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Fibre-reinforced composites — Measurement of interfacial shear strength by means of a micromechanical single-fibre pull-out test

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Contents

Page

Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

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

Fibre-reinforced composites have become an indispensable part of modern high-tech applications due to the excellent tensile properties of the reinforcement fibres incorporated in the composite. This requires that the loads in a composite need to be distributed evenly to all fibres by means of a matrix. Therefore, a high interfacial shear strength is required for a good load transfer. Hence, the interfacial shear strength is one of the key parameters in composite technology.

To characterize the interfacial shear strength, composites with unidirectional aligned fibres are manufactured. A tensile test is then performed perpendicular to the fibre orientation (transverse tensile test), or a short beam shear strength test (as defined for instance in ISO 14130 or ASTM D2344) is performed to measure the apparent interlaminar shear strength (ILSS).

However, the maximum stress found in such macromechanical tests does not only depend on the fibre-matrix adhesion strength, it is also governed by the following additional factors: the fibre content, orientation, length, diameter and fibre distribution homogeneity, the pore void of the test specimens, and the mechanical properties of the fibre and the matrix. To achieve repeatable results for the fibre-matrix adhesion strength through macromechanical tests, it is necessary to keep a rigid control of the manufacturing process of the specimen, making the overall testing procedure laborious and difficult to compare across laboratories.

Micromechanical testing techniques have several advantages over the macromechanical methods. By involving only single fibres in the test, most dependencies on the manufacturing process of the test specimen listed above are avoided. Whereas macroscopic methods can only determine the apparent interfacial shear strength, the single-fibre pull-out test as a micromechanical test method also can determine the local interfacial shear strength, the interfacial frictional stress, and the critical interfacial energy release rate. Here, the local interfacial shear strength is essential for composites applications with cyclic load, since it characterizes to which stress a composite can be loaded before the interface between fibre and matrix is damaged. The critical interfacial energy release rate and the interfacial frictional stress have a strong impact on the energy absorption of composites, which is important in crash situations as for example in mobility applications.

This document describes stringent methods for specimen preparation, conditioning, and pull-out testing. Practical trials have shown that following these procedures leads to minimum variability in the test results.

Fibre-reinforced composites — Measurement of interfacial shear strength by means of a micromechanical single-fibre pull-out test

1 Scope

This document specifies a test method for determining the interfacial shear strength between a single fibre and a matrix by means of a pull-out test. The method can be used to measure the critical energy release rate.

The method is applicable to reinforcement fibres, such as carbon fibres, glass fibres, basalt fibres and similar stiff reinforcement fibres and to thermoset, thermoplastic and fine-grained concrete matrices. It can be used for polymeric reinforcement fibres and for other inorganic matrices.

It is not applicable to:

a)   elastomeric fibres and elastomeric matrices such as rubber;

b)   matrices which cure or melt at temperatures above 400 °C;

c)   matrices that show a strong tendency to bubble formation or expansion during the sample-preparation process;

d)   foams.

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 1973, *Textile fibres — Determination of linear density — Gravimetric method and vibroscope method*

ISO 2602, *Statistical interpretation of test results — Estimation of the mean — Confidence interval*

EN 12390-2, *Testing hardened concrete — Part 2: Making and curing specimens for strength tests*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/

3.1
constant-rate-of-extension testing machine
CRE testing machine
tensile testing machine provided with one specimen holder, which is stationary, and one clamp, which moves with a constant speed throughout the test, the entire testing system being virtually free from deflection

[SOURCE: ISO 5079:2020, 3.10, modified — "clamp" was replaced with "specimen holder".]

3.2
crucible

receptacle, made of aluminium or stainless steel, to hold a drop of matrix into which one single fibre is embedded

3.3
pull-out test

method of pulling out one single embedded fibre from a solidified drop of matrix, carried out with a *CRE testing machine* ([3.1](#))

3.4
force-displacement curve

graphical representation of the force recorded during the *pull-out test* ([3.3](#)) over the displacement of the moving clamp of the *CRE testing machine* ([3.1](#))

3.5
maximum force

highest force value taken from the *force-displacement curve* ([3.4](#)) appearing just before the complete debonding of the fibre from the solidified matrix during the pull-out

3.6
debonding force

force at which the fibre starts debonding from the solidified drop of matrix, derived from the *force-displacement curve* ([3.4](#))

3.7
interfacial frictional force

force measured after complete debonding, caused by only the friction between the fibre and the solidified matrix, taken from the *force-displacement curve* ([3.4](#))

3.8
embedding depth

selected depth to which the embedding device inserts the fibre into the matrix

3.9
embedded length

resulting length of the fibre in contact with the solidified matrix, corresponds to the displacement at which the fibre completely slips out of the solidified matrix, taken from the *force-displacement curve* ([3.4](#))

3.10
final forming

process, in which the matrix maximizes the wetting of the fibre and the meniscus reaches a steady state

3.11
solidification

process of transformation of the matrix from liquid to solid

Note 1 to entry: The term solidification includes both the commonly used solidification of thermoplastics by cooling as well as the curing of thermosets and concrete matrices.

3.12
fixing

first step of the *solidification* ([3.11](#)) of matrices, after which a test specimen can be removed from the embedding device without the risk that the fibre redirects inside the matrix

3.13
curing

process of *solidification* ([3.11](#)) of thermoset and concrete matrices

3.14
apparent interfacial shear strength

maximum force ([3.5](#)) normalized to the contact area between the fibre and the solidified matrix

3.15
interfacial frictional stress
interfacial frictional force (3.7) normalized to the contact area between the fibre and the solidified matrix

3.16
local interfacial shear strength
debonding force (3.6) related to the contact area of the interface between the fibre and the solidified matrix, without the impact of the friction between the fibre and the matrix

3.17
critical interfacial energy release rate
interfacial toughness
calculated as a function of the crack length (energy-based method), taking the deformation of the fibre and matrix during the pull-out into account

4 Principle

A single fibre is embedded into a drop of matrix at a defined temperature to form a test specimen for the pull-out test by means of an embedding station. If required, the embedding can take place in an inert gas atmosphere. Embedding depth and speed are variable. After embedding, the test specimen is solidified, for instance by heating up to a curing temperature (for example for thermoset matrices) or cooling down to a suitable temperature for matrix solidification (for example for thermoplastic matrices).

After complete solidification and conditioning, the test specimen is transferred into a CRE testing machine for pull-out testing.

During the pull-out test, certain values are measured along the force-displacement curve respectively calculated from these measured values.

5 Abbreviated terms, symbols and dimensions

5.1 Symbols

Table 1 lists symbols used throughout this document.

Table 1 — Test characteristics, symbols, and dimensions

Characteristic	Matrix	Symbol	Unit
Material parameter			
Fibre density	—	ρ	g/cm^3
Fibre linear density	—	ρ_l	dtex
Fibre diameter	—	d_f	µm
Fibre breaking force	—	f_B	cN
Melting temperature	TP	T_M	°C
Solidification temperature	TP	T_S	°C
Testing procedure			
Heating rate to embedding temperature	TS, TP	r_{TE}	K/min
Time at embedding temperature	TS, TP	t_{TE}	s
Embedding temperature	TS, TP	T_E	°C
Embedding depth	TS, TP, C	l_{ED}	µm
Embedding speed	TS, TP, C	v_E	mm/min
Forming time	TS, TP, C	t_F	s

Table 1 *(continued)*

Characteristic	Matrix	Symbol	Unit
Curing temperature	TS, C	T_C	°C
Heating rate to curing temperature	TS, C	r_{TC}	K/min
Time at curing temperature	TS, C	t_{TC}	s
Below-solidification-temperature	TP	T_{BS}	°C
Cooling rate to below-solidification-temperature	TP	r_{TBS}	K/min
Final cooling rate	TS, TP	r_{TF}	K/min
Final withdrawal temperature	TS, TP	T_F	°C
Testing speed for pull-out test	All	V_P	mm/min
Evaluation			
Maximum force	All	F_{max}	cN
Debonding force	All	F_d	cN
Interfacial frictional force	All	F_b	cN
Embedded length	All	l_e	µm
Apparent interfacial shear strength	All	τ_{app}	MPa
Interfacial frictional stress	All	τ_f	MPa
Local interfacial shear strength	All	τ_d	MPa
Alternative local interfacial shear strength	All	$\tau_{d,alt}$	MPa
Shear-lag parameter	All	β	—
Residual thermal stresses	All	τ_T	MPa
Critical energy release rate	All	G_{ic}	J/m^2

5.2 Abbreviated terms

Table 2 lists abbreviated terms used throughout this document.

Table 2 — Materials and abbreviated terms

Material	Abbreviation
Fibres	
Carbon fibre	CF
Recycled carbon fibre	rCF
Glass fibre	GF
Natural fibre	NF
Matrices	
Thermoset	TS
Thermoplastic	TP
Concrete	Concrete
Polypropylene	PP
Polyamide 6	PA6
Polyether ether ketone	PEEK
Polyether block amide	PEBA
Polyurethane	PU

Table 2 *(continued)*

Material	Abbreviation
Polycarbonate	PC
Epoxy	EP
Vinylester	VE

6 Apparatus

6.1 Fibre diameter determination

6.1.1 General

The diameter of each individual fibre shall be determined close to the position where the fibre is embedded into the matrix. The following methods may be used for the determination of the individual fibre diameter.

NOTE   For the sake of simplicity, all calculations within this document assume a circular fibre cross section.

6.1.2 Vibroscopic fibre linear density and diameter test

This method uses a device measuring the linear density according to the vibroscopic principle, which shall be in accordance with ISO 1973. Here, a fibre with a known pretension is excited to its fundamental resonance frequency at a known vibrating length.

For glass fibres, due to a possible impact of the bending stiffness to the resonance curve, a vibrating length of at least 50 mm and a pretension of 3 cN/tex are strongly recommended. For carbon fibres, a vibrating length of minimum 25 mm and a pretension of 1 cN/tex are commonly used.

Based on the selected pretension and vibrating length and on the measured resonance frequency, the linear density ρ_l is calculated following the vibrating string equation. Employing the known fibre density ρ (specified by the fibre producer), the fibre diameter d_f is calculated according to Formula (1):

$$d_f = 10 \cdot \sqrt{\frac{4 \cdot \rho_l}{\pi \cdot \rho}}$$ (1)

where

d_f is the fibre diameter, in micrometres (μm);

ρ_l is the fibre linear density, in decitex (dtex, g/1 000 m);

ρ is the fibre density, in gram per square centimetres (g/cm[3]).

