



**International
Standard**

ISO 19350

**Recycled carbon fibre —
Determination of tensile strength
distribution and interfacial
shear strength of single filament
embedded in matrix polymer**

*Fibre de carbone recyclée — Détermination de la distribution
de la résistance à la traction et de la résistance au cisaillement
interfacial d'un filament unique noyé dans une matrice polymère*

**First edition
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Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastic*, Subcommittee SC 13, *Composite and reinforced fibre*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The properties of recycled carbon fibres are potentially different from those of the original virgin carbon fibres. Therefore, it is very important to evaluate the properties of recycled carbon fibres in order to reuse them as reinforcing fibres. The tensile strength distribution and interfacial shear strength of recycled carbon fibres are essential for the production of composite materials using recycled carbon fibres. This document describes how to measure the tensile strength distribution and interfacial shear strength of recycled carbon fibres.

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Recycled carbon fibre — Determination of tensile strength distribution and interfacial shear strength of single filament embedded in matrix polymer

1 Scope

This document specifies methods for the simultaneous measurement of the fibre tensile strength distribution, and the fibre resin interfacial shear strength of recycled carbon fibres using the modified fragmentation test^[1].

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10618, *Carbon fibre — Determination of tensile properties of resin-impregnated yarn*

ISO 11566, *Carbon fibre — Determination of the tensile properties of single-filament specimens*

ISO 11567, *Carbon fibre — Determination of filament diameter and cross-sectional area*

3 Terms, definitions and symbols

3.1 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.2 Symbols

Symbol		Definition
E	fibre tensile modulus, GPa	Tensile modulus of the recycled carbon fibres
D	average fibre diameter, μm	Average fibre diameter of the recycled carbon fibres
L	Gauge length, mm	Initial distance between grips
l	Total fibre length, mm	Total length of a fibre within the observed area of all specimens
l_i	Fibre length, mm	Length of a fibre within the observed area of each specimen i
m	Weibull shape parameter	Shape parameter of the 2-parameters' Weibull distribution
$N(\epsilon_{\text{fibre}})$	Total number of break points	Total number of fibre break points in all specimens at ϵ_{fibre}
Δ	Displacement, mm	Displacement given between gauges
$\Delta\epsilon_{\text{film}}$	Incremental film strain	The magnitude of the strain on the film in each step

Symbol		Definition
$\Delta n_i (\epsilon_{\text{film}})$	Incremental number of fibre break points	Incremental number of fibre break points within each specimen i at ϵ_{film} .
$\Delta N (\epsilon_{\text{fibre}})$	Total incremental number of fibre break points	Total incremental number of fibre break points within each specimen i at ϵ_{fibre}
ϵ_{film}	Film strain	Strain applied to the specimen film. Displacement divided by gauge length
ϵ_0	Thermal shrinkage	In-plane film shrinkage strain during specimen moulding. In this document, the shrinkage direction is positive direction, and the expansion direction is negative direction
ϵ_{fibre}	Fibre strain	Film strain minus thermal shrinkage during moulding
$\lambda (\epsilon_{\text{fibre}})$	Density of break points, m^{-1}	Total break point number divided by fibre length
λ_{∞}	Saturated density of break points, m^{-1}	Break point density at the end of tests
τ	Interfacial shear strength, MPa	Interfacial shear strength between recycled carbon fibre and matrix polymer
σ_0	Weibull scale parameter, MPa	Scale parameter of the 2-parameters Weibull distribution

4 Principle

The tensile strength distribution (Weibull parameters) and interfacial shear strength of recycled carbon fibres are evaluated using a modified fragmentation test^[1]. In this test, a single fibre embedded in a polymer film is pulled in steps and the number of fibre breaks at each step is counted. From the double logarithm graph of the fibre break point density and fibre strain, the fibre tensile strength distribution and interfacial shear strength are evaluated simultaneously, assuming that the strength of the recycled carbon fibre follows a two-parameter Weibull distribution.

