EATC Properties and test methods for LFT materials

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1 General notes

Testing of material properties is described below for LFT materials, processed by press or injection moulding or similar processes. It is well known that material properties vary with fibre length, fibre dispersion and fibre orientation, therefore, these factors should be taken into account, when measuring properties.
The guidelines in this document apply to polypropylene based LFT's. For other matrix materials additional guidelines may be necessary.

1.1 How to deal with fibre orientation?

Fibre orientation is mostly flow induced, and will vary with location in the product or test plate. In addition to this the fibre orientation for press moulding is also strongly influenced by the pre-orientation present in the GMT blank or extrudate as it comes from the extruder and is put into the mould. (Extrudate = extruded "sausage" of material, sometimes also called plastificate.)
When taking specimens from a moulded plate in different directions, e.g. 0° and 90° with respect to the flow direction, an indication of flow induced material anisotropy is obtained, but the average level of properties cannot be properly used for design calculations or material comparisons.
The test method for determining isotropic properties described in this document solves the problem of measuring the property level for an unknown orientation state in a test plate. The isotropic property level is that level that would be obtained for a material where the fibres are equally distributed in all directions. It is independent of the actual fibre orientation state in a test plate.
The isotropic property level may be used for e.g. material comparison or initial mechanical calculations, when fibre orientation due to mould filling is not yet known, as the injection locations or extrudate placement are not yet defined.
The isotropic properties are derived through calculation from specimen test results in 0°, 45° and 90° direction
It should also be noted that directly injection moulded tensile bars, as often used for material datasheets from material suppliers, contain a high level of orientation, and are rarely indicative for material performance in real parts.

1.2 How to deal with material quality: fibre length and dispersion?

For any particular material, two other factors determine the material quality state, as delivered by the production process:

1. Fibre length (or actually fibre length distribution)
2. Fibre dispersion

Dispersion is the term used to describe how glass fibres are distributed in the thermoplastic matrix. It can range from evenly dispersed to concentrated in glass bundles.
Different fibre length and dispersion will change the level of isotropic properties.

Different machines and process settings may yield different material property levels, therefore, it is important to note how the test plates were produced. It is recommended to choose production settings that yield a material quality that is typical of industrial applications.
To characterise the material quality state in the test plate it is recommended to make X-Ray pictures to characterise dispersion state, and to perform fibre length measurements. Examples of different dispersion states can be seen in Figure 1. (Note that at the same time, these X-Ray pictures can be used also to check the homogeneity and direction of fibre orientation as discussed further in this test method.)

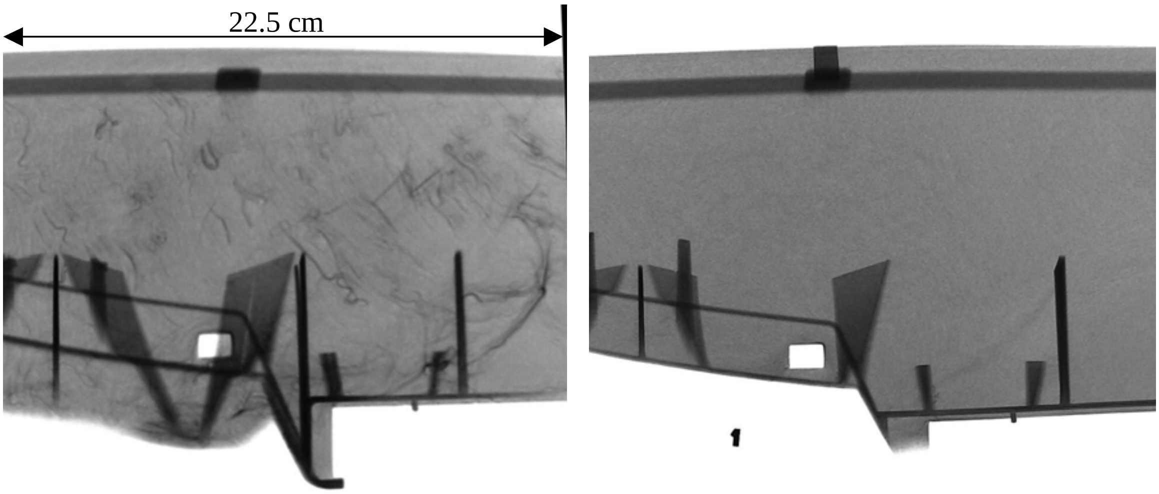

Figure 1, Example of coarse (left) and fine dispersion (right) as seen on an X-Ray picture.