The determination of the fibre diameter is required to calculate the contact area between the fibre and the matrix.

6.1.3 Optical fibre diameter determination

This method uses an optical assembly to measure the individual fibre diameter directly. Possible principles are a laser-scan micrometre, a laser diffractometer, an optical microscope, or an appropriate device according to ISO 11567.

6.2 Embedding station

This method uses a device for producing test specimens for the pull-out test, by embedding a single fibre into a drop of matrix placed in a crucible. A single fibre with a minimum length of 5 mm is placed into a cannula, after insertion the fibre protrudes out of the cannula. The protruding end is embedded into the

matrix, embedding depth and speed are variable. During embedding and solidification, the fibre can be held with low tension in the cannula by means of a holding air flow such that it can follow a possible shrinkage of the matrix.

The device is equipped with a heating system for the crucible, allowing temperature changes at rates of 0,1 K/min to 99,9 K/min up to a maximum temperature of 400 °C. The heat transfer is given by the contact between the heating system and the bottom side of the crucible. The actual temperature at the top of the matrix might be lower than the displayed actual heater temperature, due to the geometry of the setup. Temperature, temperature rates and duration of heating (time at temperature) can be adjusted.

The device is equipped with an active cooling system which can be enabled, and which rate can be adjusted. The maximum cooling shall allow the device to cool down from 400 °C to 40 °C within 6,5 min (from 100 °C to 40 °C within 3,5 min).

The device is equipped with a triaxial linear table to allow positioning of the fibre relative to the matrix, and a microscope to observe and control the positioning. The fibre can be moved along its axis for embedding using an additional z-axis of the embedding device.

The device is equipped with a system to flush the embedding zone with an inert gas to avoid oxidation of the matrix.

6.3 CRE testing machine

This method uses a device for pull-out testing, equipped with an upper holder for the test specimen mounted to the force-measuring system, and a moveable lower draw-off clamp to pull the single fibre out of the matrix.

The device shall be equipped with a high-precision load cell with a minimum force range higher than the fibre breaking force to be tested and a resolution of 1 μN or better. The distance between the draw-off clamp and the upper specimen holder can be freely adjusted, the testing speed can be adjusted to values between 0,1 mm/min to 100 mm/min.

The draw-off clamp consists of one stationary and one moveable clamp jaw with adjustable clamping pressure. The surface of the clamp jaws in contact with the specimen shall be made of a material to provide correct grip without damaging the fibre, thereby avoiding slippage and jaw breaks.

A high-precision displacement measurement sensor with a resolution of 0,1 μm or better is required. The device is resistant against deformation under load, with a maximum deflection of 0,02 mm/N.

The lateral position of the draw-off clamp is adjustable to match the position of the fibre axis. In this way, the fibre is clamped at minimum shear force even at very low distances between matrix and clamp. Adjustment is carried out by means of a camera with microscopic optics.

During the pull-out test, the displacement of the draw-off clamp and the resulting force is recorded as a force-displacement curve.

7 Test specimen

The test specimen consists of a rotationally symmetric metallic crucible, a drop of a matrix placed into the upper recess of the crucible, and a fibre embedded into the matrix. Figure 1 shows the physical dimensions of the crucible that shall be used for the preparation of the test specimen.

Dimensions in millimetres
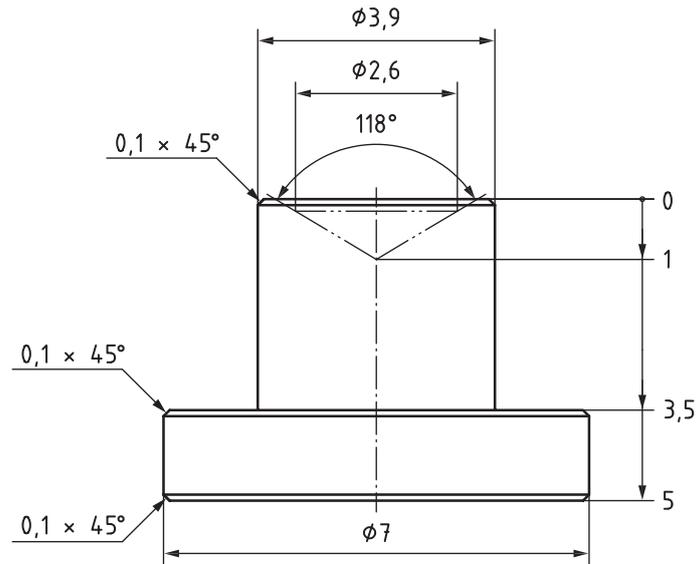
Figure 1 — Test specimen, crucible

Figure 2 shows a schematic lateral section through the test specimen.

Key
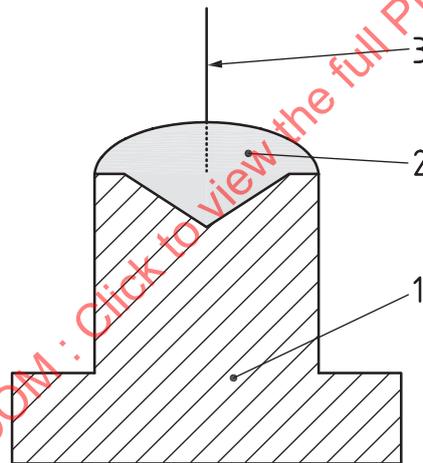
1   crucible with recess
2   matrix drop
3   embedded fibre (dashed line = embedded section)

Figure 2 — Test specimen, lateral section

An appropriate embedding device to create the test specimen is described in 6.2.

After the test specimen has been produced and solidified, it is transferred to the CRE testing machine. Figure 3 shows schematic lateral sections through the test specimen, mounted into the upper test specimen holder and the lower draw-off clamp of the CRE testing machine, described in 6.3.
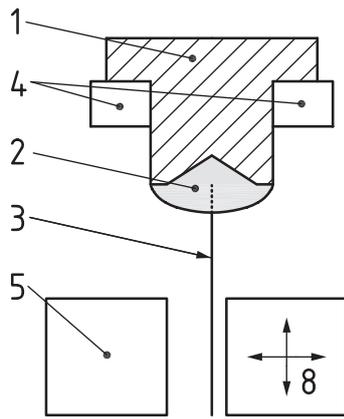
a) Draw-off clamp adjustment **b) Draw-off clamp open** **c) Draw-off clamp closed**
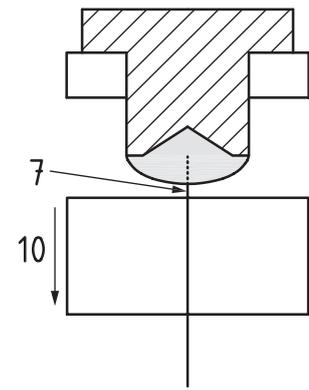
Key

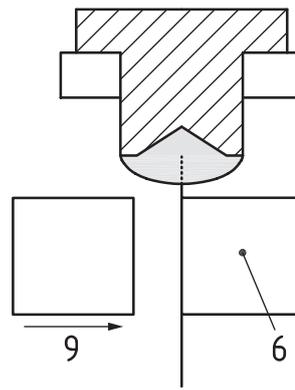|				
1	crucible with recess		6	stationary jaw of draw-off clamp
2	matrix drop		7	free fibre length
3	embedded fibre (dashed line = embedded section)		8	lateral + vertical adjustment
4	test specimen holder = measuring clamp		9	closing direction of movable clamp jaw
5	movable jaw of draw-off clamp		10	draw-off direction

Figure 3 — Test specimen in CRE testing machine, lateral section

8 Procedure

8.1 Overview

Fibre embedding and matrix solidification are carried out using complex temperature profiles, described in 8.2 to 8.10. Figure 4 and Figure 5 schematically illustrate those temperature profiles for thermoset respectively thermoplastic matrices.

Key
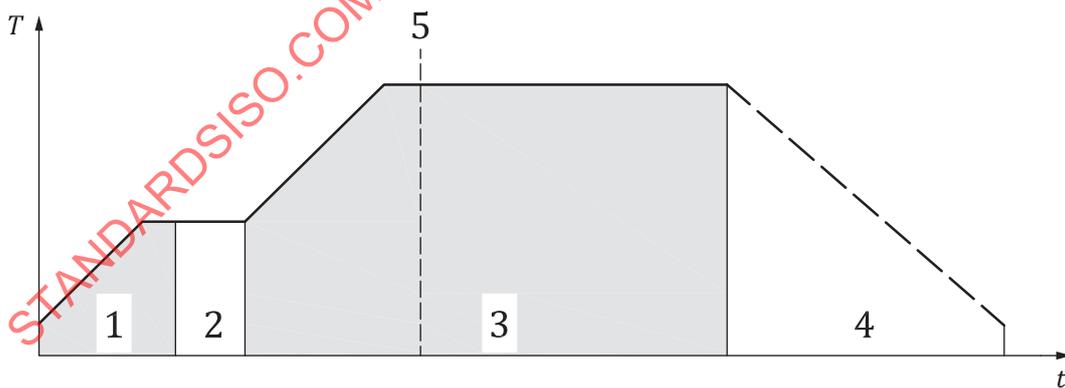
T temperature

t time

1 heating to and holding embedding temperature for degassing (heating rate to embedding temperature, time at embedding temperature, embedding temperature)

2 positioning, embedding and final forming (embedding temperature, forming time)

3   curing (heating rate to curing temperature, time at curing temperature, curing temperature; divided into several steps, if required)

4   cooling (if inside the embedding device: final cooling rate, final withdrawal temperature)

5   optional test specimen extraction from the embedding device after fixing

Figure 4 — Typical temperature profile for thermoset matrices

Key

T temperature

t time

1   heating to and holding of the embedding temperature (heating rate to embedding temperature, time at embedding temperature, embedding temperature)
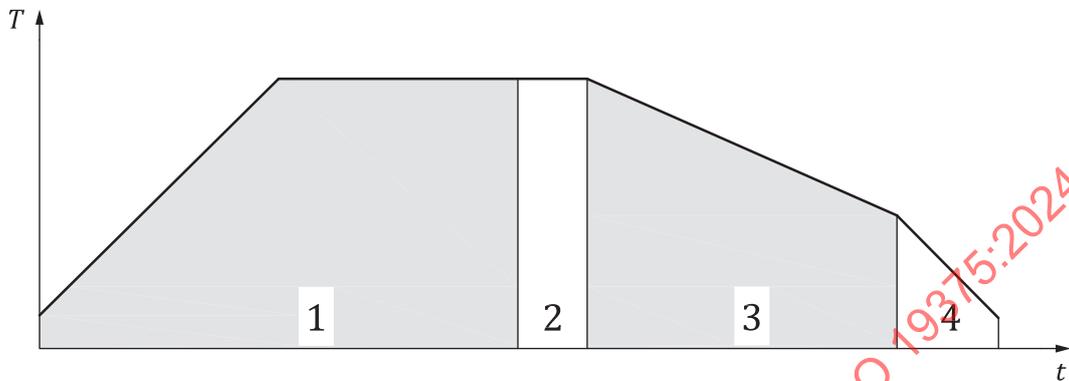
2   positioning, embedding and final forming (embedding temperature, forming time)

3   cooling down below-solidification-temperature (cooling rate to below-solidification-temperature, below-solidification-temperature)

4   final cooling (final cooling rate, final withdrawal temperature)

Figure 5 — Typical temperature profile for thermoplastic matrices

After solidification and conditioning, the test specimen is transferred to the CRE testing machine for pull-out testing.