5 Tensile test machine and specimen

5.1 Tensile test machine

The tensile test machine shall be able to measure the displacement applied to the specimen. In addition, the displacement of the crosshead of the machine shall be accurate to less than 1 μm . To observe fibre breaks, the specimen should be observed at high magnification observation during the fragmentation test. Therefore, for example, the test machine should be placed under a microscope, or a microscope should be attached to the test machine.

5.2 Grip

For accurate measurement of displacement, the grips should be free of slippage. The gripping surfaces should be parallel in order to suppress local deformation of the film.

5.3 Microscope

A microscope capable of observing the break points of a single recycled carbon fibre should be used. Video or photographic documentation is preferred.

5.4 Test specimens

The specimen is a polymer sheet with a single recycled carbon fibre embedded at the centre.

The Young's modulus and fibre diameter of the fibre to which this document applies shall already be known using ISO 11567, and ISO 10618 or ISO 11566, respectively. The thermal shrinkage of the polymer shall also be known.

The sheet embedded recycled carbon fibre is produced by press moulding. Two polymer sheets with a thickness of more than 100 µm are prepared. In the case of thermosets, semi-cured sheets are used. A recycled carbon fibre is sandwiched between two polymer sheets. The sheets are pressed with a spacer more than 200 µm thick to control the thickness of the sandwiched sheet. In the case of a long recycled carbon fibre, the ends of the recycled carbon fibre should be fixed to prevent the fibre from bending during pressing. The pressing temperature is determined by the melting or curing temperature of the polymer used. It is desirable to press under vacuum to remove the air in the sandwich sheet.

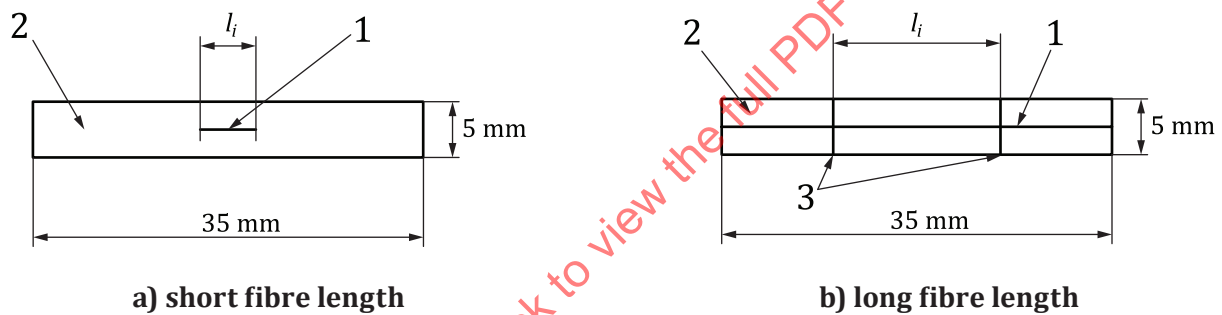
The specimen is prepared by cutting out a 5 mm × 35 mm from the sandwich sheet. The fibre should be at the centre of the film and the fibre axis should be parallel to the long side of the specimen.

Recycled carbon fibre from PAN-based or pitch-based CF can be used. The length of the recycled carbon fibre should be longer than 3 mm. The specimen thickness should be greater than 200 µm. The variation in specimen thickness should be less than 5 % to apply uniform strain.

Although thermosets or thermoplastics can be used as the polymer film material, transparency of the specimen is essential to confirm fibre breaks.

If the fibre length is longer than the observation area or if the strain is calculated from optical observations, draw the marker lines.

NOTE If the test machine is not capable of using standard specimens, the different film size can be used if the film thickness requirement is met.



