

KOMPO teemapäivä 16.5.2023 - Mittaaminen ja analysointi

Mechanical testing of composite laminates

**Research unit Mikpolis – Aapo Nylén
South-Eastern Finland University of Applied Sciences – XAMK**

RDI focus areas of South-Eastern Finland University of Applied Sciences (XAMK)



FOREST, THE ENVIRONMENT AND ENERGY

- New fibre products and processes
- Forestry and wood construction
- Electronics and materials
- Environmental safety and circular economy
- Renewable and efficient energy systems



SUSTAINABLE WELLBEING

- Effective wellbeing services
- Equality and empowerment of youth
- Smart, user-center food services
- Responsible tourism



DIGITAL ECONOMY

- Digital information management and archiving
- User-oriented services and design
- Entrepreneurship and business development
- Game technology and cybersecurity



LOGISTICS AND SEAFARING

- Maritime safety and emergency management
- Oil spill prevention and response
- Sustainable port logistics
- Railway logistics
- Future intelligent transport systems

External funding in terms of focus areas 2022



-  **Forest, the Environment and Energy:**
20.4 Million Euros
-  **Digital Economy:**
19.9 Million Euros
-  **Sustainable Wellbeing:**
16.2 Million Euros
-  **Logistics and Seafaring:**
6.7 Million Euros

EUR30.7million
RESEARCH AND DEVELOPMENT
EXPENDITURE

EUR18.8million
EXTERNAL RDI FUNDING




246 projects, including
43 international projects



1332 partners, including
755 corporate partners

Over
890 Xamk employees



-  **TEACHERS**
338
-  **RDI PERSONNEL**
299
-  **OTHER PERSONNEL GROUPS**
260

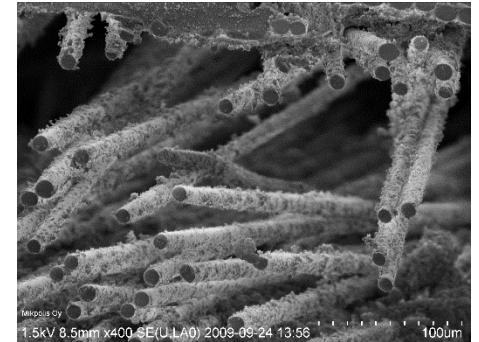
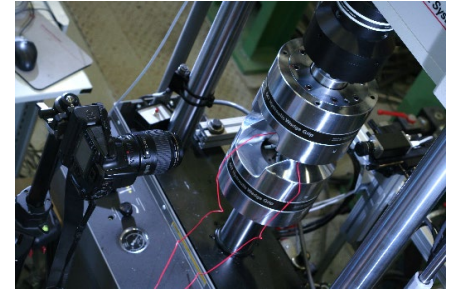
Research unit Mikpolis

- Mikpolis is a research, development and innovation unit and is part of forest, environment and energy focus area. Located in Mikkeli, it supports clients' RDI activities and knowhow in materials and manufacturing engineering industries.
- Materials technology laboratory offers a wide range of testing and analysis services for the characterization of materials, structures and products properties. Testing is performed in laboratory facilities or as on-site measurements.
- The laboratory of wood technology performs experimental impregnation of wood, thermal modification and drying of wood, designs and manufactures mold solutions for wood, furniture and reinforced plastics industry and offers a wide range of testing and analysis services.

- Annual number of customers >50 per year
- >500 customers in 2008...2022
- Annual turnover of service business ~500 k€
- A few research projects ongoing each year
- Staff is involved in customer service business and in publicly funded research projects

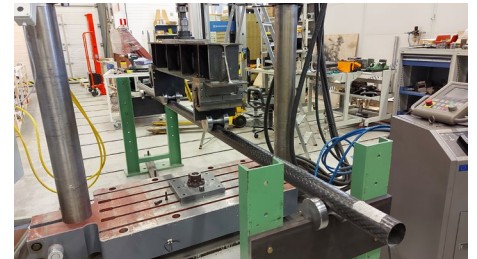
Services of materials technology laboratory