8.2   General requirements

A glove shall be used during all following steps to avoid any influence from contamination.

The embedding depth for a test series can be determined by iterative pre-tests, where the maximum pull-out force is investigated as a function of the embedding depth, following Clause 8. The embedding depth shall be chosen such that the maximum pull-out force is close to but does not exceed the fibre breaking force (for example measured as described in ISO 5079 or ISO 11566). The resulting embedded length (refer to 9.2.2) shall be at least 4 times the fibre diameter.

In case the embedded length is much smaller than the embedding depth, this can be an indicator for irregular wetting, and the processing parameters should be reconsidered.

For a comparative test of similar fibre-matrix combinations, the same embedding depth shall be chosen for test specimen preparation and, in case one of the further evaluations in 9.3 is applied, the embedded length shall be similar.

A sufficient number of test specimen shall be prepared to ensure solid average values of the evaluated parameters. After testing, invalid tests shall be eliminated, assuring that the sample size is not below 15 valid pull-out tests, unless otherwise agreed-on between the interested parties. For more information on invalid results, please refer to 8.10 and Annex B.

8.3 Fibre sampling and preparation

8.3.1 General

Fibres are drawn from a fibre sample. Typical sample forms are rovings, fabrics, flocks (for example natural fibres) and chopped fibres (including recycled fibres).

Special care shall be taken to assure that a representative set of fibres is drawn from different sections of the sample, for example by selecting different filaments from a roving or selecting fibres from different roving sections. Care shall be taken not to damage the fibres by stretching or bending. The minimum fibre length shall be sufficient to assure a fibre diameter determination (see 6.1).

8.3.2 Fibre diameter determination

The diameter of each individual fibre test specimen shall be determined as described in 6.1. Diameter determination shall be carried out on the fibre section which is embedded into the matrix.

8.3.3 Fibre insertion into the embedding device

The fibre is inserted into the cannula of the embedding device. The fibre shall be cut by means of scissors to protrude by a length of 2 mm to 5 mm from the cannula (preferred length: 2 mm to 3 mm). The fibre is held in place by means of the holding air.

8.4 Matrix preparation

8.4.1 Thermoset matrices

The matrix shall be prepared in accordance with the manufacturer's instructions, for example regarding the mixing ratio, vacuum/evacuation, the mixing procedure (manual mixing, magnetic stirring) and the processing time. A bubble-free matrix is required, for example by vacuum application. Any deviation from the manufacturer's instruction shall be indicated in the test report.

8.4.2 Thermoplastic matrices

The matrix shall be prepared in accordance with the manufacturer's instructions, for example regarding the required drying, to achieve a defined, not degraded condition. Any deviation from the manufacturer's instruction shall be indicated in the test report.

8.4.3 Concrete matrices

The matrix shall be prepared in accordance with the manufacturer's instructions, for example regarding the required mixing ratio (for example cement, water, filler and further additives). Any deviation from the manufacturer's instruction, for example to adjust the viscosity, shall be indicated in the test report.

8.5 Preparation of matrix droplet

8.5.1 General

A crucible is placed into the embedding device described in 6.2. In general, an aluminium crucible is recommended for most thermoset and thermoplastic matrices, unless a chemical reaction between the crucible and the matrix occurs during the processing. In these cases, a stainless-steel crucible is recommended, for example for concrete or polyurethane matrices. For matrix materials that tend to detach from the crucible surface during the pull-out test, the surface of the crucible can be roughened/scratched by an appropriate tool.

After the placement of the crucible, the matrix is placed into the upper crucible recess. The matrix mass shall be chosen in such a way that during embedding, the drop forms a convex shape above the crucible top without flowing over the crucible edge. Preferably, the matrix covers the whole crucible diameter.

If the matrix is prone to oxidization, the preparation zone of the embedding device shall be flushed for a sufficient period of time with an inert gas as soon as the matrix has been placed in the crucible and held under inert gas atmosphere in the following preparation steps and until the test specimen is removed from the embedding device.

8.5.2 Thermoset matrices

A pipette is used to place a drop of matrix in the upper recess of the crucible. Typically, the embedding temperature is the laboratory temperature. A higher embedding temperature can be chosen to enable degassing. In this case, the embedding temperature and time at embedding temperature shall be adjusted to remove bubbles from the matrix without substantial curing, still enabling the subsequent embedding of the fibre.

A typical temperature profile is shown in Figure 4.

The matrix preparation temperature profile consists of heating to and holding the embedding temperature for degassing (heating rate to embedding temperature, time at embedding temperature, embedding temperature). It may include an intermediate temperature step.

8.5.3 Thermoplastic matrices

In case of solid matrices, an appropriate size of pellet shall be placed into the upper crucible recess. In the case of a matrix in form of a powder or fibre snippets, an appropriate amount shall be applied in compacted form, for example by means of a suitable pressing tool. The crucible shall then be heated inside the embedding device to the embedding temperature, so that a liquid droplet forms.

The temperature profile shall be adjusted to completely melt the matrix without degradation. Referring to the test specimen geometry shown in Figure 2, it shall be considered that the actual temperature at the top of the matrix may be lower than the displayed actual heater temperature.

A typical temperature profile is shown in Figure 5.

The matrix preparation temperature profile consists of heating to and holding of the embedding temperature (heating rate to embedding temperature, time at embedding temperature, embedding temperature).

8.5.4 Concrete matrices

A spatula is used to place a drop of matrix into the upper recess of the crucible. The crucible shall be made of stainless steel.

Typically, the complete matrix preparation takes place in a laboratory atmosphere.

8.6 Fibre positioning, embedding and final forming

This step consists of positioning, embedding and final forming, as illustrated in Figure 4 and Figure 5. During this step, the embedding temperature shall be held at a constant level.

Before embedding, the matrix shall have a stable geometry.

For embedding, the fibre end protruding from the cannula shall be positioned close to the top of the matrix using the z-axis of the embedding device (6.2). By means of the triaxial linear table and the observing microscope, the fibre is positioned above the centre of the droplet pointing in vertical direction, perpendicular to the top of the matrix surface. Next, the fibre is moved vertically until it just touches the matrix surface.

Next, the fibre shall be embedded into the matrix to the predetermined embedding depth that is held constant for the full test series as described in 8.2. The embedding speed shall be chosen such that the fibre does not deform or move out of the vertical position during embedding.

NOTE 1   The embedding is usually achieved in 20 s to 40 s, for matrices requiring a high embedding length or showing a low wettability, it can take up to 60 s.

After complete embedding, a forming time of 0 s to 60 s shall be maintained to allow wetting and meniscus formation of the matrix droplet.

NOTE 2   A missing meniscus or indented matrix instead of a protruding meniscus can be a reason for larger differences between the embedding depth and embedded length. In case of thermoplastic matrices, this can be caused by inhomogeneous melting. To ensure homogeneous melting, a higher embedding temperature, a reduced matrix mass, a longer time at embedding temperature, or a longer forming time can be chosen. Additionally, the inert gas flow can be suspended temporarily to avoid cooling of the matrix surface.

Annex A shows typical images of both, valid and invalid embedding processes.

8.7   Solidification of the test specimen

8.7.1   General

After embedding the fibre into the matrix, the test specimen shall completely solidify following the manufacturer's instructions before the test procedure is continued.

The complete solidification can take place inside the embedding device.

To reduce the cycle time in case of a long solidification process, for instance for long curing thermoset matrices, the solidification can be divided into two steps. The first step is referred to as fixing and takes place inside the embedding device, after which the test specimen is sufficiently fixed to the matrix such that it can be taken out of the embedding device without changing the geometry of the test specimen. As a second step, the final solidification shall then be done externally (for example, in a lab oven).

8.7.2   Thermoset matrices

According to the manufacturer's instructions, a suitable temperature profile shall be selected to assure full curing of the test specimen. As illustrated in Figure 4, the solidification temperature profile consists of

— curing (heating rate to curing temperature, time at curing temperature, curing temperature; divided in several steps, if required), and

— cooling (final cooling rate, final withdrawal temperature).

If the test specimen is taken out of the embedding device after fixing, curing shall be continued in an external oven. In this case, the subsequent cooling is undefined.

8.7.3   Thermoplastic matrices

The temperature profile shall be selected in such a way that full solidification of the test specimen is assured. As illustrated in Figure 5 the solidification temperature profile consists of

— cooling down below solidification temperature (cooling rate to below-solidification-temperature, below-solidification-temperature) and

— final cooling (final cooling rate, final withdrawal temperature).

The cooling rate to below-solidification-temperature can have an impact on the fibre-matrix adhesion strength. Hence, the cooling rate should be selected in accordance with the process conditions in composite production.

NOTE   The cooling can be done using an intermediate holding step between the melting temperature and the glass transition temperature.

Thermoplastic matrices tend to shrink during cooling down to below-solidification-temperature. Therefore, the holding air flow shall be switched off during this step to allow the fibre to follow the matrix shrinkage.

High cooling rates can lead to a slant fibre position due to matrix shrinkage. In these cases, a lower cooling rate is preferred until the matrix has been cooled down to below-solidification-temperature.

The final cooling to the final withdrawal temperature can be performed at a higher rate.

8.7.4 Concrete matrices

A fixing step shall be performed keeping the test specimen inside the embedding device at laboratory temperature (for example for 15 min).

For final solidification, the test specimen shall then be cured for a certain time at certain climate conditions outside of the embedding device, in accordance with EN 12390-2.