Key

- 1 carbon fibre
- 2 polymer film
- 3 marker line

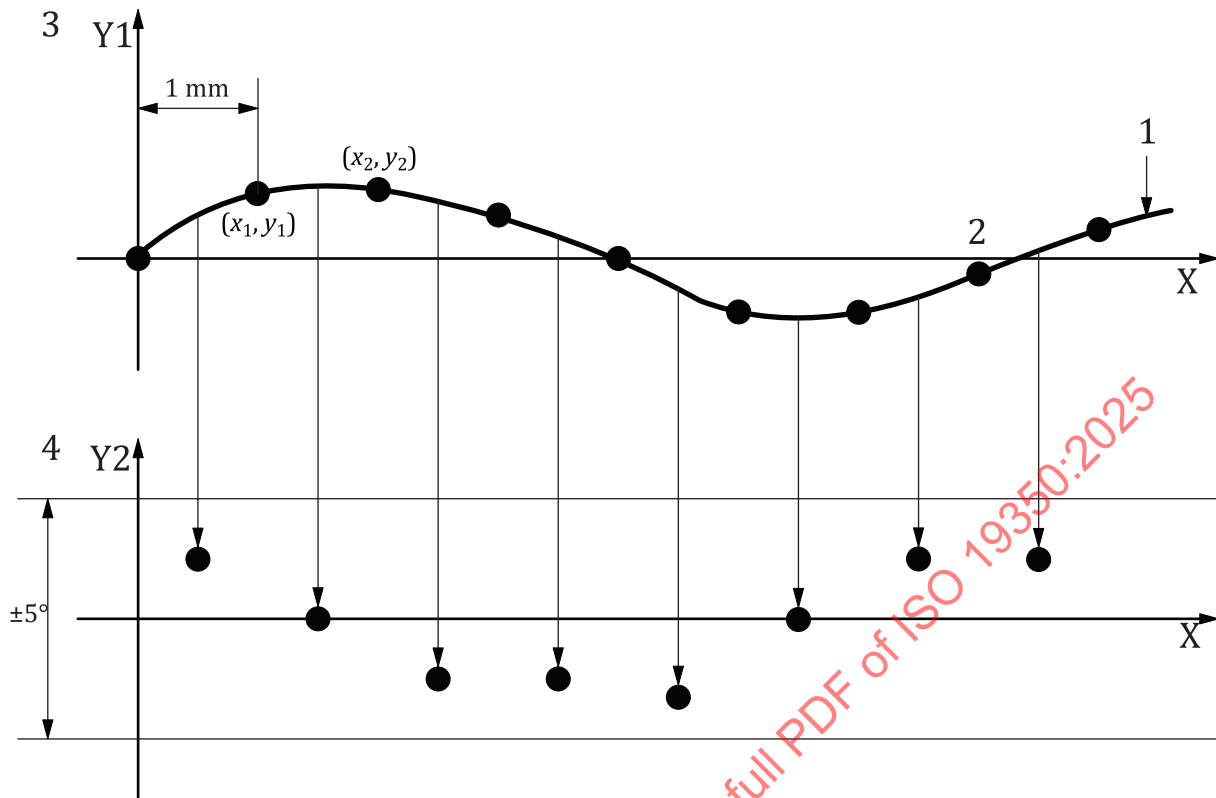
Figure 1 — Test specimens for short fibre length and long fibre length comparing to observation area

Check the straightness of the fibre in the specimen. Check the position of the fibre (x_i, y_i) approximately every 1 mm in the x-axis direction and calculate the straightness of the fibre using [Formula \(1\)](#), where the x-axis is in the tensile direction. If the observed point has a waviness of 5° or more with respect to the tensile direction, it shall not be accepted as a specimen.

$$\theta_i = \text{Arctan} \left(\frac{y_{i+1} - y_i}{x_{i+1} - x_i} \right) \quad (1)$$

where

- θ_i is the waviness angle.
- x_i is the x coordinate position of i-th point.
- y_i is the y coordinate position of i-th point.
- $\text{Arctan}(x)$ is the inverse function of $\tan(x)$.



Key

- X position in tensile direction of carbon fibre
- Y1 position in perpendicular to tensile direction of carbon fibre
- Y2 waviness angle of θ
- 1 carbon fibre
- 2 polymer film

Figure 2 — Plots of (3) fibre position and (4) fibre waviness angle for checking fibre straightness

5.5 Number of tests

The total fibre length, l , shall be at least 100 mm and preferably greater than 150 mm.

