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M109

How to measure reinforcement content (and corresponding matrix content)

Foundational knowledge method

Document Type	Method
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Themes	• Flow and consolidation management
Relevant Class	Material
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Scope[edit | edit source]

This page outlines two methods that can be used to measure the reinforcement content of a composite material. In the case of fibre reinforcement, this is the fibre volume content (fibre volume fraction) of the composite. In addition to reinforcement content, the corresponding matrix content of the composite material is also determined. The two presented methods vary in their complexity, the time involved, and the information that can be obtained by the practitioner.

Significance[edit | edit source]

Determination of the reinforcement content, and the content of the other constituent materials in the composite is necessary for analytical modelling of material properties of the composite material. The reinforcement content in particular is commonly used as a comparison basis to evaluate the mechanical properties between different composite materials.

Prerequisites[edit | edit source]

None.

Overview[edit | edit source]

Provided on this page are two test methods that measure the reinforcement content of the composite. The methods are from the ASTM D3171 test standard [1], and Hexcel literature [2].

The provided table briefly summarizes each method with the level of complexity involved, any specialized equipment necessary, and general comments about the test methods. The table is meant to be an initial guide, where it is recommended that each method be reviewed in detail in order to determine the most appropriate method for your particular use.

	Test Method	Necessary Equipment	General Comments:
Simple	**Cured composite thickness measurement**	• Analytical balance • Calipers • Thickness measuring device (e.g. micrometer) •	The simpler of the outlined methods, it is suitable for flat composite laminates with minimal surface roughness. This method also inherently determines the corresponding matrix content of the composite (assuming that no voids are present). To perform the required calculations, it is necessary to know both the areal weight of the reinforcement and the density. For Fibre reinforcement, this is per ply areal weight, and fibre density. This method cannot determine the void content of the material.
Moderate	**Matrix removal (burn off or digestion)**	• Analytical balance • Calipers • Thickness measuring device (e.g. micrometer) • Wet chemistry laboratory facilities *(depending on matrix removal method)* • High temperature furnace equipment *(depending on matrix removal method)*	In this method, the matrix portion of a composite material sample of a known mass is removed. This is achieved by ignition, digestion, or carbonization. The weight percent of the left behind reinforcement material is then determined. Unlike the previous method, it is not necessary to know the areal weight or the density of the reinforcement material if content in only weight percent is to be determined. However, the density of the reinforcement is required for the determination of volume percentage. In addition to determining the reinforcement content and the matrix content, this method can determine the void content in the composite if the matrix density is known or is measured.

Cured Composite Thickness Measurement
Matrix Removal

Scope[edit | edit source]

This method determines the reinforcement content of the composite by calculating the mass proportion of reinforcement present relative to the total mass and dimensions of the composite sample. It is appropriate for flat and low voidage laminates (ideally no voids).

The equipment involved is minimal and the procedure is straight forward to carry out. It is required that both the reinforcement areal weight and density be known. With this information, the only measurements that are required are to measure the given sample weight, and sample geometry (length, width, and thickness).

The steps outlined here are from ASTM D3171 [1] and Hexcel literature [2].

Setup[edit | edit source]

Equipment[edit | edit source]

- Analytical balance
- Calipers
- Micrometer (or other suitable thickness measurement device)

Test specimen[edit | edit source]

- Rectangular, cubical, or circular is acceptable, as long as area and thickness can be measured.
- Sample size: ASTM D3171[1] recommends a sample surface area of at least 625mm^2.

Procedure[edit | edit source]

Sample Conditioning[edit | edit source]

Moisture uptake from a humid environment can lead to potential errors and inconsistency in the sample weight measurements. It is recommended that the samples be in a dry state prior to measurement. This can be achieved by pre-conditioning the samples in a desiccator and performing measurements within a few minutes of removal from the container. Please refer to ASTM D3171 for further details and recommendations.

Test Procedure[edit | edit source]

1. Weigh composite test sample.
2. Measure all dimensions of the composite test sample.
3. Perform calculations. (shown below)

For measuring sample thickness, a micrometer is recommended for taking measurements at the centre of the sample. For uneven rough surfaces (resulting from the use of release plies, breather, etc.), a ball micrometer of 6mm diameter is recommended by ASTM D3171 [1].

Analysis[edit | edit source]

Note that all the calculation units here are assuming base SI units. Some material properties are often reported in non-base units, e.g. density (often shown on data sheets as g/cm^3 rather than base: kg/m^3). Please ensure that proper unit conversions are applied if necessary.