8.8 Test specimen requirements and final validation

All test specimens shall pass a final validation before being accepted for a pull-out test, to assure that

a) the protruding fibre has sufficient length so that it can be gripped securely by the draw-off clamp of the CRE testing machine (6.3) during the pull-out test;

b) the matrix drop forms a convex shape without flowing over the crucible edge;

c) the fibre protrudes from the top of the matrix droplet;

d) the fibre protrudes perpendicular from the matrix surface (as well as it can be achieved, see Note);

e) the embedding region of the matrix is be free of bubbles;

f) the matrix volume is not dominated by bubbles.

Inhomogeneous shrinkage or too fast cooling/curing of the matrix can cause an exceedingly slant protruding angle. In such cases, the test specimen may be discarded.

NOTE   The protruding angle of the fibre from the matrix top has been analysed; it has been found that slant angles caused by the mechanism of the embedding station only have a minor effect.

8.9 Post-preparation and conditioning

8.9.1 General

The post-preparation and conditioning shall be performed according to the matrix manufacturer's instructions and shall be consistent for all test specimen within a test series.

8.9.2 Thermoset matrices

After solidification, each test specimen shall be stored inside a desiccator for a specified storing/conditioning time interval of minimum 12 h (for example, all pull-out tests performed within 12 h to 24 h after embedding). For the comparison of different test series, the storing/conditioning time shall be consistent.

Contaminations or remaining matrix deposits on the fibre surface can cause slippage inside the draw-off clamp during the pull-out test. In this case, the test specimens may be cleaned before the pull-out test, for example by storing them inside an oven for 1 h at a temperature of 80 °C. After such a temperature treatment, the test specimens shall be cooled down to laboratory temperature inside a desiccator for at least 30 min.

8.9.3 Thermoplastic matrices

After solidification, the test specimens may be tempered for a specified time at a specified temperature to release residual stresses in the matrix droplet.

Next, each test specimen shall be stored inside a desiccator for a specified storing/conditioning time interval of at least 12 h (for example all pull-out tests performed within 12 h to 24 h after embedding). For comparison of different test series, the storing/conditioning time shall be consistent.

For certain fibre-matrix combinations, the adhesion can change over time (for example due to moisture uptake, change in residual stresses and aging). Therefore, it is preferable to define a narrow time interval for testing. Tempering can help to avoid changes in fibre-matrix adhesion over time.

8.9.4 Concrete matrices

Before continuing with the pull-out test, the test specimen shall be conditioned as specified by the manufacturer's instruction or as agreed on between the interested parties, for example in accordance with EN 12390-2.

8.10 Pull-out test

8.10.1 General

The pull-out test is carried out by means of a CRE testing machine, as specified in 6.3.

A test specimen is placed into the CRE testing machine. The fibre is pulled out of the matrix while the force-displacement curve is recorded. An ideal force-displacement curve is shown in Figure 6.
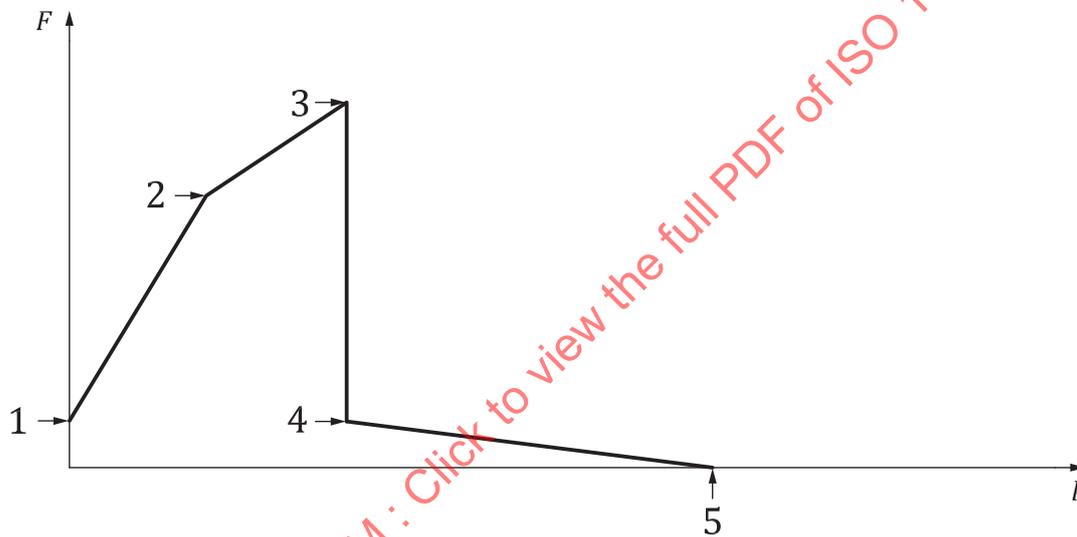
Key

l	displacement	5	embedded length l_e
F	force	1-2	elastic deformation
1	pretension	2-3	crack propagation
2	debonding force F_d	3-4	complete fibre debonding
3	maximum force F_{max}	4-5	frictional-interaction-only
4	friction force F_b		

Figure 6 — Ideal force-displacement curve

For a detailed description of the pull-out test, see Reference [11].

8.10.2 Test specimen insertion and clamping

To perform the pull-out test, the crucible of the test specimen is fixed in the specimen holder (measuring clamp) with the protruding fibre pointing downwards [refer to Figure 3 a)].

The opened draw-off clamp shall be positioned such that the pultruding fibre touches the stationary clamp jaw along the complete height, without bending the fibre. This positioning is achieved by using the lateral position adjustment of the draw-off clamp [refer to Figure 3 a) and b)].

The vertical position of the draw-off clamp shall be adjusted such that the stationary clamp is very close to the matrix surface. The resulting short free fibre length between clamp and matrix helps to reduce the influence of the stretching of the fibre during the pull-out test. At the same time, care shall be taken to prevent clamping of the possibly formed meniscus (typically reaching along the fibre up to 3 times the fibre diameter). Hence, the preferable distance between the matrix surface and the draw-off clamp ranges from 5 times to 15 times the fibre diameter [refer to Figure 3 a) and b)].

Subsequently, the moveable jaw of the draw-off clamp shall be closed. Care shall be taken that the clamp jaw does not damage the fibre when closing [refer to Figure 3 b) and c)]. The clamping pressure shall be adjusted in such a way that slippage as well as damage of the fibre is avoided.

8.10.3 Testing conditions

The pull-out test shall start at a pretension of 0,01 cN, applied automatically by the CRE testing machine. The displacement speed of the draw-off clamp shall be 0,1 mm/min. Higher speeds are permitted upon mutual agreement, for example in case of high embedding depths.

8.10.4 Testing

The fibre shall be pulled out from the matrix, while the position of the draw-off clamp and the resulting force shall be recorded resulting in a force-displacement curve (refer to Figure 6). The test shall be finished when the force drops to values constantly below 0,01 cN in the frictional-interaction-only part of the force-displacement curve (representing a full pull-out of the fibre from the matrix).

8.10.5 Validation

A final validation shall be performed by inspecting the recorded force-displacement curve and the test specimen. Annex B shows typical force-displacement curves of both, valid and invalid tests. A large range of pull-out curves for various fibre-matrix combinations can be found in Annex C, Annex D and Annex E.

Typical causes for invalid tests are:

— fibre breakage between matrix and draw-off clamp: the frictional-interaction-only part is missing;

— slippage of the fibre in the draw-off clamp: force remains at a high level and does not drop to 0;

— fibre breakage in the draw-off clamp: fibre is pulled out from between the draw-off clamp jaws typically resulting in a too-long apparent frictional-interaction-only part, fibre still protrudes from the matrix after the test;

— irregular wetting: unusual short friction-interaction-only part, embedded length is much smaller than the embedding depth;

— matrix debonds from the crucible: irregular force-displacement curve, test specimen shows crack between the matrix drop and the crucible surface.

Invalid tests shall be excluded from the evaluation and the test report.

9 Evaluation

9.1 General

The basic evaluation described in 9.2 resulting in the apparent interfacial shear strength is required to complete a valid test. If desired and depending on the shape of the force-displacement curves, more detailed analyses can be done, as described in 9.3.

The following evaluations require the identification of certain points (keys 2 to 5 in Figure 6) in the recorded force-displacement curves. This can be performed either manually by visual inspection or automatically, for example by fitting the ideal force-displacement curve illustrated in Figure 6 partially or in total to the data.

9.2 Measured values and basic evaluation

9.2.1 Fibre diameter

The individual fibre diameter d_f, measured as described in 8.3.2, is required for all further calculations.

9.2.2 Embedded length

The embedded length l_e, taken from the force-displacement curve as illustrated in 8.10, is required for all further calculations. It is derived from the displacement at the point where the force drops below 0,01 cN at the end of the frictional-interaction-only part of the curve (see Figure 6). Note that l_e can differ from the embedding depth l_{ED} chosen for the embedding of the sample.

9.2.3 Maximum force and apparent interfacial shear strength

The maximum force F_{max}, taken from the force-displacement curve as illustrated in 8.10, is required for the calculation of the apparent interfacial shear strength.

The apparent interfacial shear strength τ_{app} is the maximum force per unit contact area. It shall be calculated according to Formula (2) from F_{max} and the contact area between the fibre and the matrix. The contact area shall be calculated as the product of l_e and the fibre circumference $\pi \cdot d_f$:

$$\tau_{app} = \frac{F_{max}}{\pi \cdot d_f \cdot l_e} \qquad (2)$$

where

F_{max} is the maximum force, in centinewton (cN);

d_f is the fibre diameter, in micrometres (μm);

l_e is the embedded length, in micrometres (μm).

9.2.4 Interfacial frictional force and stress

The interfacial frictional force F_b, taken from the force-displacement curve as illustrated in 8.10, is required for the calculation of the interfacial frictional stress.

The interfacial frictional stress τ_f shall be calculated according to Formula (3).

$$\tau_f = \frac{F_b}{\pi \cdot d_f \cdot l_e} \qquad (3)$$

where

F_b is the interfacial frictional force, in centinewton (cN);

d_f is the fibre diameter, in micrometres (μm);

l_e is the embedded length, in micrometres (μm).

9.3 Further evaluations

9.3.1 General

Depending on the shape of the force-displacement curve, further evaluations can be applied to the data. These evaluations include stress-based and energy-based analyses. When modulating the embedded length l_e, the measured apparent interfacial shear strength τ_{app} values obtained by the method described above can be evaluated against the different l_e values, e.g., to analyse the decreasing τ_{app} at increasing l_e, enabling further analysis for instance with shear-lag models[8]. A review over different analysis methods is given for instance in References [9], [10] and [11]. The applied analysis method for further evaluation shall be agreed on by the interested parties. Three proposals are given in 9.3.2 to 9.3.4 which require certain additional material parameters, specified in Annex F.

