres create a plasticity zone when the yield stress is reached





### **Maximum Shear**

- If Shear failure precedes buckling Failure: For shear stress  $\tau$ :  $\tau = \sigma_{IIC} \sin\theta \cos\theta$
- Shear is maximum for  $\theta = 45^{\circ}$ ,  $\tau_{max} = \sigma_{||C}/2$
- From the rule of mixtures:

$$- \sigma_{||C}^{*} = 2 \left[ V_{f} \tau_{f}^{*} + (1 - V_{f}) \tau_{m}^{*} \right]$$

Where  $\tau^*_{f}$  kai  $\tau^*_{m}$  the shear strength of the fibre and matrix respectively









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### direct end loading

### shear loading

### mixed shear/direct loading

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Figure 6 Wyoming modified celanese compression test fixture.

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# ASTM Standard D695 compressive test fixture for rigid plastics, [17]



# Imperial College compression test rig and specimen







Typical failure modes under static compressive load.





Typical failure mode under static compressive load



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# Weibul distribution of experimental results (XAS/914 UD composite laminate).



#### Macrobuckling of the specimen prior to failure. "negative" side "positive" side specimen 29 specimen 29 1,400 1,600 1,400 1,200 1,200 1,000 stress(MPa) 00 00 00 stress(MPa) 8 8 00 90 400 400 200 200 0 0 0.005 0.01 0.015 0.02 Ő0 0.005 0.01 0.015 0.02 'n strain(%) strain(%)

Modulus reduction with increasing strain.

### ASTM D3410-87(ITRII test)

specimen 33



## Shear Properties.

The shear properties are (I): Shear modulus (II): Shear strength ✓Composites are anisotropic: ✓Three types of shear

✓ interlaminar

- $\checkmark$  in plane longitudinal
- ✓ intralaminar

#### Shear in principal planes:



Shear planes: (2-3), (1-3) каі (1-2)

### Shear testing.

### ✓ interlaminar( $\tau_{13}$ )



### Shear testing.

## ✓In plane ( $\tau_{12}$ )



### Shear testing

### Intralaminar ( $\tau_{23}$ ) – no existing standard



•Shear tests are difficult: Uniform stress field is hard to achieve

•Few standard tests. No universally accepted standards for all types and structures of long fibre composites

#### **In-plane shear test methods:**

<ul> <li>uniaxial tension of a ±45 laminate</li> </ul>	•ISO 14129
•Iosipescu shear specimen (V-notched beam, VNB method)	•ASTM D5379M-98
•uniaxial tension of a 10° off-axis specimen	•(None)
•two- and three-rail shear tests	•ASTM D4255M-83
<ul> <li>torsion of thin-walled tube</li> </ul>	•ASTM D5448M-93
<ul> <li>twisting of a flat laminate</li> </ul>	•ASTM D3044-94



# ±45° Test

Symmetric ±45° laminate in tension:

$$-\tau_{12} = 1/2 \sigma_{xx}$$

$$-\gamma_{12} = \varepsilon_{xx} - \varepsilon_{yy}$$

- The test is accepted by all standards organisations
- Both for woven fabric and prepregs



# ±45° Test

- Shear stress vs. shear strain is calculated by:
  - $\tau_{12} = 1/2 \sigma_{xx}$
  - $\ \gamma_{12} = \epsilon_{xx} \epsilon_{yy}$
- Typical curve for Boron / epoxy

Advantages Simple coupon geometry Easy to perform Disadvantages The coupling of the shear stresses between the laminae affect the measurements Minor misalignment results to large deviations

# double V notch – Isopescu test





- Pure shear in the plane defined by the two notches
- Usually emplyed for  $0^{\circ}$  or  $90^{\circ}$  laminates
- For 90° laminates, it is very reliable
- Shear strength is derived by diviiding the load with the shear cross section
- The local stress field may lead to erroneous results
- The positioning of strain gauges is crucial
- Usually yields smaller values than the cylindrical beam

# rail shear tests



# rail shear test



- A rectangular plate is fixed in side beams while the longitudinal direction is free
- The load induces shear stresses For pure shear:

$$\varepsilon_{xx} = \varepsilon_{yy} = 0$$
  $\gamma_{xy} = 2\varepsilon_{45^{\circ}}$ 

 $\boldsymbol{\gamma},$  the shear strain

The shear stress is :

- b width
- t thickness

$$\tau_{xy} = \frac{P}{bt}$$

Shear strain is measured at  $45^{\circ}$  to the rail

$$\gamma_{xy} = 2\varepsilon_{45^{\circ}}$$

### Interlaminar shear: The ILSS test.

✓From the beam theory, flexural stress can be written as:

$$\sigma_{xx} = \frac{3P}{2wt} \left(\frac{S}{t}\right)$$

✓The maximum shear is

$$\tau_{xz} = \frac{3P_{\max}}{4wt}$$





# Interlaminar shear: The ILSS test.

✓Whereas the flexural stress decreases with s/t the maximum shear stress is independent of it.

