

# Plastics and Composite Materials

Anité Pérez Fontenla

MECHANICAL & MATERIALS ENGINEERING FOR PARTICLE ACCELERATORS AND DETECTORS

## **Plastic (polymeric) Materials**



We rely on this material category for clothing, protection, and transport in our **everyday life**, but plastics are also key components of **cutting-edge technologies** that have powered the space program, created the bulletproof vest or are widely used in surgical implants to mention a few

#### Why are they so interesting?

- Easy to fabricate, durable, versatile, resistant to corrosion, lightweight, resilient...
- Provide good protection and preservation...
- Thermal, electric, acoustic insulation value...
- Different formats like fibers, sheets, foams, or complex moulded parts





## What are polymers?

**Polymeric materials** (commonly known as **plastics**) consist of macromolecular organic compounds that can be manufactured **synthetically** or transformed from **natural** products

**Polymer** refers to molecules held together by covalent bonds and composed by small units which are repeated many times to form very large molecules

- **Monomer:** HC molecules derived from fossil fuels (ethylene, propylene, butylene...) from which the polymer is synthesized
- **Repeat unit ("mer"):** Entity successively repeated along the chain

The **polymerisation** process consists in the aggregation of monomers that are chemically bonded into chains. There are two different mechanisms for polymerisation: **Addition** and **Condensation** 





*Polyethylene chain structure indicating the zigzag back-bone structure. W. D. Callister* [1]

Natural polymers: cotton, cellulose, latex... 19<sup>th</sup> century → Chemically modified: rayon, celluloid, vulcanized rubber 1907 first totally synthetic polymer: Bakelite















A. Pérez | Plastics and Composite Materials | CAS-2024

## What are polymers?

**Polymeric materials** (commonly known as **plastics**) consist of macromolecular organic compounds that can be manufactured **synthetically** or transformed from **natural** products

**Polymer** refers to molecules held together by covalent bonds and composed by small units which are repeated many times to form very large molecules

- **Monomer:** HC molecules derived from fossil fuels (ethylene, propylene, butylene...) from which the polymer is synthesized
- **Repeat unit ("mer"):** Entity successively repeated along the chain

The **polymerisation** process consists in the aggregation of monomers that are chemically bonded into chains. There are two different mechanisms for polymerisation: **Addition** and **Condensation** 





*Polyethylene chain structure indicating the zigzag back-bone structure. W. D. Callister* [1]

Natural polymers: cotton, cellulose, latex... 19<sup>th</sup> century → Chemically modified: rayon, celluloid, vulcanized rubber 1907 first totally synthetic polymer: Bakelite

Twisted chain



a) Atomic structure of the polyethylene molecule and b) An overall view of the molecule. M. Doi [2]

Analysis techniques (molecular level): Infrared (IR) and Nuclear Magnetic Resonance (NMR) Spectroscopy





### **Polymers: Structure**



The molecular chains can **bend**, **coil**, and **entangle**. This structure leads to unique properties (i. e. the large elastic extensions in rubber)

Some of the **mechanical** and **thermal characteristics** of polymers are a function of the ability of chain segments to experience rotation in response to applied stresses or thermal vibrations

**Rotational flexibility** is dependent on repeat unit structure and chemistry

<u>Example:</u> Polystyrene (PS) chains are more resistant to rotational motion than polyethylene (PE) chains due to the introduction of a (bulky) phenyl side group of atoms that restricts the rotational movement





### **Polymers: Structure**

Polymerization results in varying chain lengths and the **average molecular weight (M<sub>n</sub>)** is used to describe this distribution

 $M_n$  is calculated experimentally from the mole fraction distribution of different sized molecules in a sample, and the **weight average molecular weight (M<sub>w</sub>)** from the weight fraction distribution of different sized molecules

An alternate way of expressing average chain size of a polymer is as the **degree of polymerization (DP)** which represents the average number of repeat units in a chain

M ~ 100 g/mol → generally exist as liquids M ~ 1.000 g/mol → waxy solids (such as paraffin wax) and soft resins 10.000 g/mol < M < Several Million g/mol → Solid polymers M ≤ 100.000 g/mol → the melting/softening temperature increases with M

> Analysis techniques: Osmometry, Light Scattering Technique, End Group Analysis...



 $M_i$  represents the mean (middle) molecular weight of size range *i*, and  $x_i$  is the fraction of the total number of chains within the corresponding size range,  $w_i$  denotes the weight fraction of molecules within the same size interval [1]





## **Polymers: Structure**

Polymerization results in varying chain lengths and the **average molecular weight**  $(M_n)$  is used to describe this distribution

 $M_n$  is calculated experimentally from the mole fraction distribution of different sized molecules in a sample, and the **weight average molecular weight (M<sub>w</sub>)** from the weight fraction distribution of different sized molecules

An alternate way of expressing average chain size of a polymer is as the **degree of polymerization (DP)** which represents the average number of repeat units in a chain

Four different polymer molecular chain structures:

Linear (long continuous chain without any branches), branched ( main chain with smaller chains as branches), crosslinked (adjacent linear chains are joined one to another at various positions by covalent bonds) and **network polymers** (form three-dimensional networks and polymers that are highly crosslinked may also be classified like this)



Molecular chain structure representation: linear, branched, cross-linked and network [1]