- Materials (solids) characterization
 - Mechanical properties: static and dynamic testing, hardness tests
 - Test sets and jigs planning and machining in-situ according to the needs of customer
 - On site measurements of strain and force
 - Structural analyses
 - Surface analyses
 - Chemical composition
 - Thermal analyses
- Environmental testing
 - Heat / humidity
 - Corrosive conditons
 - UV-A/B / Sun light
 - Outdoor test field with UV / temperature / humidity data acquisition
- Surface analyses
 - Microscopy, topography / metrology
 - Color measurements
 - Wear resistance
 - Contact angle and surface energy



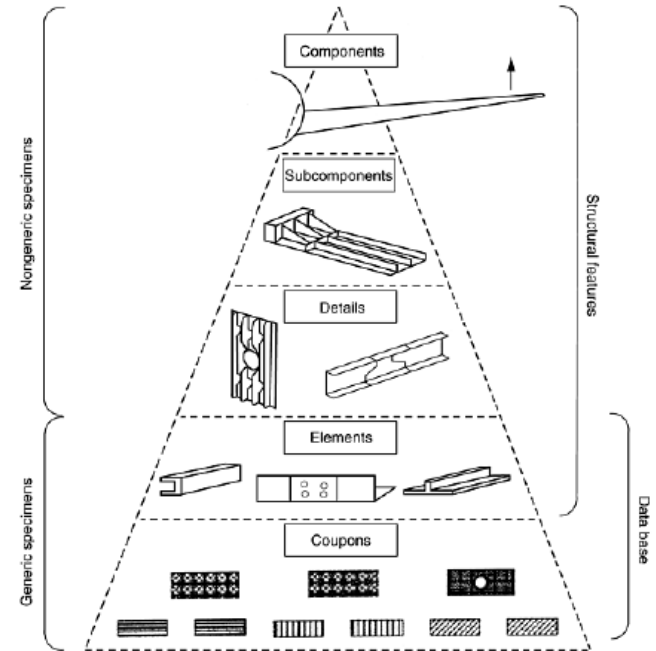
Services of materials technology laboratory

- Laboratory equipment
 - Loading frames 5 pcs (0,5-300 kN with several elongation measurement systems, modifiable according to the needs of customer)
 - Hardness testers: Brinell, Rockwell, Vickers (micro)
 - Precision data acquisition for strain gage measurements or other sensors
 - Wear testing: Taber, abrasion wear tester
 - Thermal analyzers: DSC, DMTA, TGA, cone calorimeter
 - Surface analyzers: SEM, optical 3d-profilometer, stereomicroscope, Stylus-profilometer, contact angle analyzer
 - Structural analyses: Micro-CT
 - Chemical analyses: SEM/EDS, XRF, FT-IR
 - Environmental testing: Xenon, UV, salt spray, weather cabinet
 - Other sample preparation and research equipment



Breakdown of test items (MIL-HDBK-17 “building-block” approach)

- Constituent materials testing (Coupon tests)
 - Standard test methods and testing for common properties of composite prepreg, fibers and matrices
 - Key properties of both matrix and reinforcement include density, tensile strength, and tensile modulus
- Lamina and laminate nonmechanical testing
 - Standard test methods and testing for physical, thermal, and moisture properties of advanced composites
 - Key properties include fiber areal weight, matrix content, void content, cured ply thickness, lamina tensile strengths and moduli, lamina compression strengths and moduli, and lamina shear strengths and moduli
- Lamina and laminate mechanical testing (Coupon tests)
 - Standard test methods and testing for strength and modulus testing of lamina/laminate forms
 - Key properties include laminate tensile strengths and moduli, laminate compression strengths and moduli, laminate shear strengths and moduli, interlaminar fracture toughness, and fatigue resistance
 - Focus on confirming the ability of analysis methods to predict response from the lamina properties, or it may be the primary source of the design data
- Element and subcomponent testing / Full-scale structural testing
 - Key properties include open- and filled-hole tensile strength, open- and filled-hole compression strength, compression-after-impact strength, and joint-bearing and bearing-bypass strengths
 - Some of these properties may have full statistical test samples at all major environments
 - Focus of the behavior and failure mode of increasingly more-complex structural assemblies. Data used from the lower levels of the building-block pyramid to simplify testing loads and/or environments



Purpose of testing from RDI project to production (MIL-HDBK-17)