9.3.2 Local interfacial shear strength derived from the debonding force

If the debonding force F_d can be identified in the force-displacement curve (kink at F_d, see Figure 6), the local interfacial shear strength τ_d may be calculated according to Formula (4).

$$\tau_d = \frac{F_d \cdot \beta}{\pi \cdot d_f} \cdot \coth(\beta \cdot l_e) \cdot \tau_T \cdot \tanh\left(\frac{\beta \cdot l_e}{2}\right) \tag{4}$$

For further background to Formula (4), see References [9] and [10].

9.3.3 Alternative determination of local interfacial shear strength

Alternatively, the local interfacial shear strength may be calculated using the alternative method described in Reference [9], e.g. if no or two kinks can be identified in the force-displacement curve. The alternative local interfacial shear strength $\tau_{d,alt}$ does not require the identification of F_d.

9.3.4 Critical energy release rate

The debonding process can also be analysed using the critical energy release rate G_{ic}, as described in Reference [12].

10 Expression of results

The test results of a series shall be calculated in accordance with ISO 2602:

a) the mean values, expressed to 3 significant digits;

b) the standard deviation, expressed to 3 significant digits;

c) the coefficient of variation, expressed as a percentage, to the nearest 0,1 %;

d) the 95 % confidence intervals, expressed to 3 significant digits.

Precision data for typical fibre-matrix combinations can be found in Annex G.

11 Test report

11.1 General

The test report shall include at least the following information:

a) a reference to this document, including year and month of publication, i.e. ISO 19375:2024;

b) the date of test;

c) operators for test specimen preparation and for testing;

d) any deviations from this document;

e) any unusual features observed;

f) fibre type (for example carbon, glass), fibre sample form (for example roving, fabrics, flocks and chopped fibres);

g) fibre specification (for example producer, production date and fibre breaking force, if known);

h) matrix type, matrix form (for example pellet, powder and liquid);

i) matrix specification (for example producer and production date, if known).

11.2 Fibre and matrix preparation

11.2.1 Fibre sampling and preparation

a) process for selection of single fibres from the sample;

b) method used to determine the fibre diameter of the single fibres;

c) fibre density, pretension and vibrating length, if the vibroscopic method is used to determine the fibre diameter.

11.2.2 Matrix preparation

a) for thermoset materials — matrix preparation parameters: for example mixing ratio, mixing method, vacuum applied, mixing procedure, processing time;

b) for thermoplastic materials — matrix preparation parameters: for example drying step;

c) for concrete materials — matrix preparation parameters: for example mixing ratio;

d) any deviation from the matrix manufacturer's instruction during matrix preparation.

11.3 Test specimen preparation

11.3.1 Embedding device

a) manufacturer, type, serial number.

11.3.2 Matrix droplet preparation

a) crucible material, surface roughening, if applied;

b) use and type of inert gas used during matrix preparation and fibre embedding, if applied;

c) thermoset matrices: heating rate to embedding temperature, time at embedding temperature, embedding temperature;

d) thermoplastic matrices: heating rate to embedding temperature, time at embedding temperature, embedding temperature.

11.3.3 Fibre embedding

a) embedding depth;

b) embedding speed;

c)   forming time after fibre embedding.

11.3.4   Test specimen solidification

a)   use and type of inert gas during fixing, if applied;

b)   thermoset matrices:

—   curing: heating rates to curing temperatures, time at curing temperature, curing temperatures; divided into several steps, if required;

—   fixing time: external curing steps after fixing (temperature, time), if applied;

—   final cooling: final cooling rate, final withdrawal temperature, if inside the embedding device;

c)   thermoplastic matrices:

—   cooling down to below-solidification-temperature: cooling rate to below-solidification-temperature, below-solidification-temperature;

—   final cooling: final cooling rate, final withdrawal temperature;

d)   concrete matrices:

—   fixing time;

—   external final solidification: time, climate conditions;

—   standard method for final solidification (for example reference to EN 12390-2);

e)   any deviation from the matrix manufacturer's instructions during solidification.

11.3.5   Post-preparation and conditioning

a)   thermoset matrices:

—   storage time in desiccator after solidification;

—   cleaning temperature, cleaning time and minimum cooling time after cleaning, if applied;

b)   thermoplastic matrices:

—   tempering temperature and time, if applied;

—   storage time interval in desiccator after solidification;

c)   concrete matrices:

—   conditioning parameters;

—   reference to standard method for conditioning, if applied (for example EN 12390-2);

d)   any deviation from the matrix manufacturer's instructions during post-preparation and conditioning.

11.4   Pull-out test and evaluation

11.4.1   CRE testing machine

a)   manufacturer, type, serial number;

b)   testing speed for pull-out test.

11.4.2 Material parameters for evaluation

a) specific fibre parameters required for the further evaluations, if applied (refer to Annex F);

b) specific matrix parameters for the further evaluations, if applied (refer to Annex F).

11.4.3 Basic evaluation and results

a) individual force-displacement curves of all test specimens;

b) measurement results for each test specimen:

— fibre diameter;

— embedded length;

— maximum force;

— apparent interfacial shear strength;

— interfacial frictional force;

— frictional stress.

11.4.4 Evaluation of the local interfacial shear strength from the debonding force, if applied

a) evaluation method, applied bibliographic reference;

b) measurement results for each test specimen:

— debonding force;

— local interfacial shear strength.

11.4.5 Alternative evaluation of the local interfacial shear strength, if applied

a) evaluation method, applied bibliographic reference;

b) measurement results for each test specimen/test series: local interfacial shear strength.

11.4.6 Evaluation of the critical energy release rate, if applied

a) evaluation method, applied bibliographic reference;

b) measurement results for each test specimen: critical energy release rate.

11.5 Statistics

a) number of valid tests;

b) result statistics for all chosen result parameters, according to Clause 10.

Annex A
(informative)

Images of embedding procedure

A.1 Exemplary images of a correct embedding procedure

Exemplary images of a correct embedding procedure are shown in Figure A.1 to Figure A.4.
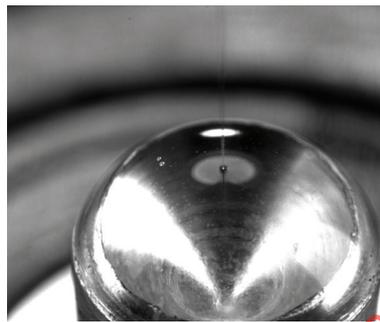
a) Matrix preparation   b) Positioning/Embedding   c) Curing
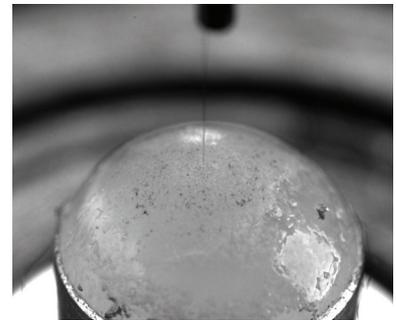
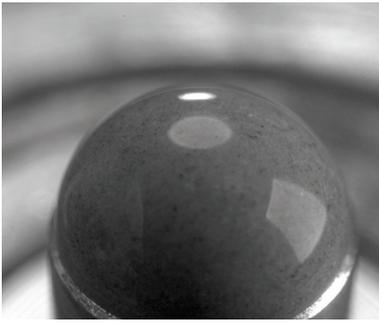**Figure A.1 — GF/Two-component EP with amine hardener**

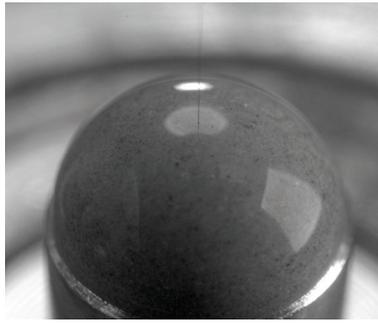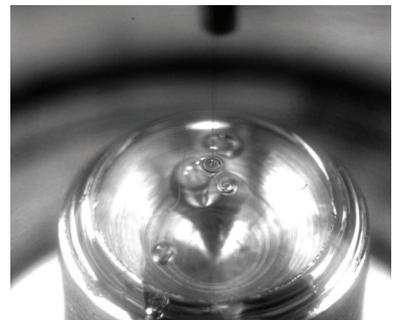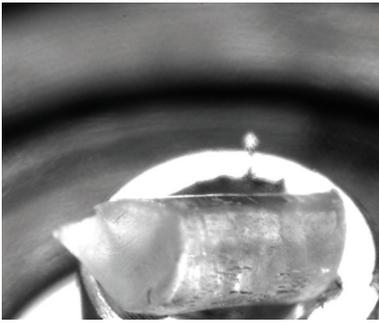a) Matrix preparation   b) Positioning/Embedding   c) Cooling

Figure A.2 — GF/PP

a) Matrix preparation   b) Positioning/Embedding   c) Fixing

Figure A.3 — CF/Concrete

a) Matrix preparation   b) Positioning/Embedding   c) Cooling

Figure A.4 — Natural fibre (Sisal)/PP

A.2   Exemplary images of invalid embedding procedures

Exemplary images of an invalid embedding procedures are shown in Figure A.5 to Figure A.9.
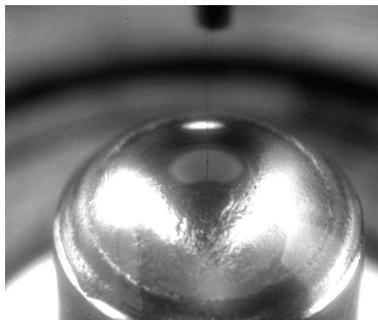
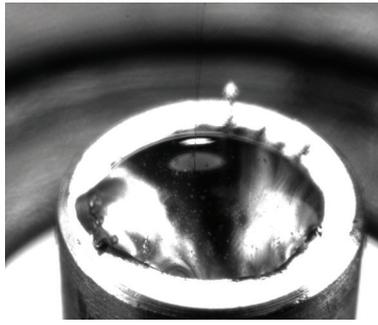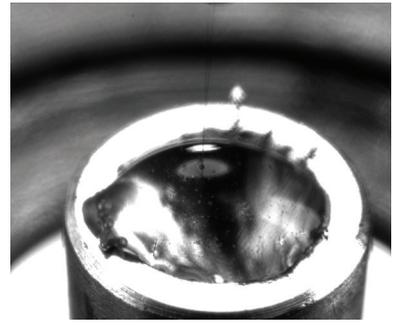a) Matrix preparation   b) Positioning/Embedding   c) Curing