$$\sigma_{xx} = \frac{3P(S)}{2wt(t)} \qquad \qquad \tau_{xz} = \frac{3P_{\max}}{4wt}$$

### For small S/t shear failure is more probable

# Shear or Flexure;



#### From the elastic beam theory:

 Maximum stress (compressive or tensile)-top and bottom surface respectively:

$$\sigma_{ult} = \frac{3P_{\max}S}{2wt^2}$$

Maximum shear stress – neutral axis:

$$\tau_{ult} = \frac{3P_{\max}}{4wt}$$

• Maximum shear to maximum bending stress:

$$\frac{\tau_{ult}}{\sigma_{ult}} = \frac{S}{2t}$$

# Shear or Flexure;


## Shear strength:

#### ✓For small S/t:



# Shear strength:

1. Interlaminar Shear



The coupon fails in shear



2. Flexure

3. Inelastic Deformation





- Two geometries
- (ASTM D3244):
  Curved coupon
  - Flat coupon



# **ILSS tests:**

#### Standards

Method	W	t	S/t	L	d <sub>1</sub>	d <sub>2</sub>	Speed (mm/min)
ASTM	10	2	5	14	3.2	6.4	1.3
BSI	*	*	*	12	6	6	1
CRAG	*	*	*	20	*	*	*

Advantages:

Simple to perform Simple test configuration Comparable data from all standards Disadvantages

 ✓The geometry defines failure The through thickness shear distribution is not parabolic
 ✓Difficult to assess acceptable failure mode



Figure 14 Fracture mechanics failure Modes I, II, and III.

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Double cantilever beam flexure test (tension)



End-notched flexure test (shear)



Figure 15 Interlaminar fracture toughness test methods for composite materials.

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Schematic diagram of apparatus

Figure 16 Mixed-mode interlaminar fracture toughness specimen and test fixture (after Crews and Reeder, 1988).

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Mode I - DCB Specimen





Mode II - 4ENF Specimen\*



\*Rod Martin, MERL - Barry Davidson, Syracuse University





#### Carbon/nylon Carbon

#### **Plain glass DCB test specimen**



#### Fibre bridging mainly due to polyester stitching



DCB test on Glass fibre : PP/Epoxy ep1



#### Four point end-notch flexure test





# Carbon nylon Carbon nylon Carbon (modified specimen)

#### **Fracture mechanics**

• Mixed Mode I/II - MMB Specimen\*





crack opening mode l

+ in plane shear mode ll

#### • ASTM D6671



#### **Fracture mechanics**

- Mode III ECT Specimen\* tearing mode III
- Failure surface G<sub>c</sub> =G<sub>c</sub>(G<sub>lc</sub>, G<sub>llc</sub>, G<sub>llc</sub>)\*\*



• Standard in development

\*James Ratcliffe,NRC at NASA Langley Research Center

\*\*James Reeder, NASA Langley Research Center

## **Durability Testing of Polymer Composites**

PAUL T. CURTIS, DERA, Farnborough, UK Volume 5, Ch 5.08 of Comprehensive Composite Materials

FATIGUE TESTING Tensile tests **Compression tests** Flexural tests Shear tests **Biaxial fatigue testing** Machines and Control Modes Presentation of Data Monitoring Fatigue Damage Growth Microscopy Ultrasonics X-radiography Thermography Potential Problems with Fatigue Testing Stress concentrators Frequency effects Edge effects **Environmental effects** 

IMPACT TEST METHODS High-energy Impact Test Methods Flexed-beam tests The drop-weight impact test Data analysis and failure modes Low-energy Impact Test Methods Ballistic impact tests Drop-weight test **Residual Strength After Impact** Crashworthiness **CREEP TEST METHODS Creep Behavior of Polymer Composites** Creep Test Methods

#### Impact – Low velocity











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#### Impact – Low velocity



i ii (Ellis 1996)

Figure 19 Schematic load-time and energy-time curves.

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# Impacted plain glass fibre specimens.







GF2/UP

GF1/EP1

GF1/EP2

# Through penetration impact Impacted glass fibre/polypropylene specimens.







PP1/UP

PP1/EP1

PP1/EP2

# Through penetration impact Impacted polypropylene fibre/polypropylene specimens.



Fig. 10. Typical impact penetration damage at different compaction temperatures and pressures. Specimen (a) consolidated at lower temperature and pressure (140 °C, 0.1 MPa), shows large amounts of fibrillation and delocalised deformation giving a circular hole, while specimen (b) consolidated at higher temperature and pressure (160 °C, 11.4 MPa) shows very localised damage and breakage along tape boundaries giving a characteristic star-shaped hole.



Fig. 12. Illustration of the out of plane deformation of all-PP composites. Specimen (a) consolidated at lower temperature and pressure (140 °C, 0.1 MPa), shows large amounts out of plane deformation and tape pull through, while specimen (b) consolidated at higher temperature and pressure (160 °C, 11.4 MPa) shows very localised damage and limited out of plane deformation.