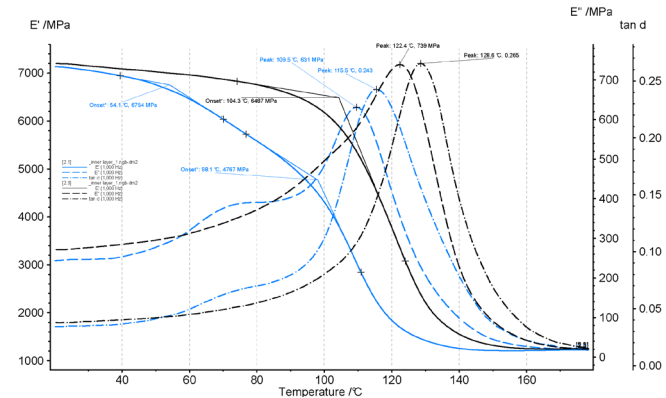
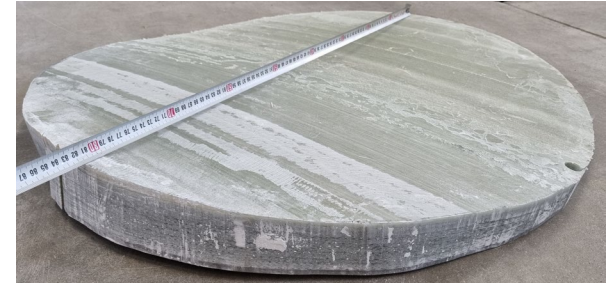
- Screening Data
 - Screening testing evaluates new material systems under worst-case conditions, identifying inadequate materials and promising candidates
 - Screening test matrix provides average values for strengths, moduli, and physical properties of small samples at the lamina and laminate levels
- Material Qualification Data
 - Material qualification testing aligns with design allowables (the maximum permissible values or limits of certain material properties that are used in the design and analysis of structures or components) to establish essential material properties for acceptance, equivalence, and quality control
- Acceptance Data
 - Acceptance tests verify material consistency, comparing small sample results with control values of key material properties
 - Aim is to ensure reliability and quality in the manufacturing process
- Equivalence Data
 - Equivalence tests assess alternate materials' similarity to known materials, aiming for cost savings
 - The alternate material is substantially the same as the known material, but may have minor process changes to the raw material or minor changes in the final end-object process
- Structural Substantiation Data
 - Structural substantiation data assess a structure's ability to meet application requirements, with design allowables derived from material qualification statistics
 - The development of design allowables, ideally derived or related to statistical data obtained during a material qualification task, continues

Differences between testing of composites and testing of isotropic materials

- Material anisotropy
 - Tested properties of composites are often significantly different depending on test direction / orientation
 - For example, uniaxial mechanical tests never produce a pure uniaxial stress state throughout an entire test specimen; three-dimensional stresses always exist at discontinuities and loading points
 - Combination of multiaxial stresses and low strengths in directions other than the test direction can result in unexpected failure, which lowers the mean value and increases scatter in the data
- Complicated and irrational failure modes
 - Correctly executed test for a strength- or fracture-based property should produce an expected failure mode, but multiple failure modes are possible
 - Identification of failure modes are often unclear even with correctly performed tests
- Unintentional inhomogeneity
 - Composites are cured into the final form at the same time that the end product is produced
 - Local fiber (and void) volume changes are common
 - Fiber orientation changes
- Environmental sensitivity of material components
 - Composite materials are sensitive to environmental conditions

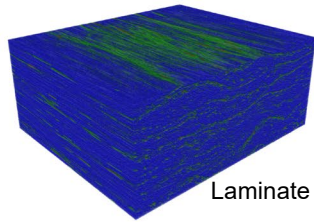
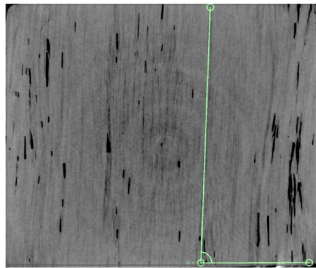
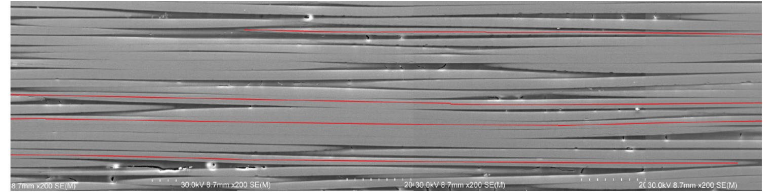
To increase the reliability and validity of the test results (essentials)