Sample (composite material) density[edit | edit source]

\(\rho_c=\frac{M}{A\times h }\)

where,

\(\rho_c = \) Sample density [kg/m^3]

\(M = \) Sample mass [kg]

\(A = \) Sample area [m^2]

\(h = \) Sample thickness [m]

Reinforcement content[edit | edit source]

Weight fraction\[W_r=\frac{A_r \times N}{\rho_c \times h}\]

where,

\(A_r = \) Reinforcement areal weight per layer/ply [kg/m^2]

\(N = \) Number of reinforcement layers/plies

\(\rho_c = \) Sample density [kg/m^3]

\(h = \) Sample thickness [m]

Volume fraction\[V_r=\frac{A_r \times N}{\rho_r \times h}\]

where,

\(A_r = \) Reinforcement areal weight per layer/ply [kg/m^2]

\(N = \) Number of reinforcement layers/plies

\(\rho_r = \) Reinforcement (e.g. fibre) density [kg/m^3]

\(h = \) Sample thickness [m]

Matrix content[edit | edit source]

Weight fraction\[W_m=100-\Biggl(\frac{A_r \times N}{\rho_c \times h}\Biggr)\]

where,

\(A_r = \) Reinforcement areal weight per layer/ply [kg/m^2]

\(N = \) Number of reinforcement layers/plies

\(\rho_c = \) Sample density [kg/m^3]

\(h = \) Sample thickness [m]

Volume fraction\[V_m=\frac{W_m \times \rho_c}{\rho_m}\]

where,

\(W_m = \) Weight fraction matrix

\(\rho_c = \) Composite density [kg/m^3]

\(\rho_m = \) Matrix (e.g. epoxy) density [kg/m^3]

Limitations[edit | edit source]

This method requires knowledge of the reinforcement areal weight (assuming relative constancy) and density. Without these two values, the required calculations cannot be applied.

This method also assumes that no voids are present in the composite. While the assumption does not affect the calculation of the reinforcement content, it can lead to errors in the subsequent matrix content determination. Any voids that are present are lumped together with the matrix proportion of the composite.

Scope[edit | edit source]

The following method summary outlines the working principle and procedure found in ASTM D3171: **Standard Test Methods for Constituent Content of Composite Materials**. Please refer to the test standard document for further details [1].

This method determines the reinforcement content of the composite material by removing the matrix component. The left behind reinforcement material is then weighed and compared to the initial weight of the composite sample. It can determine the weight fraction of the reinforcement without knowing the reinforcement density. (Determination of reinforcement volume fraction however, requires knowledge of the reinforcement density.)

Removal of the matrix component can be achieved by either digestion (by chemical reagent), using a high temperature environment to burn off, or carbonizing the matrix material. It requires that the reinforcement material is left unaffected by this matrix removal process.

Setup[edit | edit source]

Equipment[edit | edit source]

General equipment:

- Analytical balance
- Calipers
- Micrometer (or other suitable thickness measurement device)

Digestion removal of matrix:

- Suitable reagents see procedure list

Burn off removal of matrix:

- Muffle furnace

Carbonization of matrix:

- Nitrogen-purging furnace

Test specimen[edit | edit source]

- Rectangular, cubical, or circular is acceptable, as long as area and thickness can be measured.
- Sample size: ASTM D3171[1] recommends a sample weight of at minimum 0.5g. A larger the sample reduces the sensitivity to errors in the present of voids.

Procedure[edit | edit source]

Digestion Reagents[edit | edit source]

If matrix removal by digestion is intended, the following reagents are prescribed by ASTM D3171[1] as being appropriate for the following matrix materials. Note: the reagents listed are suitable for polymer and metal matrices.

Reagent	Suitable Matrix Materials
Concentrated nitric acid	epoxy, steel, copper
Aqueous mixture of sulfuric acid and hydrogen peroxide	epoxy, phenolic, polyamide, thermoplastics
Mixture of ethylene glycol and potassium hydroxide	epoxy
Sodium hydroxide solution	aluminum, brass
Hydrochloric acid	steel, titanium, copper, aluminum

Sample Conditioning[edit | edit source]

Moisture uptake from a humid environment can lead to potential errors and inconsistency in the sample weight measurements. It is recommended that the samples be in a dry state prior to measurement. This can be achieved by pre-conditioning the samples in a desiccator and performing measurements within a few minutes of removal from the container. Please refer to ASTM D3171 for further details and recommendations.

Test Procedure[edit | edit source]

Due to the complexities and procedure variations associated with the variety of methods available to remove the composite matrix component, please refer directly to ASTM D3171[1] for specific procedural steps.