*i. e. the epoxies* 



# Homopolymers

### **Thermoplastic**

Strong link into polymer chains



Weak intermolecular forces between polymer chains No cross-links between chains Softens when heated

#### Amorphous or semicrystalline

- Thermoplastics soften when heated and harden upon cooling
- This reversible process can be repeated multiple times without notable degradation. This feature gives thermoplastics a significant **recyclability advantage**
- Most linear polymers (and some branched) are thermoplastic. The lack of cross-linking allows the polymer chains to slide, making them softer and more flexible





# Homopolymers

### **Thermoplastic**

### Thermosetting

Strong link into polymer chains



Monomer

Weak intermolecular forces between polymer chains No cross-links between chains Softens when heated

Amorphous or semicrystalline

Strong cross-link bond

Monomer

Strong covalent bonds between polymer chains Remains hard when heated

- **Thermoplastics** soften when heated and harden upon cooling
- This reversible process can be repeated multiple times without notable degradation. This feature gives thermoplastics a significant **recyclability advantage**
- **Most linear polymers** (and some branched) are thermoplastic. The lack of cross-linking allows the polymer chains to slide, making them softer and more flexible
- **Thermosetting** polymers become permanently hard during their formation and do not soften upon heating
- They are generally harder and stronger than thermoplastics and have better dimensional stability
- Most of the crosslinked and network polymers are thermosets





# Homopolymers

# **Co-polymers**

### **Thermoplastic**

### Thermosetting

Strong link into polymer chains



Strong cross-link bond Monomer

Weak intermolecular forces between polymer chains No cross-links between chains Softens when heated

Amorphous or semicrystalline

Strong covalent bonds between polymer chains Remains hard when heated

**Amorphous** 

- A co-polymer is a polymer that is made up of two or more monomer species
- **Different sequencing arrangements** along the polymer chains are possible.
- For a copolymer, the degree of polymerization uses the average value "m"

 $DP = \frac{\overline{M_n}}{\overline{m}}$ 

 $\overline{m} = \Sigma f_j m_j$ 

f<sub>j</sub> and m<sub>j</sub> are, respectively, the mole fraction and molecular weight of repeat unit "j" in the polymer chain [1]



2024-06-05



### **Crystallization**

Electron micrograph of a polyethylene single crystal. 20,000x. (From A. Keller, R. H. Doremus, B. W. Roberts, and D. Turnbull 1958)



The **degree of crystallinity** may range from completely amorphous to almost entirely crystalline (up to about 95%) and can be determined from accurate **density measurements** 

% crystallinity = 
$$\frac{\rho_c(\rho_s - \rho_a)}{\rho_s(\rho_c - \rho_a)} \times 100$$

 $\rho_c$  is the density of the perfectly crystalline polymer,  $\rho_s$  is the density of a specimen for which the percent crystallinity is to be determined,  $\rho_a$  is the density of the totally amorphous polymer [1]



2024-06-05



### **Crystallization**

Electron micrograph of a polyethylene single crystal. 20,000x. (From A. Keller, R. H. Doremus, B. W. Roberts, and D. Turnbull 1958)



The **degree of crystallinity** may range from completely amorphous to almost entirely crystalline (up to about 95%) and can be determined from accurate **density measurements** 

Semicrystalline polymers contain crystalline regions (**crystallites**) with linear chains structurally oriented, surrounded by amorphous domains of randomly oriented molecules.



Semicrystalline polymer

Crystalline region





### **Crystallization**

Electron micrograph of a polyethylene single crystal. 20,000x. (From A. Keller, R. H. Doremus, B. W. Roberts, and D. Turnbull 1958)



Different densities of the amorphous and crystalline regions will affect for example optical properties (transmitted light gets diffracted and scattered due to  $\neq$  refractive indices)

Polymer crystallinity was firstly observed experimentally in the SEM by growing single crystals from a dilute solution. X-ray measurements indicated that the chains were perpendicular to the face of the lamellae...But how?







### **Crystallization**

**Chain-folded model:** Crystals are regularly shaped in thin platelets (**lamellae**) about 10-20 nm thick and ~10  $\mu$ m long. The molecular chains within each platelet fold back and forth on themselves, with folds occurring at the faces

In many cases, crystals are arranged in larger aggregates known as **spherulites** that grow radially from nucleation points until they meet other spherulites

The temperature range for crystallization is between the glass transition temperature and the crystalline melting temperature



Schematic representation of the detailed structure of a spherulite [1]

Spherulites grow radially until another spherulite is encountered. Hal F. Brinson [3]





 $y = 1 - \exp(-kt^n)$ 

In many polymers the progress of the crystallization from the melt with time (crystallized fraction "") is described by the Avrami equation where "k" and "n" are time-independent constants dependant on the crystallizing system [1]





### **Melting (crystalline phase)**

The **melting of a polymer** corresponds to the transformation of a solid material into a viscous liquid. This phenomenon occurs on the crystalline regions upon heating at the **melting temperature**  $(T_m)$ 

Melting behaviour depends on many variables like the crystallization temperature or annealing treatment, thickness the lamellae, heating rate or impurities/imperfections in the crystals

### **Glass transition (amorphous phase)**

The **glass transition** occurs in the amorphous regions and is due to a reduction in motion of large segments of molecular chains with decreasing temperature. The temperature at which the polymer experiences the transition is the **glass transition temperature** ( $T_g$ )

T<sub>g</sub> is governed by the competition between **thermal mobility** and the **attraction forces between the chains**. Abrupt changes in other physical properties accompany this glass transition like stiffness, heat capacity, coefficient of thermal expansion (CTE)...