Fibre length measurements can be made by a direct method (counting a lot of fibre length's) or indirectly by falling dart impact testing. For the latter case a relation of fibre lengths against falling dart impact values should be available for the specific material, while the influence of specimen thickness and dispersion level must also be known. In general this indirect method gives a good indication for moulded material quality, but will nevertheless be less accurate.
For ease of reference, material quality may be expressed with a single number for weight averaged fibre length (l_w), although this number does not give a measure for fibre dispersion. At present, fibre dispersion can only be qualitatively measured by using X-Ray pictures.

By characterising the material quality state in such a way, material quality in real parts can be compared to material quality in the used test plates. In those plates the material can be fully tested on isotropic and other properties, which is mostly not possible on real parts due to geometric restrictions.

1.3 Why different plates/specimens for compression and injection moulding?

Different plate and specimen geometries are described for compression and injection moulding. There are two reasons for this:

The main reason is that the fibre orientation distribution in moulded plates will be different for the two processes. Especially for the injection moulding process fibre orientation will often show more or less circular shaped patterns in the core. This is even the case when film gates used, leading to uni-directional filling of the plate. The circular patterns are the result of material flowing from a small sized injection point into the mould. Because of this circular pattern, one should take care that specimens are always taken on the same position, and fibre orientation is constant over the measurement section of the specimen. A circular pattern means by definition not a constant fibre orientation distribution, and thus a constant distribution can only be approximated to by taking specimens small enough in relation to the plate size. Small specimens however yield less accurate results than larger specimens. Therefore a compromise must be found between realistic plate sizes and optimum specimen sizes.
In contrast, press moulded rectangular plates can be made with a more homogeneous orientation in one direction only, not showing any circular pattern. However some local variations due to bundle structure may be present. Accuracy is then improved by taking relatively large specimens.

The second reason for a difference in plate sizes is the availability of plate moulds at different companies' or institutes, which in general are matched to the plastification machine size. E.g. very large plates cannot be filled via relatively small injection moulding machines. Therefore, given the previous recommendations, the optimum mould dimensions must be selected, within the given dimensional restrictions.

2 Plate geometry and manufacture

As plate geometry, specimen positions and specimen type are different for compression and injection moulding they will be treated in separate subchapters below.

2.1 Compression moulding

2.1.1 Plate geometry

A rectangular plate shall be used, where the extrudate or plastificate is placed on one end, and the specimens are taken from the flow region as shown in Figure 2:

