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BSI Standards Publication

Carbon-fibre-reinforced plastics — Determination of the size and aspect ratio of crushed objects

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National foreword

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Carbon-fibre-reinforced plastics — Determination of the size and aspect ratio of crushed objects

Plastiques renforcés de fibres de carbone — Détermination des dimensions et du rapport d'apparence d'objets broyés

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Foreword

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The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

INTERNATIONAL STANDARD ISO 30012:2016(E)

Carbon-fibre-reinforced plastics — Determination of the size and aspect ratio of crushed objects

1 Scope

This International Standard specifies test methods for measurement of the size and aspect ratio of crushed carbon-fibre-reinforced plastics (CFRP), especially for recycling purpose. In this International Standard the shape of crushed CFRP, the fragment, is treated as a rectangular shape, and the measurement of the long and short sides of the shape is described. It applies to fragments of the following average dimensions:

— length of the long side: 5 mm to 50 mm;

— width of the short side: 1 mm to 10 mm.

This International Standard provides three measuring methods, two methods are manual methods using microscope and scale and the third method is an automatic method using a measuring apparatus.

Crushed CFRP obtained from thermosetting or thermoplastic resin matrices are covered by this International Standard.

NOTE If the crushed CFRP contain a lot of small fragments and fine particle, it is intended to screen out by a sieve of 1 mm size before the measurement.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 apply.

4 Symbols

W	width of a CFRP fragment represented by the short side of a rectangular shape
L	length of a CFRP fragment represented by the long side of a rectangular shape
$\overline{W}$	average width of CFRP fragments
$\overline{L}$	average length of CFRP fragments
R_A	aspect ratio of CFRP fragments, calculated as the ratio of the length to the width of CFRP fragment
$\sigma(L)$	standard deviation of length of CFRP fragments

$\sigma(W)$ standard deviation of width of CFRP fragments

5 Principle

The fragments obtained from crushing CFRP laminates are impartially sampled and measured.

Most fragments of CFRP have a shape that can be approximate to a rectangle. Each fragment is observed with an optical microscope or equivalent equipment and the width and length of the rectangle are measured.

At least 100 fragments are measured. The size and aspect ratio of crushed CFRP fragments are reported as average and standard deviation of W, L, and average of aspect ratio R_A.

6 Apparatus

6.1 Stereomicroscope, comprised of a light source, scales and a stage which supports a glass plate carrying the fragment of CFRP. Magnification should be variable up to 10×. Scales shall be readable to 0,1 mm. (Method A).

6.2 Projection microscope, comprised of a projector, light source, scales, and a stage which supports a slide carrying the fragment of CFRP. Magnification should be variable up to 10×. Scales shall be readable to 0,1 mm. (Method B).

NOTE A personal computer, connected to the projection microscope and with a software for measuring the length and width of the fragments enables faster and easier measurements to be made (Method B).

6.3 Automatic size and shape measurement apparatus, such as an image analyser with magnification for carrying out semi-automatic measurement of the length and width of the CFRP fragments. (Method C).

6.4 Calibrated rule or **scale**, used to calibrate measurement scales on microscope and projection equipment.

7 Sampling

Unless otherwise specified, the following sampling procedure shall be followed.

7.1 Crushed CFRP is sampled from a pile into a bag of 20 l to 50 l volume.

CAUTION — Use caution when handling CFRP fragments. Handling them with unprotected hands can easily result in minor irritation/injury due to CFRP slivers. Protective gloves made of impervious material should be used.

7.2 Take three samples from one bag. Avoid samples of outer locations of around 20 % of the total volume. Take three samples from inner locations impartially, e.g. upper, centre and lower locations.

NOTE Distribution of size or shape of the CFRP fragments may not be identical among locations in the bag, due to sampling or transportation causes.

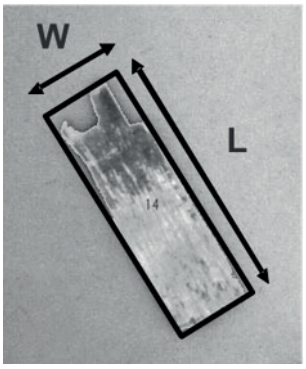
7.3 Mix the three samples impartially for the measurement. The total amount of the sample shall be at least 100 fragments to be measured.

8 Measurement

8.1 General

The shape of each fragment is approximated to a rectangle circumscribing the fragment as represented in Figure 1. The shape of the rectangle is defined so as to minimize the value of *W*.

NOTE Short fragments with length of 2 mm or shorter can be omitted from the following measurements. Total amount of the non-measured fragments can be counted and recorded in percentage.



Key

L length of the fragment

W width of the fragment

Figure 1 — Length and width of the fragment of CFRP

8.2 Method A: Manual method (stereomicroscope)

8.2.1 Each fragment is arranged on the plate on the stereomicroscope. The magnification of the microscope is adjusted appropriately to the average size of the fragments.

Figure 2 a) and Figure 2 b) are examples of unidirectional fibre type (UD) CFRP and fabric type CFRP on the plate of microscope, respectively.