Volume as a function of temperature: Crystalline material (top), Amorphous polymer (bottom). L. E. Govaert [4]







2024-06-05

# **Other important properties**

### Viscosity

Measure of a material's **resistance to flow** by shear forces. The viscosity of liquid polymers is very low and depends strongly on their molecular weight

It is a key property for the **processing technology** as most of the techniques are carried out in the fluid phase (lower viscosity  $\rightarrow$  better processability)

### **Viscoelastic Creep**

Many polymeric materials are susceptible to **time-dependent deformation** when the stress level is maintained constant. Creep results are represented as a time-dependent creep modulus  $E_c(t)$ 

The creep modulus is also temperature sensitive  $\downarrow E_c(t) \leftrightarrow \uparrow T$  and susceptibility to creep decreases  $\uparrow E_c(t) \leftrightarrow \uparrow \%$  of crystallinity

### **Thermal expansion**

Polymers generally have higher thermal expansivities than metals and ceramics and their thermal expansion coefficients are not truly constants (the polymers expand in a nonlinear way with temperature)



Load versus time, where load is applied instantaneously at time t<sub>a</sub> and released at t<sub>r</sub>. For the load–time cycle in (a), the strain-versus-time responses are totally elastic (b), viscoelastic (c), and viscous (d) behaviours [1]





#### **Example at CERN: Adhesive in sensors**

What? Adhesive SE4445 in ATLAS Inner Tracker (ITk) Where? Silicon modules are glued on "stave cores" support structures

Why? The adhesive material is applied as a low-viscosity liquid, to cover the adherend surfaces evenly and completely and allow for maximum bonding interactions to position the modules and provides thermal conductivity (titanium cooling pipes into the support structures carry  $CO_2$  at -40C).

**Case of Study:** Failure analysis on cracked modules.

Basic building block of the ITk detector. Courtesy of G. Sciolla [5]









#### **Example at CERN: Adhesive in sensors**

What? Adhesive SE4445 in ATLAS Inner Tracker (ITk) Where? Silicon modules are glued on "stave cores" support structures

Why? The adhesive material is applied as a low-viscosity liquid, to cover the adherend surfaces evenly and completely and allow for maximum bonding interactions to position the modules and provides thermal conductivity (titanium cooling pipes into the support structures carry  $CO_2$  at -40C).

**Case of Study:** Failure analysis on cracked modules. The CT pointed out that the pattern, procedure and/or parameters used for the adhesive application, did not resulted in the desired surface coverage. Sensor



Optical microscopy (OM) and Computed Tomography (CT) for glue pattern evaluation [6] Images courtesy of M. Celuch CERN









#### **Example at CERN: Adhesive in sensors**

Sensor

What? Adhesive SE4445 in ATLAS Inner Tracker (ITk) Where? Silicon modules are glued on "stave cores" support structures

Why? The adhesive material is applied as a low-viscosity liquid, to cover the adherend surfaces evenly and completely and allow for maximum bonding interactions to position the modules and provides thermal conductivity (titanium cooling pipes into the support structures carry  $CO_2$  at -40C).

#### **Case of Study:** Failure analysis on cracked modules.

The CT pointed out that the pattern, procedure and/or parameters used for the adhesive application, did not resulted in the desired surface coverage.

The elevated viscosity might have limited the adhesive displacement leading to different local behaviour when subjected to thermomechanical solicitations and resulting in some modules cracked.

Additional analyses are on-going to solve the issue



Optical microscopy (OM) and Computed Tomography (CT) for glue pattern evaluation [6] Images courtesy of M. Celuch CERN







19

cracked

# Mechanical behaviour of Polymers

#### **Stress-Strain Behaviour**

Polymeric vs metallic materials: Modulus: Polymers < 4 GPa; Metals 48 - 410 GPa TS: Polymers 100 MPa; Metals 4100 MPa Elongation: Elastomers > 1000%; Metals < 100%

Three typically different types of stress-strain behaviour are found for polymeric materials: brittle (curve A), plastic (curve B), and highly elastic polymers – elastomeric - (curve C)

Elastic response: Stiffness of polymers is lower than in other materials  $\rightarrow$  Relevant interactions are the **intermolecular interaction between chains** and the **entanglement (crosslink) network.** A further increase in modulus can be achieved by using particulate fillers (e. g. glass fibers), increasing cross-links (vulcanization) and by molecular orientation (e. g. HPPE commercially available in drawn fibers and tapes with moduli up to 200 GPa).





# **Mechanical behaviour of Polymers**

#### **Stress-Strain Behaviour**

Three typically different types of stress–strain behaviour are found for polymeric materials: brittle (curve A), plastic (curve B), and highly elastic polymers – elastomeric - (curve C)

Beyond the elastic regime: the yield point is taken as a maximum on the curve, which occurs just after the linear-elastic region. The stress at this maximum is the yield strength ( $\sigma_y$ ) and tensile strength (TS) corresponds to the stress at which fracture occurs. The mechanical characteristics of polymers are much more sensitive to temperature changes near room temperature



#### Example at CERN: Epoxy resin

What? Epoxy resin system CTD101K (Composite Technology Development Inc.)