For example, if the fibre length, l_f , is 5 mm, at least 20 specimens should be prepared.

6 Testing technique

6.1 Test procedure

Place the specimen in the tensile test machine so that the gauge length, L , is 20 mm. When the film is held by the grips, the grips should hold the specimen evenly in the thickness direction.

The specimen should be gripped without wrinkles, because the wrinkles of the specimen lead to the error in the calculation of the strain. In order to attach the specimen without wrinkles, when one side of the film is gripped, the other side of the edge of the film is slightly tensioned and gripped in order to prevent wrinkles. The tension load should be less than 0,5 N.

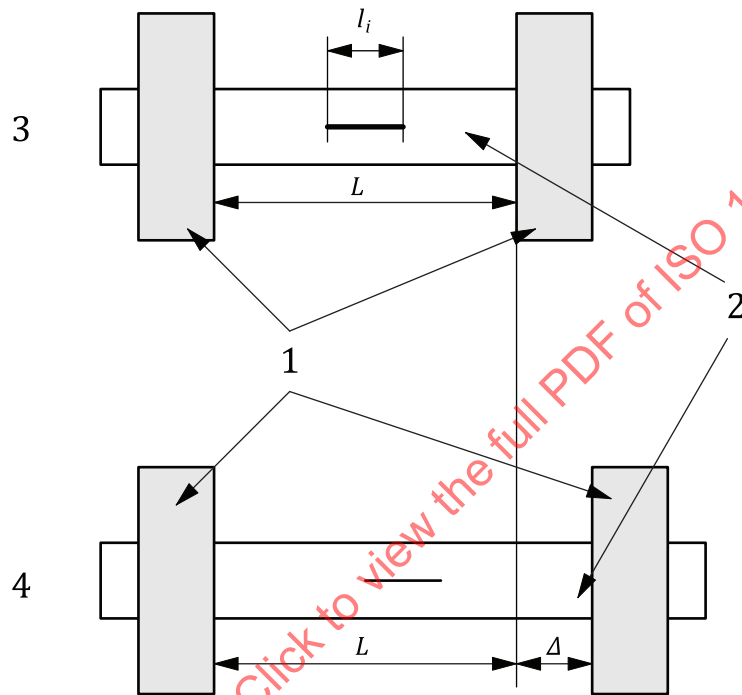
Start by stretching the film so that film strain, ϵ_{film} , is 3,5 %. If many break points appear, change the specimen, and give a film strain smaller than 3,5 %.

Then apply the film strain, ϵ_{film} , in steps and measure the incremental number of fibre break points, $\Delta n_i(\epsilon_{\text{film}})$, in the observation area. The incremental film strain, $\Delta\epsilon_{\text{film}}$, given in steps, should be less than 0,25 % up to 7,5 % and less than 0,5 % above 7,5 %. The strain rate should be less than 0,05 %/s and the maximum strain should be greater than 10 %.

NOTE 1 If observation records can be kept by videotaping or photography, the number of incremental fracture points can be determined from the records after the experiment.

NOTE 2 The incremental film strain, $\Delta\epsilon_{\text{film}}$, can be calculated from the amount of movement of the marker lines.