Figure A.5 — Development of bubbles while curing (GF/PU)
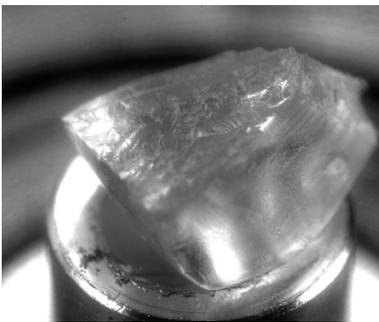
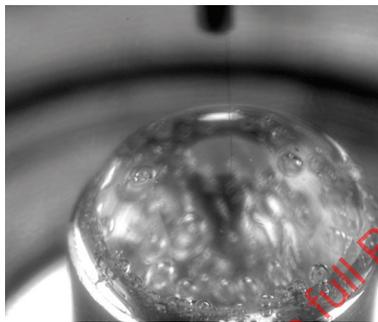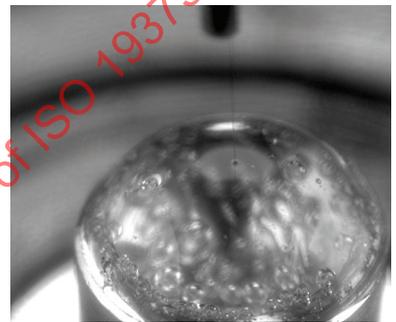a) Matrix preparation   b) Positioning/Embedding   c) Cooling

Figure A.6 — Drop forms no convex shape — too little matrix material (CF/PC)
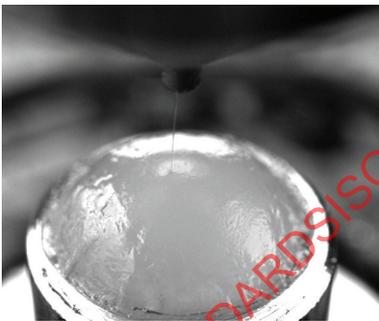

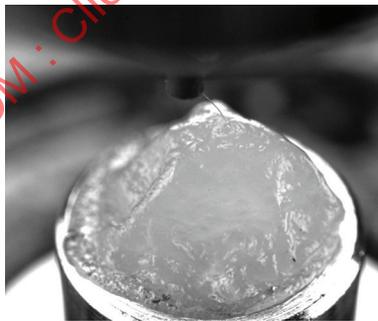a) Matrix preparation   b) Positioning/Embedding   c) Cooling

Figure A.7 — Development of bubbles while melting (CF/PC)

a) Preparing a valid test specimen using slow cooling of the matrix material — the fibre remains straight

b) Preparing an invalid test specimen due to fast cooling of the matrix material — the surface of the matrix shrinks irregularly, no perpendicular angle between fibre and matrix
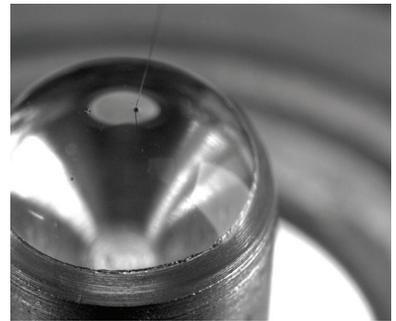
c) Preparing an invalid test specimen due to a misaligned fibre — occurred during cooling down

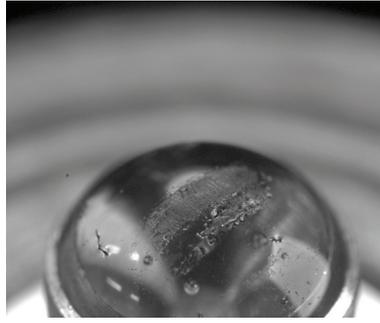**Figure A.8 — Misalignment of the fibre after fast cooling**

Figure A.9 — Incomplete melting — Preparing an invalid test specimen due to an incompletely melted matrix

Annex B
(informative)

Images of force-displacement curves of pull-out tests

In this annex, the exemplary force-displacement curves of pull-out tests of various fibre-matrix combinations is displayed, see Figure B.1 to Figure B.5.

First, data from typical pull-out tests are shown. For each combination, 3 curves are given indicating the variation from individual test to test. Second, regular pull-out curves are displayed in contrast to faulty tests that have failed due to typical error patterns.
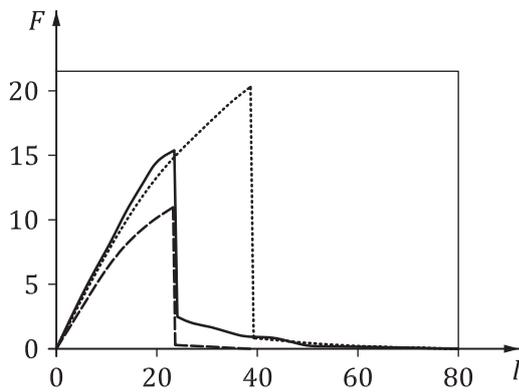
a) CF/One-Comp. EP
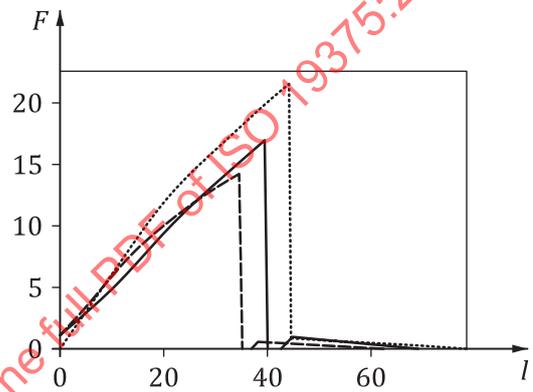
b) CF/Two-Comp. EP with anhydride hardener
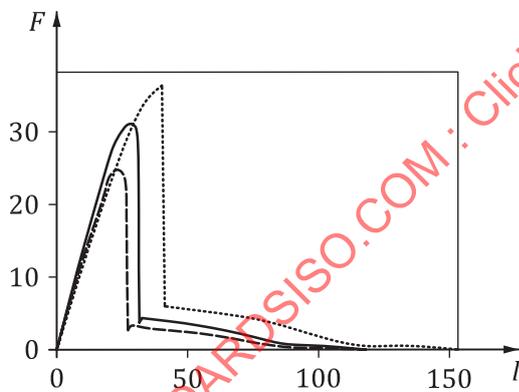
c) GF/Two-Comp. EP with amine hardener
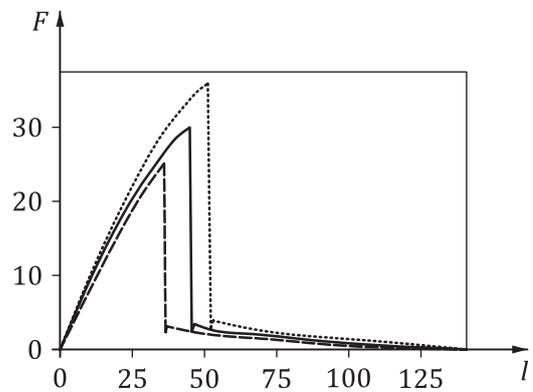
d) GF/PU
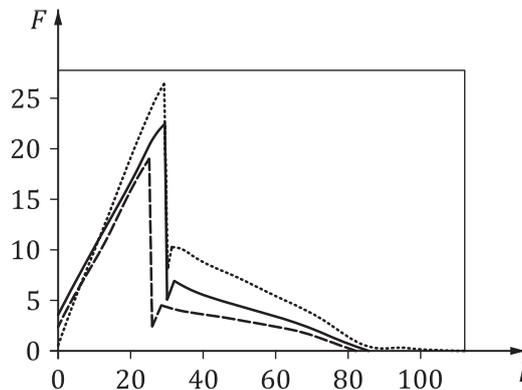
e) GF/VE