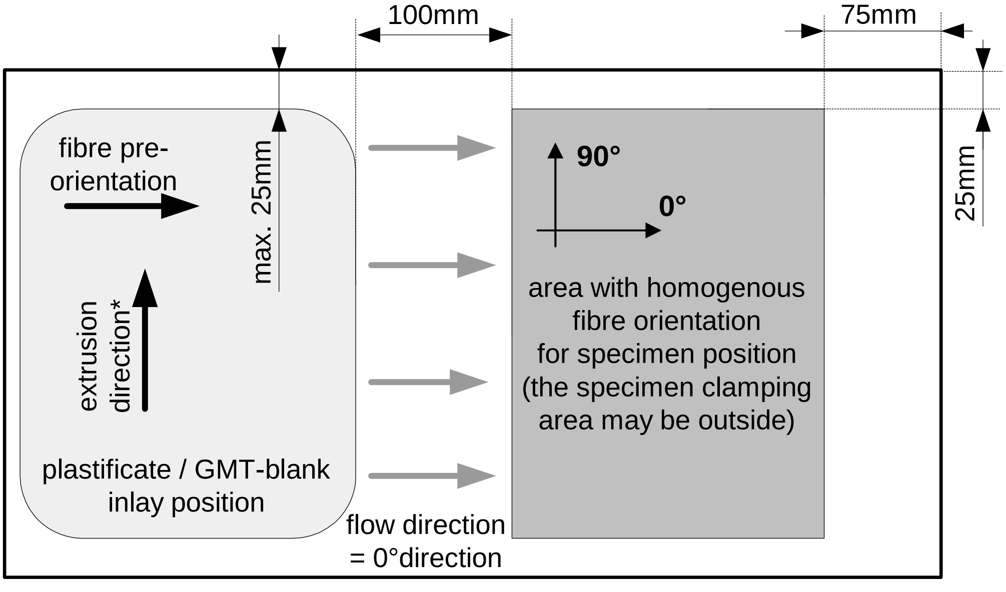

Figure 2, Press moulded plate
**For extruded materials, fibre pre-orientation is usually perpendicular to the extrusion direction.*

When the blank or plastificate is put in the position as shown in Figure 2, the compression moulding process provides a relatively large area with homogenous fibre orientation. The 0°-direction is defined as the flow direction. In most cases this will be the same as the main fibre direction.
While the fibre orientation is homogenous very close to the side edges, it may be not so at the edge which is positioned at the end of the flow. At this region knit lines may occur, and this is why a certain larger distance has to be kept from that side, shown as 75 mm in Figure 2.
Specimens are to be taken out of the region with homogenous fibre-orientation. An X-Ray picture of the plate is recommended to check the homogeneity of the fibre-orientation and the homogeneity of fibre concentration over the flow length. A sufficient homogenous example is shown in Figure 3.