- Generally, correct test method, suitable test setup, calibrated equipment and trained personnel
- Test specimen preparation
 - Specimen orientation, depending on the source of the tested laminate; cutout from a product vs. pultruded UD profile
 - Specimen labelling (traceability)
 - Correct manufacturing method with geometry that meets the criteria of applied test standard
 - Conditioning at certain temperature and humidity if needed
 - Post-curing or current state of material?
- Determination resin, fiber and void content
 - Density of composite determined by immersion method (ISO 1183-1)
 - Resin, fiber and void content by calcination method (ISO 1172)
 - Must have precise density data of cured resin and fiber
 - The correct calcination temperature determined by thermogravimetric analysis (TGA), especially with CFRP materials
 - Micro-CT analyses can be utilized
- Transition temperatures and curing properties
 - Glass transition temperature T_g (α transition) and other possible transition temperatures and degree of cure
 - DSC method applied if relatively low fiber content. DSC analysis provides clear indication of post-curing (exothermal reaction)
 - DMTA method is applied for advanced composites. Practice for determining the transition, elastic and loss moduli over a range of temperatures, frequencies or time

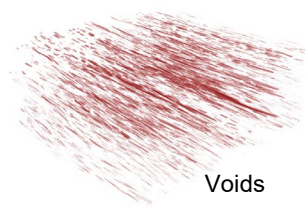


To increase the reliability and validity of the test results (essentials)

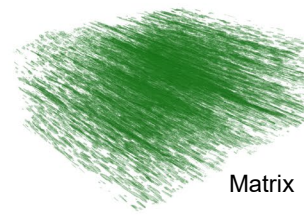
- Fiber orientation / undulation and homogeneity
 - Cross-sectional analysis by SEM or by microscopy
 - Limited analysis area with high accuracy
 - Determination of fiber angles
 - Micro-computed tomography (Micro-CT)
 - Limited analysis volume to achieve high enough resolution
 - Determination of fiber angles (and void, resin fiber contents)



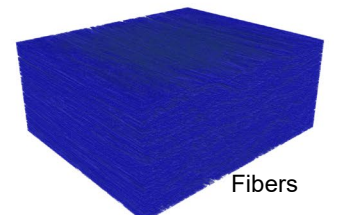
Laminate



Voids



Matrix



Fibers

- Calcination and image analysis
 - Larger sample size, up to 100 mm x 100 mm
 - Results only indicative
 - Measurement of orientation of carbon fiber tows / glass fiber rovings

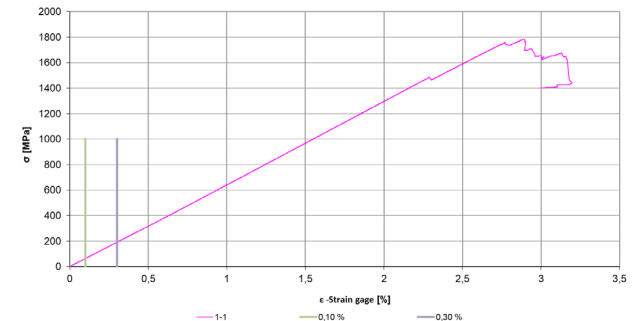
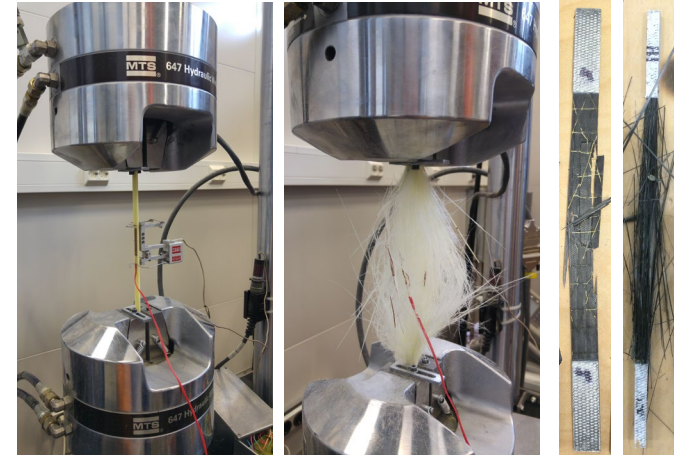