Where? Specifically developed for the impregnation of high-field magnet coils

Why? Epoxy resin is used for electrical insulation [7] and to provide mechanical stability, stiffness and protection to the coil during handling and operation. Important property of the resin system is the ability to resist crack formation and crack propagation.

- Low viscosity → For void free impregnation (long coil length + geometry)
- Improved adhesion property  $\rightarrow$  To reduce bond failure with other components
- *Moderate "pot life"*  $\rightarrow$  *time that passes from initial mixing to reaching a target viscosity*
- Low temperature cure  $\rightarrow$  to reduce internal thermal stresses
- **Toughness and resistance to cracking at RT and 1.9K** → FT, shock test...
- Acceptable strength and flexibility after irradiation → The epoxy is the first magnet material to incur radiation damage

#### We need well-balanced overall combination of properties! -> New formulations

Thermal shock samples with brass bolts after the 1st thermal cycle and the 20th thermal cycle. Y. Shijian et al. [9]









Side view of coil section cut from a 11 T dipole magnet GE13. The flat Rutherford cables can be seen encased in cured CTD101K epoxy with some defects highlighted: a) Shrinkage cavities and metal-tometal crack across the coil interlayer b) Metal-to-metal crack and a large decohesion c) Conductor-to-pole crack d) Large decohesion between resin and pole piece I. Avilés et al. [8]





## **Polymer additives**

Foreign substances intentionally introduced to enhance or modify the polymer properties and render a polymer more usable

### **Fillers**

- Added to improve tensile and compressive strengths, abrasion resistance, toughness, dimensional and thermal stability...but also to reduce the cost
- Particle sizes range from 10 nm to macroscopic, and fillers include wood flour, silica, sand, glass, clay, talc, limestone, and synthetic polymers

### **Plasticiser**

- Added to improve the flexibility, ductility, and toughness of polymers reducing hardness and stiffness.
- Plasticizers are usually low vapor pressure, low molecular weight liquids that fit between large polymer chains, increasing interchain distance and reducing secondary intermolecular bonding

### Colorants

 Added to provide a specific colour to a polymer in the form of dyes or pigments

### **Flame retardants**

 Interfering with the combustion process either through the gas phase or by initiating a different → slowing or stopping the burning

### **Stabilizers**

- Added to counteract deterioration processes due to environmental conditions like exposure to UV radiation
- Also, antioxidants that prevent the chemical interaction between the oxygen and the polymer molecules





## **Radiation on Polymers**

**Radiation modifies the structure** of polymeric materials making them either softer or harder and causing gas production, acid formation, colour change, oxidation, and separation of multi-phase materials like greases.

A deep knowledge of the **radiation tolerance** of lubricants, glues, elastomeric sealants, resins, insulators for magnets, cables, vacuum components, etc. minimizes the risk of radiation-induced failures and reduces the design safety margins.

Total absorbed dose of polymers during operation: 0.1 MGy threshold for many polymeric materials > 1 MGy → experimentally tested radiation tolerance > 10 MGy → avoid, or regular monitoring/ maintenance planned

Important research activity at CERN in this field includes experimental testing of commercial and custom-made nonmetallic materials (yellow reports [11][12][13], R2E Project [14], CARE Project,...)

External or in-house facilities Total doses 0.1 MGy to 20 MGy

> *Characterization techniques:* Characterization: Visual testing, mechanical testing, swelling test, Differential Scanning Calorimetry (DSC), outgassing......



Representative examples of installed cables subjected to different total absorbed dose. CARE Project. Courtesy of J. Gascón [15]



A. Pérez | Plastics and Composite Materials | CAS-2024



## **Radiation on Polymers**

**Radiation modifies the structure** of polymeric materials making them either softer or harder and causing gas production, acid formation, colour change, oxidation, and separation of multi-phase materials like greases.

A deep knowledge of the **radiation tolerance** of lubricants, glues, elastomeric sealants, resins, insulators for magnets, cables, vacuum components, etc. minimizes the risk of radiation-induced failures and reduces the design safety margins.

- Formation of free radicals: depending on chemical structure, crystallinity, solvent content, additives, plasticizer, stabilizers, temperature, oxygen, humidity...
- Formation of hydrogen & light HC
- Formation of C-C bonds between molecules (crosslinking)
- Rupture of C-C bond (chain scission)
- Increase in unsaturation
- Breakdown of crystalline structure
- Discoloration
- Oxidation



(a) Draka NE48 jacket.

(b) Draka NE48 insulation.