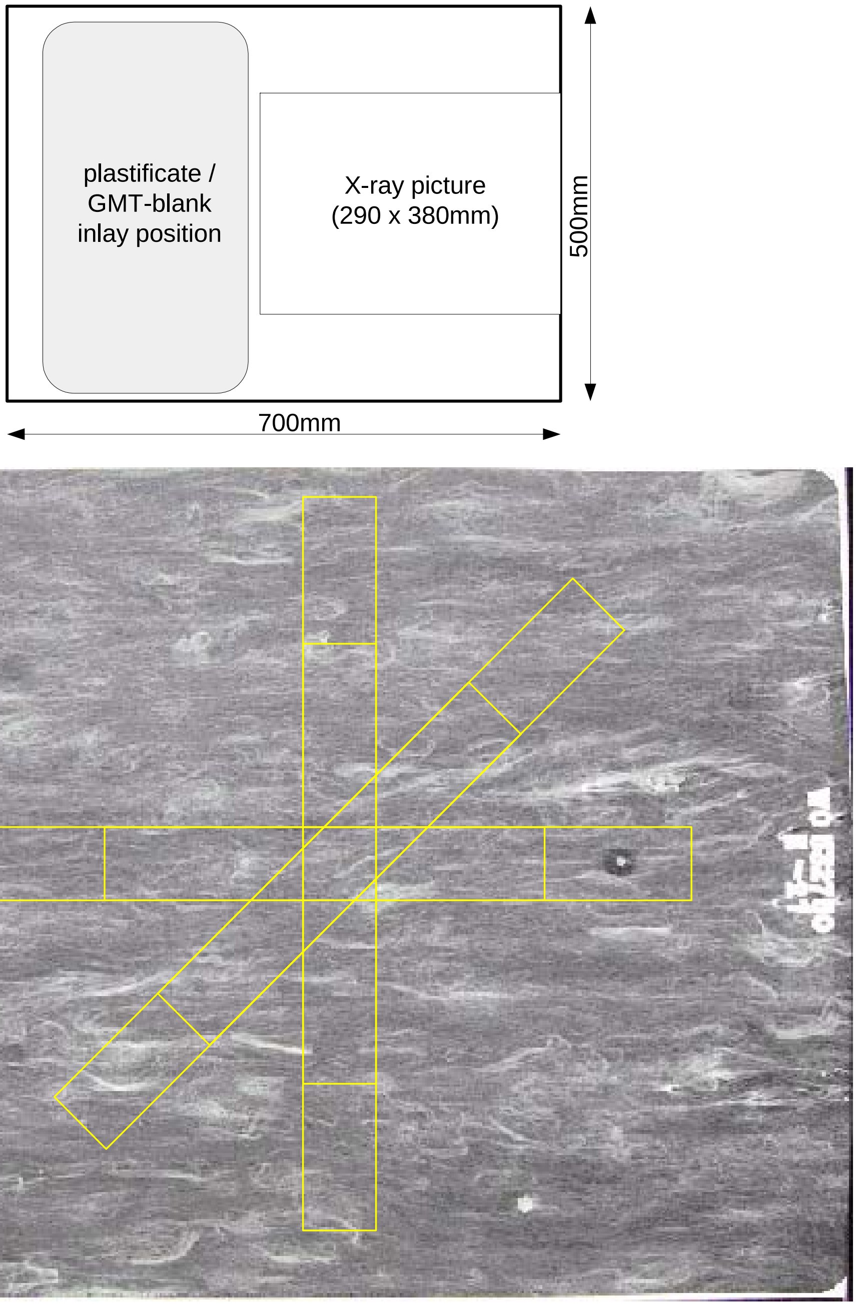

Figure 3, Example of X-Ray picture of moulded plate in region of homogenous fibre orientation. Sample dimensions are shown as an example.

The width of the plate-tool has to be 200mm or more. A tool geometry of 250 x 600mm is recommended. For more specific guidelines see under chapter "Tensile properties, isotropic", page 11.
Preferred thickness values are 2, 3 and 4 mm especially when testing properties as a function of thickness. If one single value for thickness is chosen, 3 mm is preferred.

2.1.2 Plate manufacture

Fibre pre-orientation of GMT-blank or LFT-plastificate should be in 0° direction.
(Note: most extrudates will have this pre-orientation, which is normal to the extrusion direction.)
LFT-Plastificate or GMT-blank should cover the whole width of the plate-tool in order to have a linear flow lengthwise.

All plates shall be moulded under equilibrium conditions at a constant cycle time, taking care to discard sufficient plates during start-up.

Moulding conditions should represent a typical material quality as would be found on larger scale industrial applications. It is advised to record all relevant process parameters.

2.2 Injection Moulding

2.2.1 Plate geometry

In contrast to the press moulded plates, injection moulded plates will be used to cut out one specimen only per plate, either in the 0, 45 or 90° direction.
Any moulded plate can be used for taking out tensile bar test specimens, provided that the orientation state doesn't change too much within the test specimens. This is explained in Figure 4 to Figure 7 below:

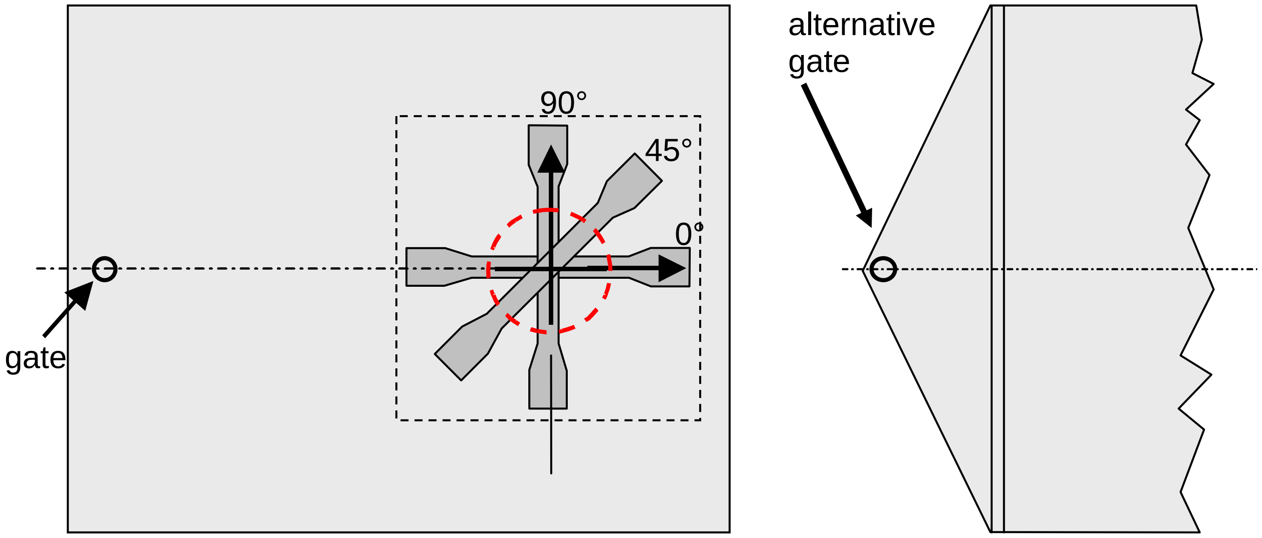

Figure 4, Example plate, showing tensile specimens in 3 directions.
Note: many alternative gating positions or lay-outs are possible, e.g. film gates are also allowed. Dotted line indicates allowable area for falling dart impact specimens.