Tensile properties of lamina / laminates (in-plane)

- Typical test standards: ISO 527-1/4/5, ASTM D 3039
- Requires precision fixtures and accurate force application with accurate extension measurement
- Specimen protected by end tabs to avoid premature fiber breakage in the clamped area, especially with unidirectional / high-modulus composites (force applied through a mechanical shear interface)
- Dogbone-shaped specimens used with multi-directional materials
- Data acquisition: force, displacement and strain
 - Strain measurements by extensometer(s) or by strain gage(s)
- Measured properties: tensile strength, tensile strain, Poisson's ratio, tensile modulus, stress-strain response

$$\sigma = \frac{F}{A} \quad \epsilon = \frac{\Delta L_0}{L_0} \quad \mu_n = -\frac{\epsilon_n}{\epsilon} \quad E_t = \frac{\sigma_2 - \sigma_1}{\epsilon_2 - \epsilon_1}$$

- Modulus affected by calculation area (depends on applied standard)
- If 'tensile strain at break' or 'tensile strain at tensile strength' is need, strain gage measurement is mandatory
 - In most cases, ultimate strain values have to be calculated / estimated from stress-strain response, since failure modes differs depending on the test direction and the tested laminate
- Test results affected by:
 - Alignment of test fixtures
 - Forces on specimen during gripping (force-controlled equipment recommended)
 - Specimen orientation: fiber versus load axis misalignment in a UD specimen (due to either specimen preparation or test error or both), can reduce strength as much as 30 % due to an initial 1° misalignment
 - Grip pressure (hydraulic grips recommended) and geometry of end tab areas
 - Asymmetrical laminate structure
 - Specimen geometry: low width-to-thickness ratio or circular specimen geometry requires extended clamping area to ensure shear-induced tensile loading

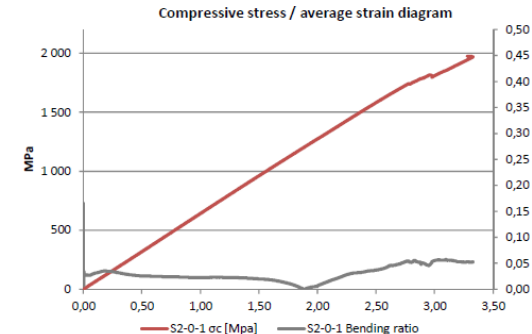
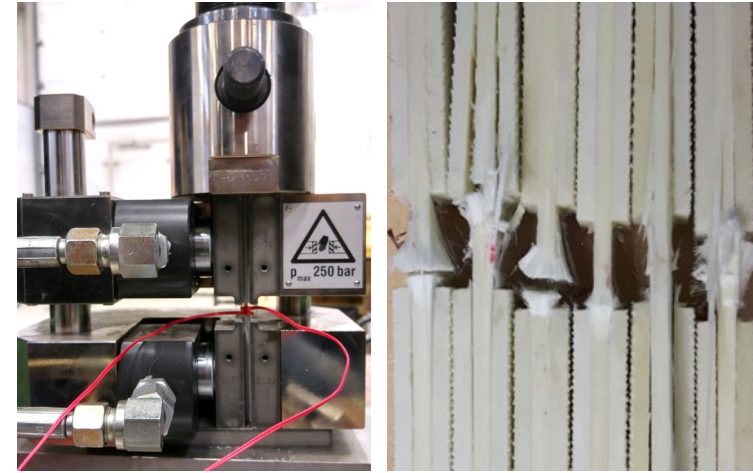


Compressive properties of lamina / laminates (in-plane)

- Typical test standards: ISO 14126 method 2, ASTM D 3410
- Requires precision fixtures with combined end and shear loading
- Specimen protected by end tabs to avoid premature fiber breakage in the clamped area, especially with unidirectional / high-modulus composites
- Data acquisition: force, displacement and strain
 - Strain measurements by strain gages
- Measured properties: bending / buckling strain, compressive strength, compressive modulus, Poisson's ratio, stress-strain response