8.2.2 Calibrate the measurement scale with a calibrated ruler or scale

8.2.3 The fragment is aligned with the long side parallel to one of the scales.

8.2.4 The length of the long side of the fragment is measured as length *L* and the short side as width *W*. Measure the length and width to 0,1 mm.

8.2.5 Repeat the measurement for at least 100 fragments.

8.3 Method B: Projection microscope

8.3.1 Arrange an adequate number of fragments on the plate. Distribute fragments in order to avoid any contact or overlapping between them.

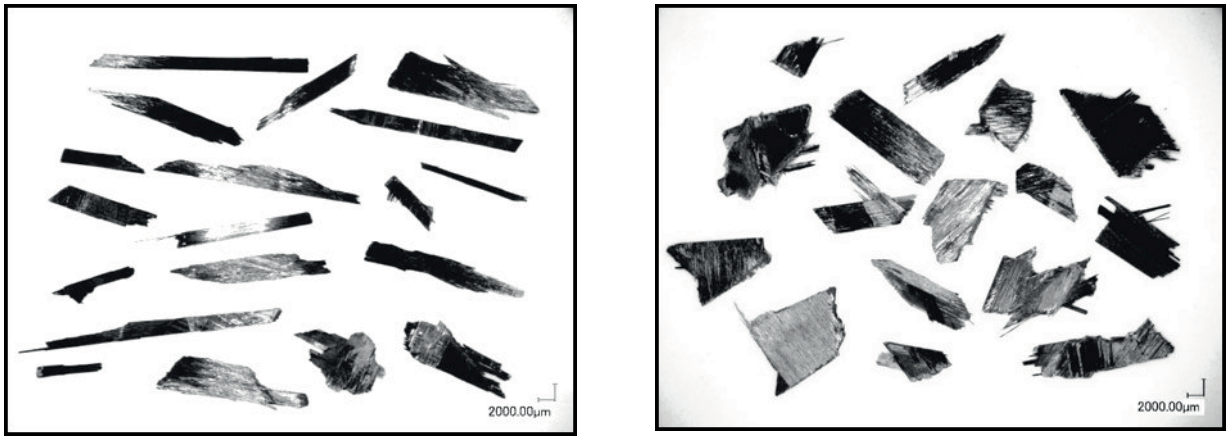
8.3.2 Set the glass plate on the stage of the microscope connected to the personal computer.

8.3.3 Calibrate the personal computer measurements using a calibrated ruler.

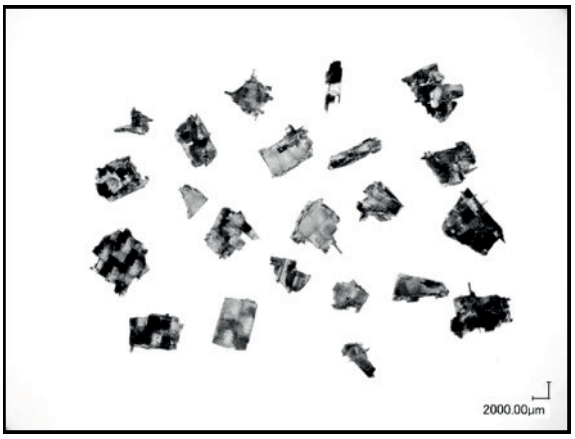
8.3.4 The magnification of the microscope is adjusted appropriately to the average size of the fragments.

8.3.5 The observed fragments are projected on the monitor. Measure the length *L* and the width *W* of the fragment to 0,1 mm.

8.3.6 Repeat the measurement for at least 100 fragments.



a) Examples of crushed UD type CFRP



b) Example of crushed fabric type CFRP

Figure 2 — Examples of crushed CFRP fragments on the plate of the microscope

8.4 Method C: Automatic shape and size measurement apparatus

8.4.1 Arrange an adequate number of fragments on the stage of the apparatus. Distribute fragments in order to avoid any contact or overlapping between them.

8.4.2 The observed fragments are projected on the monitor.

8.4.3 All CFRP fragments are approximated as a rectangular shape by the construction lines shown in Figure 3. The length *L* and width *W* of each fragment is measured to 0,1 mm, and recorded automatically.

8.4.4 Repeat the measurement for at least 100 fragments.

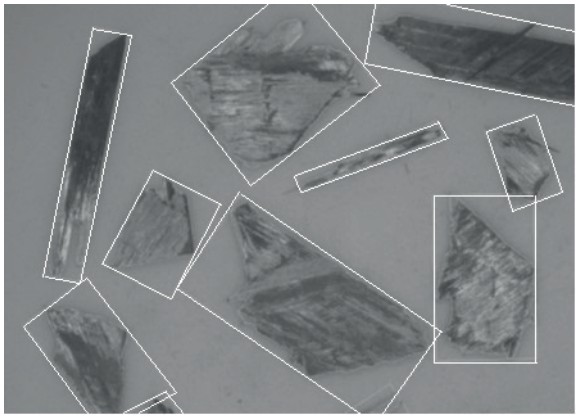


Figure 3 — Example of crushed CFRP fragments, approximated as rectangular shapes (shown by the lines) for method C

9 Calculation and expression of results

9.1 The average length, $\bar{L}$, and the average width, $\overline{W}$, of fragments are calculated using Formula (1) and Formula (2), respectively:

$$\bar{L} = \frac{\sum L_n}{N} \quad (n = 1 \text{ to } N) \tag{1}$$

$$\overline{W} = \frac{\sum W_n}{N} \quad (n = 1 \text{ to } N) \tag{2}$$

where

$\bar{L}$ is the average length of fragments;

$\overline{W}$ is the average width of fragments;

n is the measurement number;

N is the total number of fragments assessed;

L_n is the measurement length of the n-th fragment;

W_n is the measurement width of the n-th fragment.