Analysis[edit | edit source]

The following calculations only apply to the techniques where the entire matrix component of the composite is removed. For the matrix carbonization technique, the residual matrix reminants must be accounted for in the calculations. Please see ASTM D3171 for the details.

Note that all the calculation units here are assuming base SI units. Some material properties are often reported in non-base units, e.g. density (often shown on data sheets as g/cm^3 rather than base: kg/m^3). Please ensure that proper unit conversions are applied if necessary.

Reinforcement content[edit | edit source]

Weight fraction$$W_r=\frac{M_f}{M_i}$$

where,

$M_f =$ Final mass of sample after matrix removal [kg]

$M_i =$ Initial mass of composite sample [kg]

Volume fraction$$V_r=\frac{M_f}{M_i} \times \frac{\rho_c}{\rho_r}$$

where,

$\rho_c =$ Composite sample density [kg/m^3]

$\rho_r =$ Reinforcement (e.g. fibre) density [kg/m^3]

Matrix content[edit | edit source]

Weight fraction$$W_m=\frac{M_i - M_f}{M_i}$$

Volume fraction$$V_m=\frac{M_i - M_f}{M_i} \times \frac{\rho_c}{\rho_m}$$

where,

$\rho_m =$ Matrix (e.g. epoxy) density [kg/m^3]

Void content[edit | edit source]

Volume fraction\[V_v=100 - \bigl(V_r + V_m\bigr)\]

Limitations[edit | edit source]

This method is highly limited by the equipment the practitioner may have available, and their individual training background in chemistry or a high temperature laboratory setting.

For matrix removal by digestion, a proper wet chemistry facility is required, and the practitioner must also be appropriately trained for the chemical laboratory.

Matrix removal by burn off or carbonization requires a high temperature furnace. Temperatures above 500°C are required to burn off removal thermoset matrix materials, and temperatures even higher than this are required for thermoplastics.

It also has to be ensured that the reinforcement material is left unaffected by whatever matrix removal process is employed. This must be considered when deciding upon an appropriate matrix removal method.

Related pages

Page type	Links
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References

1. ↑ [1.0] [1.1] [1.2] [1.3] [1.4] [1.5] [1.6] [1.7] [Ref] ASTM International (2015), *ASTM D3171-15, Standard Test Methods for Constituent Content of Composite Materials*, ASTM International, doi:10.1520/D3171-15
2. ↑ [2.0] [2.1] [Ref] Hexcel Composites (1997), *Prepreg Technology (Publication No. FGU 017)*, Hexcel Corporation

About **Help**

The continuous material phase that binds the reinforcement together, maintains shape, transfers load, protects the reinforcement from environment and damage, and provides the composite support in compression.

Desirable characteristics:

- Moisture/chemical resistance
- Low density
- Processability

Engineered materials (designed to have specific properties) made from two or more constituent materials with different physical or chemical properties. The constituents remain separate and distinct on a macroscopic level within the finished structure.

Volume fraction of either matrix or fibres with respect to total composite volume (matrix + fibre).

The individual materials that combine to form the composite material. The constituent materials are separate and distinct on a macroscopic level.

The use of multiphysics models to predict the outcome of real-world scenarios. May be analytical (closed form, "hand calculations") or computational (implementation on computers is required due to the large number of calculations involved. e.g. finite element method, finite difference method)

Most often in composite materials engineering, modelling refers to either:

- Process modelling (predicting manufacturing outcomes, or the inverse problem of predicting manufacturing parameters to achieve a desired outcome), or
- Performance modelling (predicting the stiffness/strength/suitability of a structure, or the inverse problem of the material properties and dimensional requirements to achieve a desired stiffness/strength/suitability of a structure)

(same as "Simulation")

Areal weight (or fibre areal weight, AW) refers to the mass/weight of fibre per unit area. (Typically in g/m^2 gsm) or ounces/$yard^2$ (often just called ounces). Areal weight depends on tow size and fibre architecture (weaving, density, etc.).

Weight fraction of either matrix or fibres with respect to total composite weight (matrix + fibre).

Thermosets are a class of polymer that undergo polymerization and crosslinking during curing with the aid of a hardening agent and heating or promoter. Initially they behave like a viscous fluid. During curing, they change from viscous fluid to rubbery gel (viscoelastic material) and finally glassy solid.

If heated after curing, initially they become soft and rubbery at high temperatures. If further heated, they do not melt but decompose (burn)

Comes in two parts: part A (resin) and B (hardener). When mixed, curing reaction starts and is not reversible.

Examples include epoxy or polyester.