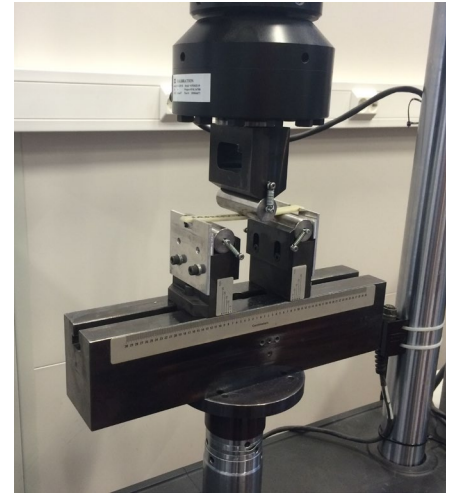
$$\left| \frac{\varepsilon_{11b} - \varepsilon_{11a}}{\varepsilon_{11b} + \varepsilon_{11a}} \right| \leq 0,1 \quad \sigma_{cM} = \frac{F_{max}}{bh} \quad E_c = \frac{\sigma_c'' - \sigma_c'}{\varepsilon_c'' - \varepsilon_c'}$$

- Modulus affected by calculation area (depends on applied standard)
- Test results affected by:
 - Test fixture and test method
 - Specimen orientation / fiber versus load axis misalignment
 - Improper coupon machining, specimen preparation, surface quality and application of strain gages
 - Grip pressure (hydraulic grips recommended) and geometry of end tab areas
 - Asymmetrical laminate structure induces bending
 - Specimen geometry: correct width-to-thickness ratio and minimal free length between end tabs



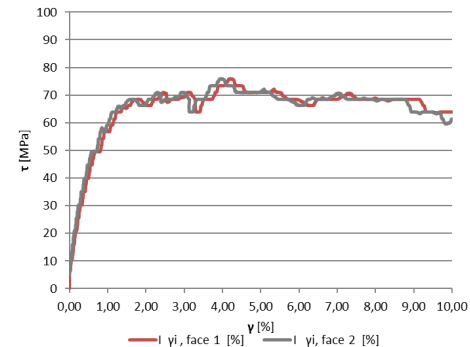
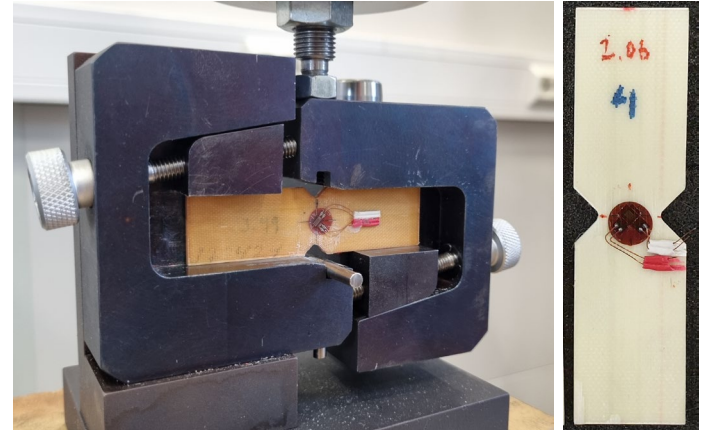
Flexural properties of lamina / laminates (in-plane)

- Typical test standards: ISO 14125, ASTM D 790
- Measured flexural properties are often highly dependent on stacking sequence, for highly orthotropic laminates, the maximum stress of a flexure specimen may not occur in the outermost fibers
- Other aspects:
 - Interactions with the loading surface geometry
 - End forces developed by large support span-to-specimen depth ratios
 - Shear deflections that result from low span-to-depth ratios
 - Three-point or four-point flexure
- The requirements for the test equipment are lower
- Data acquisition: force, displacement
- Measured properties: flexural strength, strain of outer surface, flexural modulus, stress-strain response
 - Three-point method $\sigma_f = \frac{3FL}{2bh^2}$ $\varepsilon = \frac{6sh}{L^2}$ $E_f = \frac{L^3}{4bh^3} \left(\frac{\Delta F}{\Delta s} \right)$
 - Four-point method $\sigma_f = \frac{FL}{bh^2}$ $\varepsilon = \frac{4,7sh}{L^2}$ $E_f = \frac{0,21L^3}{bh^3} \left(\frac{\Delta F}{\Delta s} \right)$
- Test results affected by:
 - Results sensitive to specimen and loading geometry and strain rate
 - Failure mode may be tension, compression, shear or combination
 - Compressive stresses on loading and support members