Elongation at break (EaB %) vs absorbed dose on cable jacket (EVA) and insulation (XLPE). Courtesy of J. Gascón

Radiation induced effects are usually described as a function of the **total absorbed dose**, but depend also on the irradiation conditions (radiation type, dose rate, temperature, humidity, oxygen, electrical or mechanical stress) and their **synergetic effect** (simultaneous stressors damage higher than sequential equivalent stressor damage separately)





# **Radiation on Polymers**

Understanding the effect of radiation on the properties of **epoxy resins** used in the vacuum impregnation of **magnet coils under relevant irradiation conditions** is crucial for the future accelerator facilities

In this case the irradiation source, temperatures (ambient and 4.2 K), atmosphere (absence of oxygen) or the specific additives can strongly influence the aging

 $T_g$  vs dose evolutions allow to compare aging rates



Evolution of the Tg (G" max) of different epoxy resins as a function of the absorbed dose. D. M. Parragh et al. [16]

Images from [7]



Materials candidate for the protective covers of magnets after gamma irradiation at 10 MGy. Courtesy of C. L. Marraco Borderas [10]



Knowledge of the behaviour of cable insulation and the thermomechanical properties of the **composite** is essential for the mechanical design of the magnet





### **Composite Materials**



This relatively new family of materials marked a significant advancement in civil construction, energy or aeronautics...meeting the growing need for **stronger** yet lighter materials

#### Why are they so interesting?

- Possible to achieve combinations of properties not attainable with metals, ceramics or polymers alone
- Design of materials with optimized properties
- Developed in parallel with high stiffness and strength fibers
- Can be produced by various processing techniques
- Large part size possible



2024-06-05



### What are composites?

Composed of at least two phases: the **matrix**, which is continuous and surrounds the **dispersed phase** 

The **properties of composites** are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase (shape and size of the particles, distribution, and orientation)

**Classification according to the matrix** (polymeric, metallic or ceramic, including the carbon-carbon composites) or to the **reinforcement geometry** (particle-reinforced, fiber- reinforced, structural and nanocomposites)



Comparison between conventional materials and composite materials. K. Chawka [17]



Comparison of the mechanical properties of composites with other materials. (a) Specific stiffness versus specific strength and (b) strength versus fracture toughness. CFRP, carbon-fiber reinforced polymers; GFRP, glass—fiber reinforced polymer [18]





## **Polymer-Matrix Composites**

Polymeric Matrix Composites (**PMCs**) consist in a plastic matrix with fibers as reinforcement and are the most widely used to manufacture composites.

Two types of matrix:

 Thermoplastics → low flow (difficult to infiltrate), expensive but good mechanical properties at high temperature (i. e. Polyetheretherketone-PEEK, Polyphenylene Sulphide-PPS and polyetherimide-PEI)

> Film stacking, thermoforming, injection moulding...

- Thermosets (resins) → low viscosity before curing, cheap, resistant to chemical attack but brittle, very low fracture toughness and limited properties at high temperature (i. e. unsaturated polyester resins, epoxy resins and polyimides)
  - Produced by hand layup and spray techniques, filament winding, pultrusion, autoclave-based methods ("prepreg")...

**Most typical fibers** are Glass Fiber–Reinforced Polymer (**GFRP**), Carbon Fiber– Reinforced Polymer (**CFRP**) and Aramid Fiber–Reinforced Polymer Composites









**Continuous fibers**  $\rightarrow$  Maximum fiber volume fraction in real composites is in the range 60-70% (matrix infiltration becomes very difficult above these values)

- Highly anisotropic and the maximum strength and reinforcement are achieved along the alignment (longitudinal) direction
- Properties perpendicular to the fibers are controlled by the matrix

**Short fibers**  $\rightarrow$  Length from 100 µm to few mm. The fiber volume fraction, average length and orientation are relevant parameters

Fiber orientation (2D or 3D)



Schematic representations of the various geometrical and spatial characteristics of particles of the dispersed phase that may influence the properties of composites [1]





**Continuous fibers**  $\rightarrow$  Maximum fiber volume fraction in real composites is in the range 60-70% (matrix infiltration becomes very difficult above these values)

- Highly anisotropic and the maximum strength and reinforcement are achieved along the alignment (longitudinal) direction
- Properties perpendicular to the fibers are controlled by the matrix

**Short fibers**  $\rightarrow$  Length from 100  $\mu$ m to few mm. The fiber volume fraction, average length and orientation are relevant parameters

Longitudinal

Tensile

Strength (MPa)

700

1000-1900

1200

Comparison of longitudinal and transverse tensile strength in various FRC [1]

Fiber orientation (2D or 3D)



Schematic representations of (a) continuous and aligned, (b) discontinuous and aligned, and (c) discontinuous and randomly oriented fiber—reinforced composites [1]

Property	Unreinforced	Value for Given Amount of Reinforcement (vol%)		
		20	30	40
Specific gravity	1.19–1.22	1.35	1.43	1.52
Tensile strength [MPa (ksi)]	59–62 (8.5–9.0)	110 (16)	131 (19)	159 (23)
Modulus of elasticity [GPa (10 <sup>6</sup> psi)]	2.24–2.345 (0.325–0.340)	5.93 (0.86)	8.62 (1.25)	11.6 (1.68)
Elongation (%)	90–115	4–6	3–5	3–5
Impact strength, notched Izod (lb <sub>f</sub> /in.)	12–16	2.0	2.0	2.5

Properties of unreinforced and reinforced polycarbonates with randomly oriented glass fibers [1]



Material

Glass-polyester

Kevlar-epoxy

Carbon (high modulus)-epoxy

Transverse Tensile

Strength (MPa)

47-57

40-55

20



31

**Continuous fibers**  $\rightarrow$  Maximum fiber volume fraction in real composites is in the range 60-70% (matrix infiltration becomes very difficult above these values)

- Highly anisotropic and the maximum strength and reinforcement are achieved along the alignment (longitudinal) direction
- Properties perpendicular to the fibers are controlled by the matrix