Fig. 14. Stress dispersion in different angles to tape direction showing the ease of plastic deformation during loading at  $\pm 45^{\circ}$  to tape direction.

#### Impact – Ballistic



#### Impact – Ballistic





Figure 5. Scheme of the role of the interface on the erosion of UD fiber-reinforced composites under parallel (Pa) and perpendicular (Pe) impact conditions.

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CEM micrographs taken on the graded surface of compositos impacts

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Fig. 4. Scanning electron micrographs taken on the eroded surfaces of GF/PP composites with 40 wt.% fibre content (erosion at 30° angle for 600 s)—illustration of orientation influence on surface topography: (a) parallel (Pa) erosion direction and (b) perpendicular (Pe) erosion direction.



Figure 2 Possible mechanisms of solid particle erosion; (a) abrasion at low impact angles, (b) surface fatigue during low speed, high impingement angle, (c) brittle fracture or multiple plastic deformation during medium speed, large impingement angle, (d) surface melting at high impact speeds, (e) macroscopic erosion with secondary effects (after [12]).

## **Compression after impact**





## Compression after Impact : specimens and jig



#### Residual strength after impact







Figure 21 Typical residual compressive strength data vs. impact energy for a noncrimp fabric and comparable UD carbon fiber/epoxy quasi-isotropic laminate (after Kemp and Curtis, 1996).

#### Residual strength after impact



*Key resin properties.....strain to failure* 

## Fatigue



Figure 1 Typical applied stress-strain-time diagram for fatigue loading.



Figure 2 Coupons with varying gauge profiles tested: (a) statically, (b) fatigue.

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## Fatigue



Figure 4 UD fatigue coupon-schematic showing split growth back to grips.



Figure 7 Interlaminar shear test specimen.



Figure 6 Typical antibuckling guide.

## Fatigue



Figure 9 (a) Fatigue cycling under stress or strain. Differences in (b) stress-controlled and (c) straincontrolled fatigue tests of polymeric composites.

## Creep





#### **Four Design Classes**

where creep is important

 Displacement-limited applications, in which precise dimensions must be maintained (the disks and blades of turbine)

 Rupture-limited applications, in which dimensional tolerance is relatively unimportant, but fracture must be avoided (as in pressure-piping)

• Stress-relaxation-limited applications, in which an initial tension relaxes with time (as in the pretensioning of cables or bolts)

• Buckling-limited applications, in which slender columns or panels carry compressive load

(upper wing skin of an aircraft)

### Creep



Figure 8.3 Strain time curve for a creep test

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## Creep



Figure 8.2 Typical creep test set-up

## Test Methods for Physical Properties

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FIBER/VOID VOLUME FRACTIONS AND FIBER DIRECTION

MOISTURE ABSORPTION AND CONDITIONING OF COMPOSITE MATERIALS

Mechanism of Moisture Absorption

Effects of Moisture Absorption

THE GLASS TRANSITION

DIFFERENTIAL SCANNING CALORIMETRY

DYNAMIC MECHANICAL ANALYSIS

THERMOPHYSICAL PROPERTIES

POLYMER COMPOSITE MATERIAL DEGRADATION

# FIBER/VOID VOLUME FRACTIONS AND FIBER DIRECTION





## FIBER/VOID VOLUME FRACTIONS AND FIBER

DIRECTION



#### **Moisture absorption**



Figure 1 Plot of moisture content vs. time<sup>1/2</sup> (and determination of diffusion constant) for a material obeying Fick's law.

#### **Moisture absorption**



Figure 2 Percentage mass increase for five composite materials in steam as a function of the square root of time (*Comp. Sci. and Tech.*, 1996, 56, 977, reproduced by permission of Elsevier).

## **Environmental testing**



Temperature and relative humidity representative cycles reproduced after Reynolds and Mc Manus (Reynolds and Mc Manus 2000)

## **Environmental testing**



Weight gain versus square root of time for the neat and modified epoxy matrices reproduced after Barkoula et al. (Barkoula et al. 2009)

DSC



Figure 6 A DSC thermogram showing three transitions for a polyethyleneterephtalate specimen under dynamic heating (recreated with permission from TA Instruments Ltd.).

## DSC



Figure 7 DSC thermogram of a high-temperature epoxy/carbon fiber prepreg (using EFA-CFC-TP-017), indicating the key thermal transitions.



Figure 13 Diagram showing the phase angle between stress and strain in a viscoelastic material.



Figure 3 Dynamic mechancial analysis of a carbon/epoxy specimen showing the major response curves and the determination of  $T_g$  (onset), peak E" and peak tan  $\delta$ .



Figure 14 DMA thermograms of polycarbonate showing the variation in E' (drop off) and E'' (peak) with the test frequency (recreated with permission from TA Instruments Ltd.).



Figure 20 DMA thermograms showing the testing of wet conditioned specimens of an RTM laminate (a) without binder and (b) including a binder system.