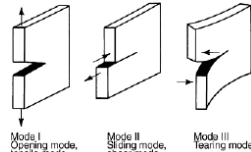
Shear properties of lamina / laminates (in-plane)

- Typical test standards: ASTM D 5379 (V-notch shear), ASTM D4255 (rail shear method), EN ISO 14129 ($\pm 45^\circ$ tension)
- V-notched beam shear test is often called the Iosipescu shear test
 - Provides the best shear response of the standardized methods
 - Notches influence the shear strain along the loading direction, making the distribution more uniform
- Requires precision fixtures, end tabs recommended if thin laminate is tested
- Several uncertainties: edge effects, material coupling effects, nonlinear behavior of the matrix or the fiber-matrix interface, imperfect stress distributions, or the presence of normal stresses
- Data acquisition: force, displacement and strain
 - Strain measurements by strain gages
- Measured properties: shear strength, shear strain, shear chord modulus, stress-strain response
$$F^u = P^u/A \quad \gamma_i = |\epsilon_{+45}| + |\epsilon_{-45}| \quad G^{chord} = \Delta\tau/\Delta\gamma$$
 - Shear strains in fiber-reinforced structures of 5 % is a practical upper bound, even with the most ductile fibers available. Also limited by strain gages
- Test results affected by:
 - Coarse features, such as fabrics with large filament count tows (12K or more) or certain braided materials
 - Localized failure points
 - Improper coupon machining and application of strain gages
 - Asymmetrical laminate structure may cause twisting during the loading



Toughness properties of laminates

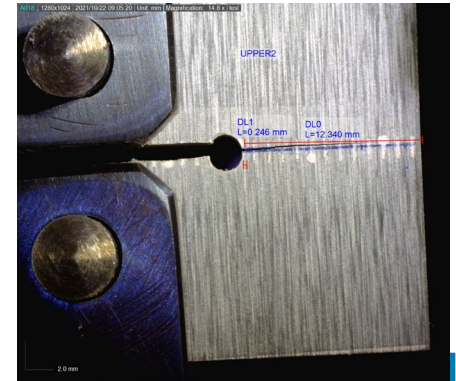
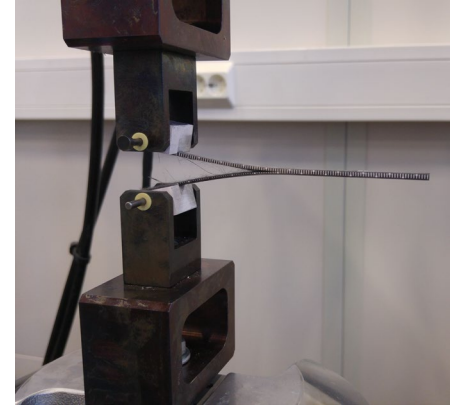
- Typical test standards: ASTM D 5528, ASTM D 5045, ISO 15024
- The critical energy release rate (G_c) is defined as the energy per unit of surface needed to propagate a crack over a known distance.
 - Mode I – Crack opening, tensile mode
 - Mode II – In-plane shear, sliding mode
 - Mixed Mode I/II Bending
 - Mode III – Tearing mode



- Data acquisition: force, displacement, propagation of crack
- For example, measured properties of compact tension test method (mode I): K_Q (conditional or trial K_{Ic}), critical-stress-intensity factor or interlaminar fracture toughness K_{Ic} critical strain energy release rate G_{Ic}