**Short fibers**  $\rightarrow$  Length from 100  $\mu$ m to few mm. The fiber volume fraction, average length and orientation are relevant parameters

- Fiber orientation (2D or 3D)
- $\succ$  Critical length "l<sub>c</sub>" of the fiber



Variation of some mechanical properties of a composite as a function of fiber length [17]

The deformation pattern in the matrix surrounding a fiber that is subjected to an applied tensile load [1]



Where "d" is the fiber  $\emptyset$  and " $\sigma_f^*$ " the ultimate (or tensile) strength and  $\tau_c$  the fiber–matrix bond strength [1]



A. Pérez | Plastics and Composite Materials | CAS-2024



**1D:** Is the easiest fiber arrangement in a **unidirectional lamina** also called ply or layer

**2D fabrics:** Multidirectional laminates (built by stacking unidirectional lamina with different orientations and can provide quasi-isotropic properties), woven fabrics, knit, braided, nonwovens (from a set of disordered fibers)

3D fabrics (woven fabrics, non-crimp fabrics, stitching)



Effect of fiber orientation on the tensile strength of E-glass fiberreinforced epoxy composites. D. R. Askeland [19]

**Post-lay-up processing techniques** include autoclave moulding, pressure-bag moulding, and vacuum-bag moulding to reduce the porosity





Continuous fibers







The laminate structures can be manufactured from continuous fibers plies. S. Kalpakjian [20]

#### **Example at CERN: PMCs**

#### What? CFRP Composite

#### Where? ATLAS Inner Tracker (ITk) structural components

Why? The tracker needs to record particle paths with very high precision, yet be lightweight, to disturb the particles as little as possible  $\rightarrow$  Very thin, robust and accurate carbon fiber profiles are needed

For small series production an important invest on the development phase the mechanical simulation is key to validate the configuration of the laminates arranged so that the maximum service stress lies in the <u>direction that has</u> the highest strength

#### Design and simulation

- Definition of the material properties
- Definition of the stacking sequence
- Verify the compatibility with the detector envelope

#### Tooling and mould

- *Conception of the different parts*
- CNC machining

#### Prototype production

- Cutting of the carbon plies
- Lamination of the carbon plies
- Polymerization in autoclave
- Demoulding of the part

Main design and manufacturing steps of structural ring from CFRP composite "pre-preg" raw material. Courtesy of F. Boyer <u>Composite Laboratory</u> at CERN









"Prepreg" twill weave





A. Pérez | Plastics and Composite Materials | CAS-2024



The **mechanical characteristics** of a fiber-reinforced composite depend not only on the properties of the fiber, but also on the degree to which an applied load is transmitted to the fibers by the matrix phase  $\rightarrow$  Matrix-fiber interface

The **interface area is very large** in composites (100000  $m^2/m^3$ ) and is more important if the fiber radius decreases (i. e. nanotubes reinforcement). We need an **optimized bonding that improves the toughness** of the component

- Wettability → Ability of the liquid to spread on a solid surface (infiltration!)
  - Important effect of surface roughness (intimate contact), and coupling agents are frequently used to improve the wettability between the components
- Bonding → Mechanical bonding (interlocking or mechanical gripping when the matrix contracts), physical bonding (van der Waals forces...), chemical bonding (dissolution bonding, and reaction bonding). Interlock effect between rough surfaces

Interface mechanical behaviour is characterized by shear stress (// to the interface), normal strength and interface fracture energy: Flexural test, bending test, interlaminar shear strength (ILSS), fibers push-in/push out test, pull-out test, instrumented indentation test...



a) Good mechanical bond and b) lack of wettability can make a liquid polymer or metal unable to penetrate the asperities on the fiber surface, leading to interfacial voids [17]







35

(1)

50

40

Load (mN) 8







#### **Example at CERN: MMCs**

What? Cooling adaptors machined from MMC Aluminium-CF (AI-CF) with chopped fibers randomly oriented in XY plane (50 vol%)

Where? Outer tracker of the CMS detector with silicon sensors at -30C during operation

Why? Thermal expansion coefficient should match that of silicon to avoid deformations during cooling cycles, dimensional stability over time (few μm) and non-magnetic **Case of study:** Material from two different providers A (Casted Al-CF) vs. B (Sintered Al-CF). Format from "B" (rectangular blocks) is more suitable for the application but important dimensional variations were observed with time.







Images from [22]



#### **Example at CERN: MMCs**

What? Cooling adaptors machined from MMC Aluminium-CF (AI-CF) with chopped fibers randomly oriented in XY plane (50 vol%)

Where? Outer tracker of the CMS detector with silicon sensors at -30C during operation

Why? Thermal expansion coefficient should match that of silicon to avoid deformations during cooling cycles, dimensional stability over time (few μm) and non-magnetic Case of study: Material from two different providers A (Casted Al-CF) vs. B (Sintered Al-CF). Format from "B" (rectangular blocks) is more suitable for the application but important dimensional variations were observed with time.









Images from [22]

SEM images after wet testing confirmed galvanic corrosion signs at the matrix-CF interface on sintered samples. K. Buchanan et al. [23]





A. Pérez | Plastics and Composite Materials | CAS-2024



37

### **Particle-Reinforced Composites**

#### Large-particle composites

Polymers with fillers like synthetic rubber with carbon black particles in a tire, concrete used as structural building material...