Key

l displacement, in μm

F force, in cN

Figure B.1 — Exemplary pull-out curves of thermoset matrices

a) GF/PP

b) rCF/PP

c) NF (Sisal)/PP

d) GF/PA6

e) CF/PA6

f) CF/PC

Key
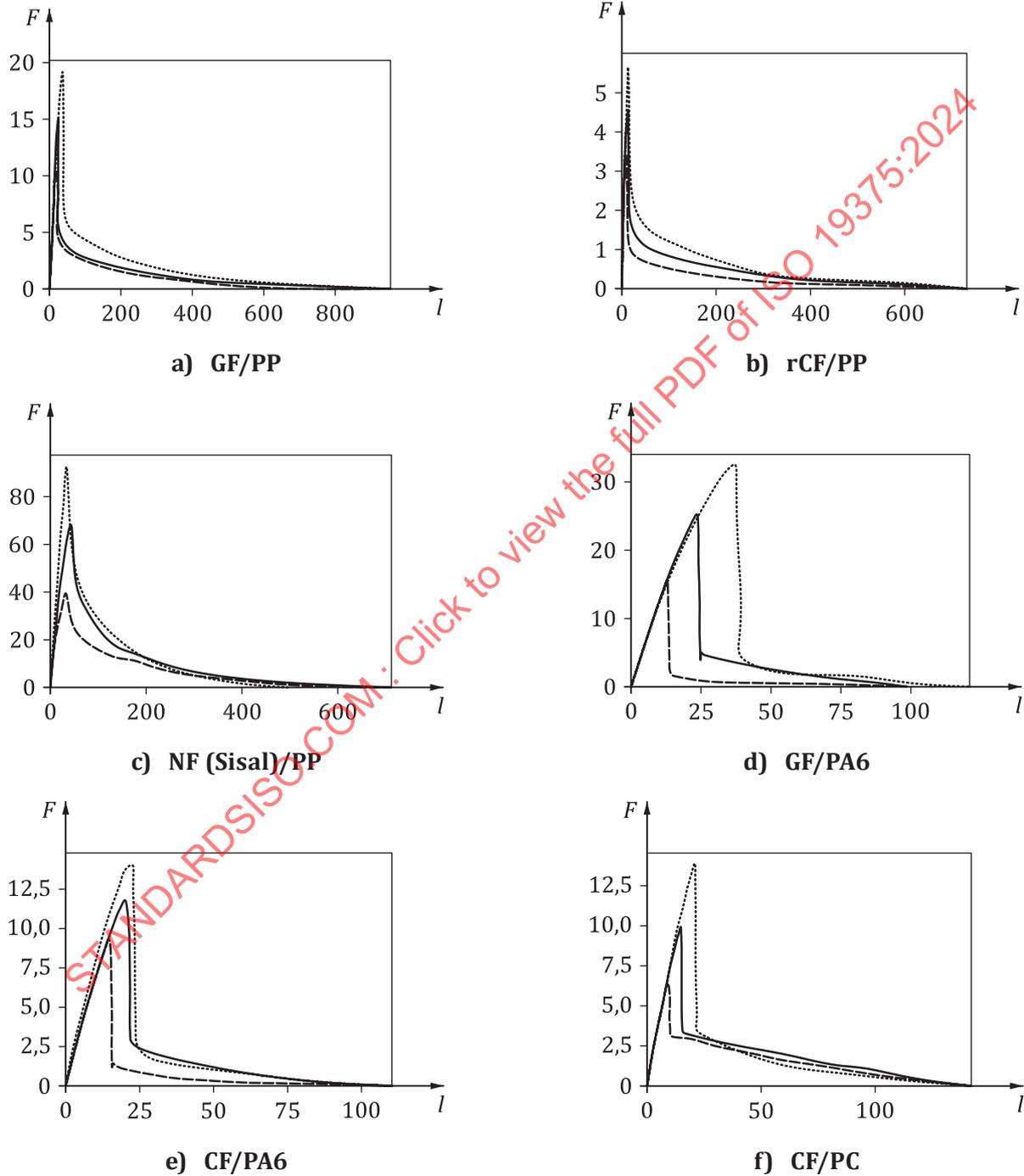
l   displacement, in μm

F   force, in cN

Figure B.2 — Exemplary pull-out curves of thermoplastic matrices, part 1

a) CF/PEEK

b) CF/PEBA

Key
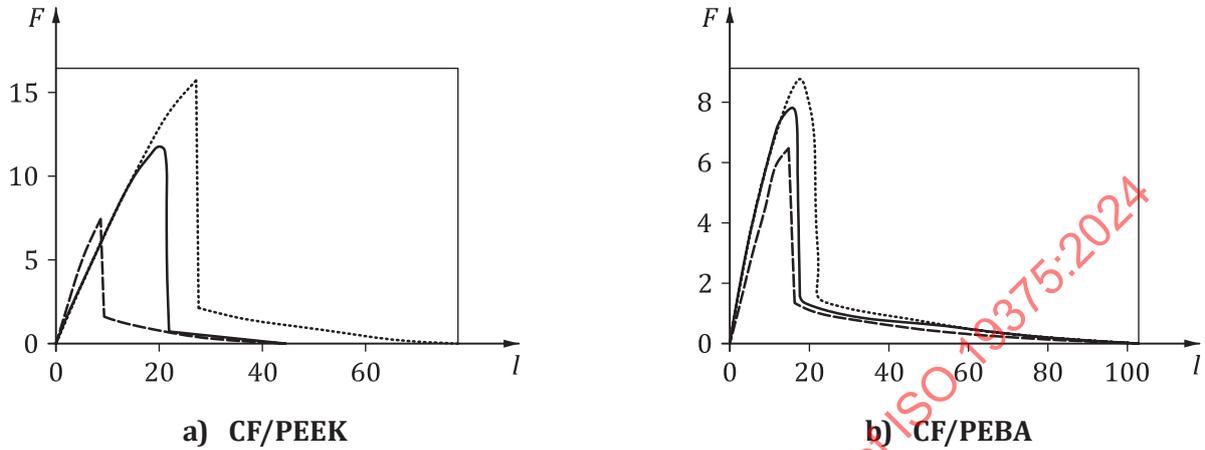
l   displacement, in μm

F   force, in cN

Figure B.3 — Exemplary pull-out curves from thermoplastic matrices, part 2

CF/Concrete

Key
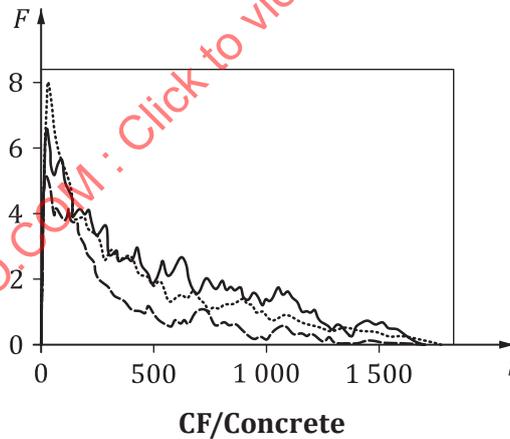
l   displacement, in μm

F   force, in cN

Figure B.4 — Exemplary pull-out curves from a concrete matrix
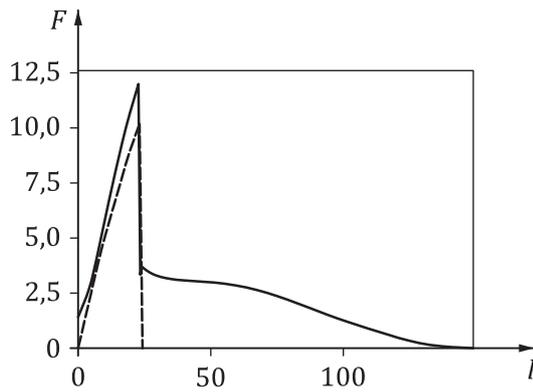
a) CF/PC — regular versus invalid:
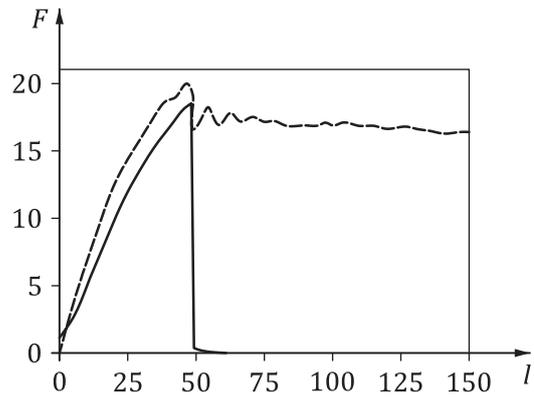
b) CF/One-Comp. EP — regular versus invalid:
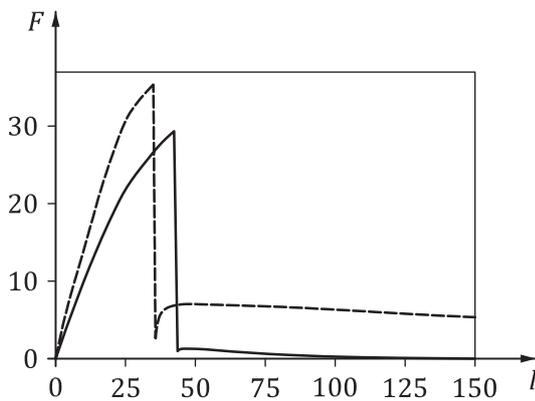
c) GF/Two-Comp. EP with amine hardener — regular versus invalid:
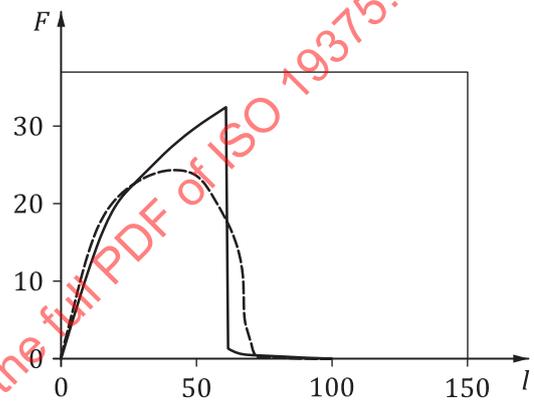
d) GF/Two Comp. EP with amine hardener — regular versus invalid:

Key

l   displacement, in µm

F   force, in cN

NOTE 1   Figure B.5 a): Regular pull-out (solid) vs. fibre breaking between clamp and matrix or inside the matrix (dashed). In the latter case, the friction-interaction-only part of the curve is missing (see Figure 6).

NOTE 2   Figure B.5 b): Regular pull-out (solid) vs. fibre slipping inside the clamp (dashed). In the latter case, the drop from the maximum force to the friction force is missing (see Figure 6) and the force does not reduce to 0 even at displacements much larger than the embedding depth.

NOTE 3   Figure B.5 c): Regular pull-out (solid) vs. fibre breaking inside the clamp (dashed). In the latter case, the fibre is pulled out from the clamp rather than the matrix. This typically results in apparent embedded lengths that exceed the expected range. This fault can also be identified and rejected by inspecting the test specimen.

NOTE 4   Figure B.5 d): Regular pull-out (solid) vs. fibre that has not been properly wetted by the matrix or has not been embedded deep enough (dashed). Embedding depths that are too low are marked by a smooth transition from an increasing force to the level of interfacial frictional force (round-off top). Furthermore, the maximum force and the embedded length may be exceedingly low.

Figure B.5 — Exemplary regular vs. invalid pull-out curves

Annex C
(informative)

Exemplary embedding and pull-out settings for thermoset matrices

The selection of fibre-matrix combinations is performed according to the industrial relevance in the different sectors. Table C.1 contains exemplary settings for embedding and pull-out of test specimens with thermoset matrices. It shows the settings for the specific fibres and matrices used during development of this document, in the form of a recipe. Note that even similar fibres and matrices can require different settings for successful tests. Table C.1 may serve as a starting point to find suitable settings.