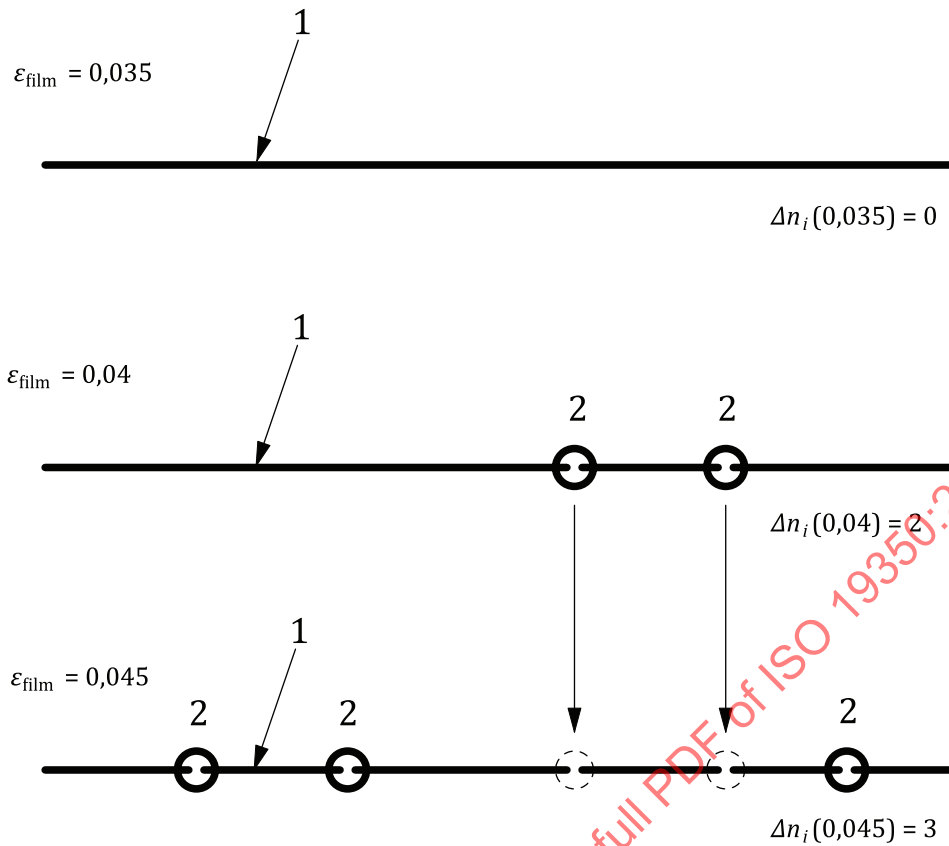
NOTE 3 If the test machine is not capable of using standard specimens, the gauge length can be changed if the strain requirement is met.



Key

- 1 grip
- 2 specimen

Figure 3 — Schematic of the test for (3) initial position and (4) after deformation



Key

- 1 carbon fibre
- 2 break

Figure 4 — Method of counting incremental number of fibre break points

6.2 Validity of the test

If a specimen is pulled out of the grips, the result shall be excluded.

If the specimen is locally deformed due to necking, etc., during the tensile test, the result shall be excluded.

If the number of break points does not reach saturation, the result shall be discarded.

7 Calculation of results

7.1 Summary of measurement data

The fibre strain, $\varepsilon_{\text{fibre}}$, is given by [Formula \(2\)](#):

$$\varepsilon_{\text{fibre}} = \varepsilon_{\text{film}} - \varepsilon_0 \quad (2)$$

where: $\varepsilon_{\text{film}}$ is the film shrinkage; ε_0 is thermal shrinkage.

The total number of break points, $N(\varepsilon_{\text{fibre}})$, at each fibre strain, $\varepsilon_{\text{fibre}}$ is given by [Formula \(3\)](#):

$$N(\varepsilon_{\text{fibre}}) = \sum_{\varepsilon \leq \varepsilon_{\text{fibre}}} \Delta N(\varepsilon) \quad (3)$$

where $\Delta N(\varepsilon_{\text{fibre}})$ is the total incremental number of break points and given by [Formula \(4\)](#):

$$\Delta N(\varepsilon_{\text{fibre}}) = \Delta N(\varepsilon_{\text{film}} - \varepsilon_0) = \sum_i \Delta n_i(\varepsilon_{\text{film}}) \quad (4)$$

where $\Delta n_i(\varepsilon_{\text{film}})$ is the incremental number of fibre break points of each specimen.

The density of break points, $\lambda(\varepsilon_{\text{fibre}})$ is given by [Formula \(5\)](#):

$$\lambda(\varepsilon_{\text{fibre}}) = \frac{N(\varepsilon_{\text{fibre}})}{l} \quad (5)$$

Where l is the total fibre length and given by [Formula \(6\)](#):

$$l = \sum_i l_i \quad (6)$$

where l_i is the fibre length of each specimen.