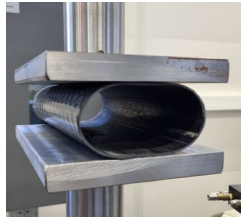
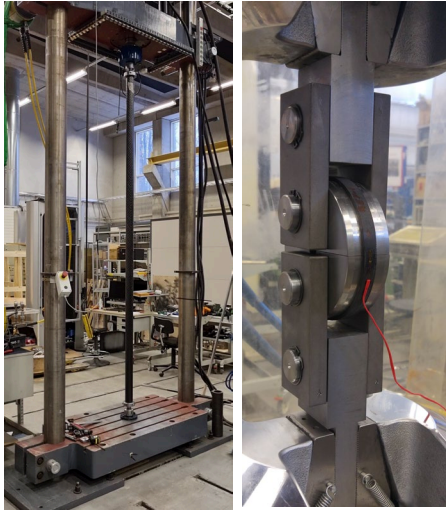
$$G_{Ic} = \frac{(1 - \nu^2)K_{Ic}^2}{E} \quad K_Q = (P_Q/BW^{1/2})f(x) \quad f(x) = \frac{(2+x)(0.886+4.64x - 13.32x^2 + 14.72x^3 - 5.6x^4)}{(1-x)^{3/2}}$$

- Validity criteria for specimen geometry must be satisfied (based specimen dimensions, K_Q and yield stress of tested material $B, a, (W - a) > 2.5 (K_Q/\sigma_y)^2$)
- Test results affected by:
 - Specimen geometry
 - Preparation of initial crack
 - Type of tested material and behavior during the test
 - Fiber bridging, artificially high initial toughness values, unstable propagation of delamination front

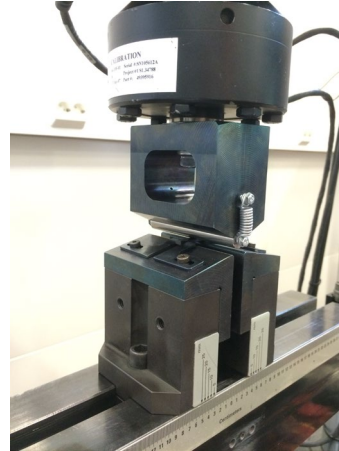


Other examples

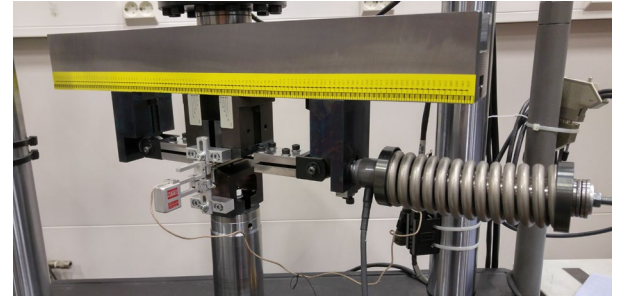
Pipe / tube structures



Quality control of laminates (adhesion)



Customer-specific tests



Test data normalization

- Normalization is a post-test adjustment process that eliminates artificial variation caused by local changes in constituent content
- Variation is normally due to locally changing fiber volume caused by locally greater or lesser amounts of matrix
- Normalization involves adjusting test values to account for changes in fiber volume, ensuring consistent results
- Normalization of the results can be done for the same product / the same laminate structure
- Fiber volume V_f and tested laminate property is assumed to be linear for all proportions of fiber and matrix, for example 0° tensile strength of a unidirectional laminate can be expressed:
$$F = V_f F_f + (1 - V_f) F_m$$
 or simplified
$$F = V_f F_f$$
- If an actual fiber volume V_f of tested material is determined by suitable method, measured value can be adjusted. Normalized tensile strength F_n for normalized fiber volume V_{fn} with tested strength F by a direct approach:

$$F_n = F \times \frac{V_{fn}}{V_f}$$

- Other option is to use cured ply thickness (CPT) approach with known fiber areal weight (FAW), fiber density ρ_f and normalized fiber volume V_{fn} . Normalized tensile strength F_n can be expressed:

$$F_n = F \times \frac{\text{CPT} \times \rho_f}{\text{FAW}} \times V_{fn}$$

- And for normalized cured ply thickness CPT_n , normalized tensile strength F_n :

$$F_n = F \times \frac{\text{CPT}}{\text{CPT}_n}$$

- Void content is not so large or localized that basic load-carrying capability is reduced
- Data normalization can be done, if mechanical test are properly performed (low variation in test results)

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