Particle-matrix interactions are analysed using macroscopic mechanics

### **Dispersion-strengthened (DS) composites**

Diameters between 10 and 100 nm

Particle–matrix interactions occur on the atomic or molecular level

The matrix carries most of the applied load, while the small dispersed particles hinder dislocation movement



## **Particle-Reinforced Composites**

### Large-particle composites

### Example at CERN

What? CuCD with diamond particles dispersed in a Cu matrix Where? Absorber material in beam intercepting devices (BIDs) Why? Combination of properties provided by the two main material constituents—copper contributes to the thermal and electrical conductivity of the material, while the diamond particles further improve thermal conductivity and aid in reducing the density and the coefficient of thermal expansion



Microscopy image of CuCD, with diamond particles dispersed in a copper matrix. A. Bertarelli et al. [24]

### **Dispersion-strengthened (DS) composites**

### **Example at CERN**

**What?** GLIDCOP<sup>\*</sup> is the trademark for DS copper (DSC) containing  $Al_2O_3$  particles. Fore CERN BID the grade Al-15 is of interest

#### Where? Collimators jaws

Why? The alumina particles are stable at high temperatures preventing the softening and of the copper when subjected to high temperatures and maintaining the requested jaw flatness to maintain their beam cleaning efficiency



New secondary collimator, 3D view of the jaw. F. Carra et al. [25]



Comparison of hardness vs. T for different copper alloys and DSC. Total Materia [26]



A. Pérez | Plastics and Composite Materials | CAS-2024

### **Structural Composites:**

- Multilayered and normally low-density composite used in applications requiring structural integrity, ordinarily high tensile, compressive, torsional strengths and stiffnesses
- The properties depend not only on the constituent materials, but also on the geometrical design of the structural elements (i. e. the core thickness)

**Sandwich panel** consists of two outer sheets (faces or skins) that are adhesively bonded to a thicker core (typically rigid polymeric foams, wood, and honeycombs) **Laminar composite** produced by lay-up (i. e. laminate on fiber-reinforced composites)



Schematic diagram showing the construction of a honeycomb core sandwich panel [1]





### **Structural Composites:**

- **Multilayered** and normally low-density composite used in applications requiring structural integrity, ordinarily high tensile, compressive, torsional strengths and stiffnesses
- The properties depend not only on the constituent materials, but also on the geometrical design of the structural elements (i. e. the core thickness)

**Sandwich panel** consists of two outer sheets (faces or skins) that are adhesively bonded to a thicker core (typically rigid polymeric foams, wood, and honeycombs) **Laminar composite** produced by lay-up (i. e. laminate on fiber-reinforced composites)



Schematic diagram showing the construction of a honeycomb core sandwich panel [1]

#### **Example at CERN: Laminar**

**What?** Gas Electrons Multiplier (GEM) thin composite sheet with a thin polymer foil (insulator) metal-coated on both sides and pierced with a high density of holes (50  $\mu$ m Kapton with 5  $\mu$ m Cu)

Where? The multiplier can be used as detector on its own, or as a preamplifier in a multiple structure

Why? The component can reach large overall gains in harsh radiation environment. The sensors are durable and easy to assemble using prefabricated components. They offer various configurable options, enabling customization of sensor parameters to meet specific needs. Flexible detector shape, robust and low cost



Electric field in the region of the holes of a GEM electrode. F. Sauli [27]





41



A. Pérez | Plastics and Composite Materials | CAS-2024

### **Nanocomposites:**

#### Diameters < 100 nm

on both matrix **Properties** depend and nanoparticle, nanoparticles properties + shape + content + matrix/nanoparticle interface

Most of today's **commercial nanocomposites** use nanocarbons, nanoclays, and particulate nanocrystals (typically inorganic oxides such as silica, alumina, zirconia, halfnia, and titania)

For most applications, the nanosized particles must be dispersed uniformly and homogeneously within the matrix  $\rightarrow$  **Big challenge!** 

Novel fabrication techniques are constantly being developed

#### **Example at CERN: SC wires**

What? Artificial Pinning Center (APC) Nb<sub>3</sub>Sn wires

Where? Development studies for future 100 TeV hadron collider

Why? The presence of a uniformly distributed oxygen source (OS) during the thermal treatment reduces the average Nb<sub>3</sub>Sn grain size, leading to an enhancement of the layer critical current density (Jc) at 4.2 K [28][29][30]



SEM image of a heat treated Zr-core OS wire (700  $^{\circ}C \times 50$  h) and detail of filament with the central unreacted Nb-alloy surrounded by a region of Nb<sub>3</sub>Sn fine grains and then the large grains of Nb3<sub>s</sub>n and other phases. F. Lonardo et al. [30]

> STEM image (Collaboration CERN -UNIGE on dimensional assessment of nano particles in APC Nb<sub>3</sub>Sn wires). Courtesy of S. Pfeiffer and A. Moros









EHT = 30.00 kV Detector = aSTEM4 A 19 Jul 2023

### Conclusions

Understanding polymer and composite structures helps us to understand our component properties and performance An overview of their typical classification, strengths and limitations of each type was presented

Face sheet

Face sheet

Specific examples of those materials use in accelerator complex has been provided