7.2 Calculation of tensile strength distribution

Plot a double logarithm graph of both the breakpoint density, $\lambda(\varepsilon_{\text{fibre}})$, and the fibre strain, $\varepsilon_{\text{fibre}}$.

Find the slope of the graph, a , and the intercept of the graph, b , by linearly fitting the linear part of both logarithmic graphs in the low strain region.^[1] Find the density of saturated break points, λ_{∞} , from the maximum value of the breakpoint density.

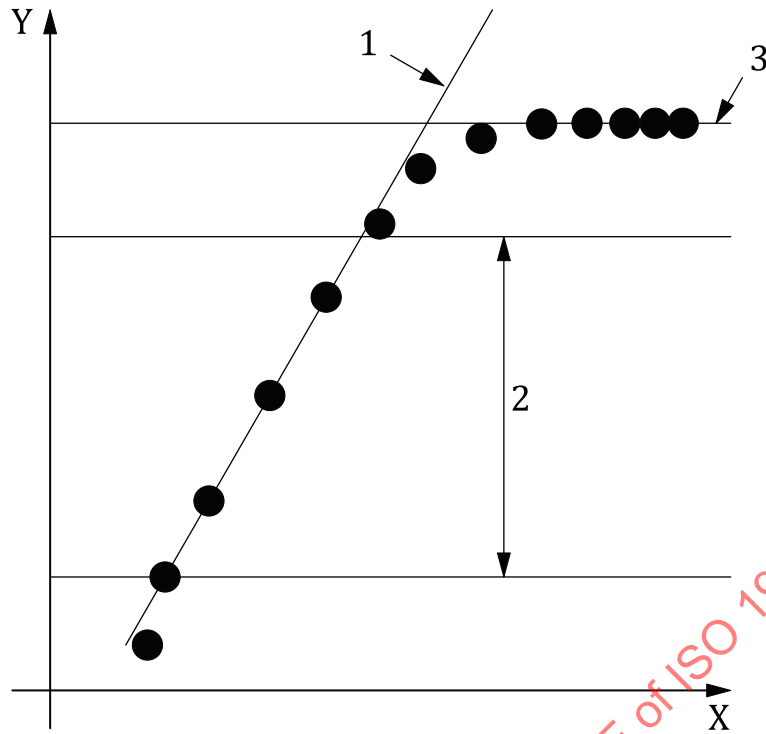
Calculate the Weibull parameter and the interfacial shear strength.

Weibull shape parameter, m , is given by [Formula \(7\)](#):

$$m = a \quad (7)$$

Weibull scale parameter, σ_0 , is given by [Formula \(8\)](#):

$$\sigma_0 = E \exp\left(-\frac{b}{a}\right) \quad (8)$$



Key

- X $\ln(\epsilon - \epsilon_0)$
- Y $\ln(\lambda)$
- 1 fitting line
- 2 fitting range
- 3 $\ln(\lambda_\infty)$

Figure 5 — Double logarithm graph of the density of break points and the fibre strain

7.3 Calculation of interfacial shear strength

The interfacial shear strength, τ , is given by [Formula \(9\)](#):

$$\tau = \frac{3D\sigma_f}{2l_c} \quad (9)$$

where l_c is the critical fibre length, and σ_f is the fibre strength.

The critical fibre length, l_c , and the fibre strength, σ_f , are given by [Formulae \(10\)](#) and [\(11\)](#), respectively:

$$l_c = \frac{4}{3(\lambda_\infty + 1)} \quad (10)$$

$$\sigma_f = \sigma_0 \left(\frac{l_c}{L_0} \right)^{\left(-\frac{1}{m} \right)} \Gamma \left(1 + \frac{1}{m} \right) = \sigma_0 \left(\frac{4}{3L_0(\lambda_\infty + 1)} \right)^{\left(-\frac{1}{m} \right)} \Gamma \left(1 + \frac{1}{m} \right) \quad (11)$$

where $\Gamma(x)$ is the gamma function.

7.4 Validity of the test result

The following results are unacceptable.