Table C.1 — Exemplary embedding and pull-out settings for thermoset matrices

Parameter	Clause	Material					Remarks
		One-Comp. EP	Two-Comp. EP with anhydride hardener	Two-Comp. EP with amine hardener	PU	VE	
Matrix		One-Comp. EP	Two-Comp. EP with anhydride hardener	Two-Comp. EP with amine hardener	PU	VE	
Fibre		CF	CF	GF	GF	GF	
Fibre sampling and preparation	8.3	Wear gloves Remove 1st layer from roving then random fibre selection from roving piece Speed for linear density measurement: 2 mm/min GF: 50 mm vibrating length, 3 cN/tex pretension CF: 50 mm vibrating length, 3 cN/tex pretension Insert the fibre in the capsula of the FIMABOND with a protruding fibre length of about 2 mm to 3 mm Use inert gas flow of 0,1 LPM, if inert gas flow is switched on					
Matrix preparation	8.4						
General	8.4	Heating a small amount to 80 °C in water bath to soften the hard resin	—	—	Evacuate/dry hardener and resin separately for 45 min in vacuum	—	
Mixing ratio	8.4	—	According to manufacturer's instructions				
Mixing method	8.4	—	Manual	Manual	Dual asymmetric centrifuge, 2 000 min^{-1} for 6 min	Manual	
Vacuum/evacuation	8.4		No	No	Yes	No	
Preparation of matrix droplet	8.5						
Crucible material	8.5	Aluminium	Aluminium	Aluminium	Stainless steel	Aluminium	For PU: stainless steel crucible to avoid bubble formation
Amount of matrix material	8.5						Sufficient to form a convex shaped droplet without flowing over the edge of the crucible
Inert gas flow/Extra inert gas flushing step	8.5			Yes/No			
Degassing/Pre-heating	8.5	$T_1 = 90$ °C $r_1 = 99,9$ K/min $t_1 = 90$ s $T_E = 240$ °C $r_{TE} = 99,9$ K/min $t_{TE} = 90$ s Inert gas flow: On Holding air: On	$T_E = 80$ °C $r_{TE} = 99,9$ K/min $t_{TE} = 90$ s Inert gas flow: On Holding air: On	$T_E = 25$ °C $r_{TE} = 99,9$ K/min $t_{TE} = 90$ s Inert gas flow: On Holding air: On	$T_E = 80$ °C $r_{TE} = 99,9$ K/min $t_{TE} = 60$ s Inert gas flow: On Holding air: On	$T_E = 80$ °C $r_{TE} = 99,9$ K/min $t_{TE} = 90$ s Inert gas flow: On Holding air: On	T_1, r_1 and t_1 are parameters of a first intermediate step, necessary to adjust the viscosity of the one-component EP
Fibre embedding	8.6						
Inert gas flow for positioning and embedding/Holding air	8.6			Inert gas flow: On Holding air: On			

Table C.1 *(continued)*

Parameter	Clause	Material					Remarks
		One-Comp. EP	Two-Comp. EP with anhydride hardener	Two-Comp. EP with amine hardener	PU	VE	
Matrix		One-Comp. EP	Two-Comp. EP with anhydride hardener	Two-Comp. EP with amine hardener	PU	VE	
Fibre		CF	CF	GF	GF	GF	
Embedding speed v_E	8.6	100 µm/min	100 µm/min	200 µm/min	300 µm/min	200 µm/min	
Embedding depth l_{ED}	8.6	80 µm	30 µm	120 µm	110 µm	75 µm	
Final forming	8.6	No	No	No	No	No	
Solidification of the test specimen	8.7						
Curing step	8.7	$T_C = 240\ °C$ $t_{TC} = 13$ min Inert gas flow: On Holding air: On	$T_C = 170\ °C$ $r_{TC} = 99,9$ K/min $t_{TC} = 5$ min Inert gas flow: On Holding air: On	$T_C = 105\ °C$ $r_{TC} = 99,9$ K/min $t_{TC} = 120$ min Inert gas flow: On Holding air: On	$T_C = 120\ °C$ $r_{TC} = 99,9$ K/min $t_{TC} = 10$ min Inert gas flow: On Holding air: On	$T_C = 160\ °C$ $r_{TC} = 99,9$ K/min $t_{TC} = 4$ min Inert gas flow: On Holding air: On	
Cooling step	8.7	$T_F = 50\ °C$ Active cooling: Off (free cooling) Inert gas flow: On Holding air: On	$T_F = 40\ °C$ $r_{TF} = $ max Active cooling: On Inert gas flow: Off Holding air: Off	$T_F = 40\ °C$ $r_{TF} = $ max Active cooling: On Inert gas flow: Off Holding air: Off	$T_F = 40\ °C$ $r_{TF} = $ max Active cooling: On Inert gas flow: On Holding air: Off	$T_F = 40\ °C$ $r_{TF} = $ max Active cooling: On Inert gas flow: Off Holding air: Off	
Test specimen requirements and final validation	8.8	See 8.8					
Post-preparation and conditioning	8.9						
Storing in desiccator	8.9	> 24 h	> 12 h				
Cleaning step	8.9	80 °C for 1 h, cooling down in desiccator to lab temperature for 30 min	Not required	80 °C for 1 h, cooling down in desiccator to lab temperature for 30 min	Not required	Not required	The post preparation at 80 °C was conducted to remove resin deposits from the fibre surface which is clamped in the FAVIMAT+ to ensure proper clamping without fibre slippage
Pull-out test	8.10						
Distance between matrix and draw-off clamp	8.10	GF: 5 to 10 × fibre diameter CF: 5 to 15 × fibre diameter					
Pretension	8.10	0,01 cN					
Pull-Out speed		0,1 mm/min					
End of test	8.10	0,01 cN					

Typical test results and precision data for these thermoset fibre-matrix combinations can be found in Annex G.

Annex D
(informative)

Exemplary embedding and pull-out settings for thermoplastic matrices

The selection of fibre-matrix combinations is performed according to industrial relevance in the different sectors. Table D.1 contains exemplary settings for embedding and pull-out of test specimens with thermoplastic matrices. It shows the settings for the specific fibres and matrices used during development of this document, in the form of a recipe. Note that even similar fibres and matrices can require different settings for successful tests. Table D.1 may serve as a starting point to find suitable settings.

Table D.1 — Exemplary embedding and pull-out settings for thermoplastic matrices

Parameter	Clause	Material					Remarks
		PP	PA6	PC	PEEK	PEBA	
Matrix							
Fibre		GF, rCF, NF	GF, CF, rCF	CF	CF	CF	
Fibre sampling and preparation	8.3	Wear gloves Remove 1st layer from roving then random fibre selection from roving piece Speed for linear density measurement: 2 mm/min GF: 50 mm vibrating length, 3 cN/tex pretension CF: 50 mm vibrating length, 3 cN/tex pretension rCF: 50 mm vibrating length, 3 cN/tex pretension or 25 mm, 1 cN/tex NF: 25 mm vibrating length, 3 cN/tex pretension Insert the fibre in the cannula of the FIMABOND with a protruding fibre length of about 2 mm to 3 mm Use inert gas flow of 0,1 LPM if inert gas flow is switched on					
Matrix preparation	8.4	Dry at 80 °C for 4 h	Dry at 80 °C for 4 h	Dry at 80 °C for 4 h	Dry at 120 °C for 6,5 h	Dry at 80 °C for 4 h	If bubbles appear during melting: increase drying
Preparation of matrix droplet	8.5						
Crucible material	8.5	Aluminium, roughened	Aluminium, roughened	Aluminium, roughened	Aluminium, roughened	Aluminium, roughened	
Amount of matrix material	8.5	1/2 of a pellet	1/2 of a pellet	1/2 of a pellet	2/3 of a pellet	1/2 of a pellet	Sufficient to form a convex shaped droplet without flowing over the edge of the crucible
Inert gas flow/extra inert gas flushing step	8.5	On/no	On/3 min before heating up	On/no	On/no	On/3 min before heating up	To avoid degradation of sensitive materials, an extra flushing step should be included
Heating to embedding temperature	8.5	T_E = 240 °C t_{TE} = 7 min r_{TE} = 25 K/min Inert gas flow: On Holding air: On	T_E = 250 °C t_{TE} = 2 min r_{TE} = 99,9 K/min Inert gas flow: On Holding air: On	T_E = 265 °C t_{TE} = 3 min r_{TE} = 99,9 K/min Inert gas flow: On Holding air: On	T_E = 400 °C t_{TE} = 3 min r_{TE} = 99,9 K/min Inert gas flow: On Holding air: On	T_E = 230 °C t_{TE} = 2 min r_{TE} = 99,9 K/min Inert gas flow: On Holding air: On	Droplet shall be completely melted after this step, otherwise increase the time to embedding or the embedding temperature
Fibre embedding	8.6						
Inert gas flow for positioning and embedding/Holding air	8.6			Inert gas flow: Off Holding air: On			
Embedding speed v_E	8.6	GF: 1 000 µm/min rCF: 1 600 µm/min NF: 1 000 µm/min	GF: 200 µm/min CF: 500 µm/min rCF: 500 µm/min	CF: 500 µm/min	CF: 300 µm/min	CF: 500 µm/min	
Embedding depth l_{ED}	8.6	GF: 1 000 µm rCF: 800 µm NF: 1 000 µm	GF: 100 µm CF: 100 µm rCF: 100 µm	CF: 225 µm	CF: 90 µm	CF: 100 µm	
Final forming	8.6	Not required	Inert gas flow: On t_F = 60 s	Not required	Inert gas flow: On t_F = 30 s	Inert gas flow: On t_F = 60 s	

Table D.1 *(continued)*

Parameter	Clause	Material					Remarks
Matrix		PP	PA6	PC	PEEK	PEBA	
Fibre		GF, rCF, NF	GF, CF, rCF	CF	CF	CF	
Solidification of the test specimen	8.7						
Inert gas flow/Holding air	8.7	Inert gas flow: On Holding air: Off					
Cooling step 1	8.7	T_{BS} = 120 °C r_{TBS} = 50 K/min Inert gas flow: On Holding air: Off	T_{BS} = 180 °C r_{TBS} = 10 K/min Inert gas flow: On Holding air: Off	T_{BS} = 150 °C r_{TBS} = 50 K/min Inert gas flow: On Holding air: Off	T_{BS} = 300 °C r_{TBS} = 25 K/min Inert gas flow: On Holding air: Off	T_{BS} = 150 °C r_{TBS} = 25 K/min Inert gas flow: On Holding air: Off	
Cooling step 2	8.7			T_F = 40 °C maximal r_{TF} Active cooling: On Inert gas flow: Off Holding air: Off			
Test specimen requirements and final validation	8.8			See 8.8			
Post-preparation and conditioning	8.9	Storing in desiccator for 12 h to 24 h	Tempering at 150 °C for 60 min, then storing in desiccator for 12 h to 24 h	Storing in desiccator for 12 h to 24 h	Storing in desiccator for 12 h to 24 h	Storing in desiccator for 12 h to 24 h	
Pull-out test	8.10						
Distance between matrix and draw-off clamp	8.10	GF: 5 to 10 × fibre diameter CF: 5 to 15 × fibre diameter NF: 5 to 10 × fibre diameter					
Pretension	8.10	0,01 cN					
Pull-Out speed		0,1 mm/min					
End of test		0,01 cN					