ENGINEERING DEPARTMENT MECHANICAL & MATERIALS ENGINEERING FOR PARTICLE ACCELERATORS AND DETECTORS

### **References "Polymers"**

[1] Materials Science and Engineering An Introduction by William D. Callister, Jr., David G. Rethwish (11th edition)

[2] Introduction to Polymer Physics by M. Doi

[3] Polymer Engineering Science and Viscoelasticity by Hal F. Brinson & L. Catherine Brinson (2nd edition)

[4] Polymers: From Structure to Properties by L. E. Govaert, A. K, van der Vegt & M. van Drongelen, (2019)

[5] The ATLAS ITk Strip Detector for the Phase-II LHC Upgrade. Gabriella Sciolla, Brandeis University. Presented at DIS2023

[6] Failure Analysis: Early High Voltage Breakdown of ATLAS ITK Stave's Modules. EDMS Report 3031317 by A. T. Pérez Fontenla, CERN (2024)

[7] Electrical Insulation for Magnets: dielectrics, design and construction. Lecture by R. Piccin CAS Magnet (2023)

[8] Advanced Examination of Nb3Sn Coils and Conductors for the LHC Luminosity Upgrade: A Methodology Based on Computed Tomography and Materialographic Analyses I. Avilés et al. (Manuscript submitted 2024)

[9] Design of a High Toughness Epoxy for Superconducting Magnets and Its Key Properties. Y. Shijian et al. (2019)

[10] Elasticity, plasticity and fracture toughness at ambient and cryogenic temperatures of epoxy systems used for the impregnation of high-field superconducting magnets. A. Brem et al. (2021)

[11] Compilation of radiation damage test data. Part I by M. Tavlet and H. Schonbacher (1989)

[12] Compilation of radiation damage test data. Part II by M. Tavlet, A. Fontaine and H. Schonbacher (1998)

- [13] Radiation tests on selected insulated materials for high power and high voltage application. G. Liptak and R. Schuler (1985)
- [14] "Radiation to Materials" at CERN. M. Ferrari et al. (2023)
- [15] Cable Irradiation Activity. Talk by J. Gascón at 12<sup>th</sup> HL-LHC Collaboration meeting, Uppsala (2022)

[16] Irradiation Induced Aging of Epoxy Resins for Impregnation of Superconducting Magnet Coils. D. M. Parragh et al. (2024)





### **References "Composites"**

[17] Composite materials Science and Engineering by K. Chawla (4th edition)

- [18] Multiscale Modelling of Composites: Toward Virtual Testing ... and Beyond. J. Llorca et al. (2012)
- [19] The Science and Engineering of Materials. D.R. Askeland, P. P. Phulé (5th Edition)
- [20] Manufacturing Processes for Engineering Materials. S. Kalpakjian and S. Schmid
- [21] A novel trench fiber push-out method to evaluate interfacial failure in long fiber composites, R. M. G. De Meyere et al. (2021)
- [22] Tilted TBPS Ring Mechanics for CMS Tracker Upgrade Phase II. Talk by P. Rose at Forum on Tracking Detector Mechanics (2022)
- [23] Assessment of Two Advanced Aluminium-Based Metal Matrix Composites for Application to High Energy Physics Detectors, K. E. Buchanan et al. (2023)
- [24] Results on simulations of new materials and composites, EuCARD2-Del-D11-1-Final, A. Bertarelli et al. (2016)
- [25] Mechanical Engineering and Design of Novel Collimators for HL-LHC. F. Carra et al. (2014)
- [26] Dispersion Strengthened Copper (DSC) Alloys. Part 2, Total Materia
- [27] The gas electron multiplier (GEM): Operating principles and applications, F. Sauli (2016)
- [28] Internally Oxidized Nb3Sn Strands with Fine Grain Size and High Critical Current Density, X. Xu et al. (2015)
- [29] Effects of the oxygen source configuration on the superconducting properties of internally-oxidized internal-Sn Nb<sub>3</sub>Sn wires, G. Bovone et. Al (2023)
- [30] Influence of the Heat Treatment on the Layer J<sub>C</sub> of Internal-Sn Nb<sub>3</sub>Sn Wires With Internally Oxidized Nanoparticles, F. Lonardo et al. (2024)





**1D:** Is the easiest fiber arrangement in a **unidirectional lamina** also called ply or layer  $\rightarrow$  Maximum fiber volume fraction 60-70%

**2D fabrics:** Multidirectional laminates (built by stacking unidirectional lamina with different orientations and can provide quasi-isotropic properties), woven fabrics, knit, braided, nonwovens (from a set of disordered fibers)

**3D fabrics** (woven fabrics, non-crimp fabrics, stitching)





twill









2024-06-05

A. Pérez | Plastics and Composite Materials | CAS-2024



### **Polymer-Matrix Composites**



a) In hand layup, fibers are laid onto a mould by hand, and the resin is sprayed or brushed on. b) In spray-up, resin and fibers (chopped) are sprayed together onto the mould surface [17]





Schematic of filament winding process (up) and schematic of the pultrusion process 9down) [17]

Autoclave or bag moulding processes → curing under (high) pressure and temperature → low content of air inclusions



"prepreg" is thin sheet or lamina of unidirectional (or occasionally woven) fiber/polymer composite protected on both sides with easily removable separators



A large autoclave that is used to make the wings of Boeing 787



A. Pérez | Plastics and Composite Materials | CAS-2024