NOTE Log or Log natural of $\bar{L}$ or $\overline{W}$ can be used instead of the measured data.

9.2 The standard deviation $\sigma(L)$ and $\sigma(W)$ of length L and width W of fragments are calculated using Formula (3) and Formula (4), respectively:

$$\sigma(L) = \left[\frac{\sum \left(L_n - \overline{L} \right)^2}{N-1} \right]^{1/2} \quad (n = 1 \text{ to } N) \tag{3}$$

$$\sigma(W) = \left[\frac{\sum \left(W_n - \overline{W} \right)^2}{N-1} \right]^{1/2} \quad (n = 1 \text{ to } N) \tag{4}$$

where

$\sigma(L)$ is the standard deviation of length L;

$\sigma(W)$ is the standard deviation of width W.

NOTE Log or Log natural of $\overline{L}$ or $\overline{W}$ can be used instead of the measured data.

9.3 The aspect ratio R_A of each fragment and the average of the R_{An} are calculated using Formula (5) and Formula (6), respectively:

$$R_{An} = \frac{L_n}{W_n} \tag{5}$$

$$\overline{R_A} = \frac{\sum R_{An}}{\mathrm{N}} \quad (n = 1 \text{ to } N) \tag{6}$$

where

R_{An} is the aspect ratio of the n-th fragment;

$\overline{R_A}$ is the mean aspect ratio of fragments.

10 Precision

The precision of the test methods is not known because interlaboratory data are not available. When interlaboratory studies are conducted, a precision statement will be added at the following revision.

Table 1 gives the repeatability data from tests conducted at one laboratory with three materials. Four samples were sampled from a pile of each crushed CFRP, respectively. Each test result was the average of 100 individual measurements by method B.

Table 1 — Average and repeatability — Method B

Material	Parameter	Unit	Average	s_r	r
UD TYPE CFRP	L	mm	7,8	0,47	1,31
	W	mm	1,4	0,07	0,19
	R_A	-	7,4	0,66	1,83
s_r is the inter-sample standard deviation from the mean.					
r is the inter-sample repeatability limit (= 2,8 s_r; 95 % confidence limits).					

Table 1 *(continued)*

Material	Parameter	Unit	Average	s_r	r
FABRIC TYPE CFRP	L	mm	5,8	0,40	1,12
	W	mm	3,1	0,11	0,32
	R_A	-	2,1	0,07	0,18
MIXTURE CFRP	L	mm	6,0	0,99	2,76
	W	mm	1,9	0,33	0,94
	R_A	-	4,9	0,34	0,95
s_r is the inter-sample standard deviation from the mean.					
r is the inter-sample repeatability limit (= 2,8 s_r; 95 % confidence limits).					

Table 2 provides the results of a comparison study of the three test methods at one laboratory with three materials. One sample was sampled from a pile of each crushed CFRP. Each test was conducted with the same 100 fragments. Each result was the average of 100 individual measurements by method A, B and C.

Table 2 — Average and comparison of the three test methods

Material	Parameter	Unit	Average			Mean	s_m	CV
			Method A	Method B	Method C			
UD TYPE CFRP-1	L	mm	15,0	15,2	14,3	14,8	0,46	3,1 %
	W	mm	2,6	2,7	2,6	2,6	0,05	1,8 %
	R_A	-	7,9	8,3	7,5	7,9	0,40	5,0 %
UD TYPE CFRP-2	L	mm	12,2	12,3	12,0	12,2	0,16	1,3 %
	W	mm	5,8	5,8	5,8	5,8	0,03	0,5 %
	R_A	-	2,7	2,7	2,5	2,6	0,11	4,4 %
FABRIC TYPE CFRP	L	mm	6,8	6,6	7,0	6,8	0,21	3,1 %
	W	mm	4,1	4,0	4,4	4,2	0,18	4,3 %
	R_A	-	1,7	1,8	1,7	1,8	0,03	1,9 %
s_m is the inter-test method standard deviation from the mean.								
CV is the coefficient of variation.								

11 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 30012;

b) the method used;

c) all the details necessary for the complete identification of the crushed CFRP tested;

d) the average width $\overline{W}$ and the standard deviation $\sigma(W)$;

e) the average length $\overline{L}$ and the standard deviation $\sigma(L)$;

f) the average aspect ratio $\overline{R_A}$.

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