The centre of each specimen should be on the same location as shown in Figure 4.
In general the fibre orientation will not be homogeneous over the whole plate but will follow more or less the curved shape as given by the expansion flow at the gate. (Note that a film gate does not change this pattern very much, even though the filling of the plate may look different). The location where the specimens should be taken, indicated by the dashed circle in Figure 4, should be such that the variation in fibre orientation in the measurement section with the specimen loading direction should not be larger than 10° maximum, as shown in Figure 5.
Although the picture shows an example for the 90° specimen, the max 10° deviation also applies to the other directions. In general however the 90° specimen will be the most critical, and requires plates wide enough to have acceptable orientation differences within the specimen.
Note that for long fibre materials the core orientation can be made easily visible by X-Ray pictures.

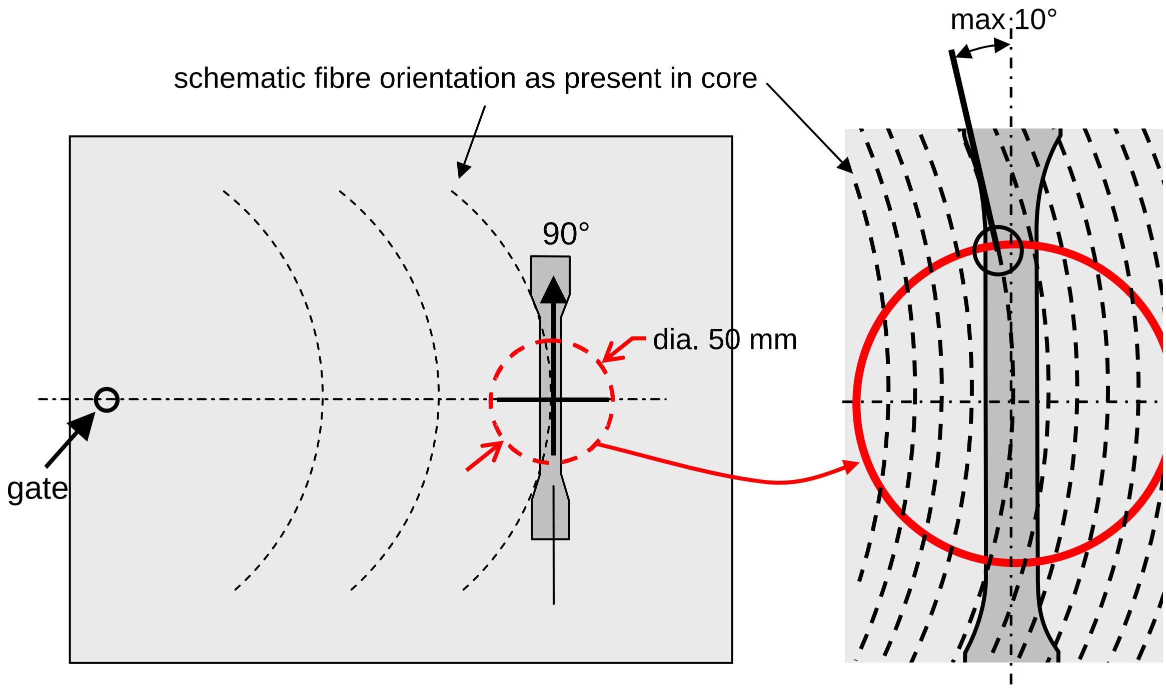

Figure 5, Example of 90° specimen, shown together with fibre orientation in the core (dashed lines).At the edge of the 50mm E-modulus measurement area, a line drawn tangential to the core orientation at this point may not deviate more than 10° with the specimen axis.

One example of an acceptable plate geometry is shown in Figure 6 below, including the core orientation as shown by X-Ray:

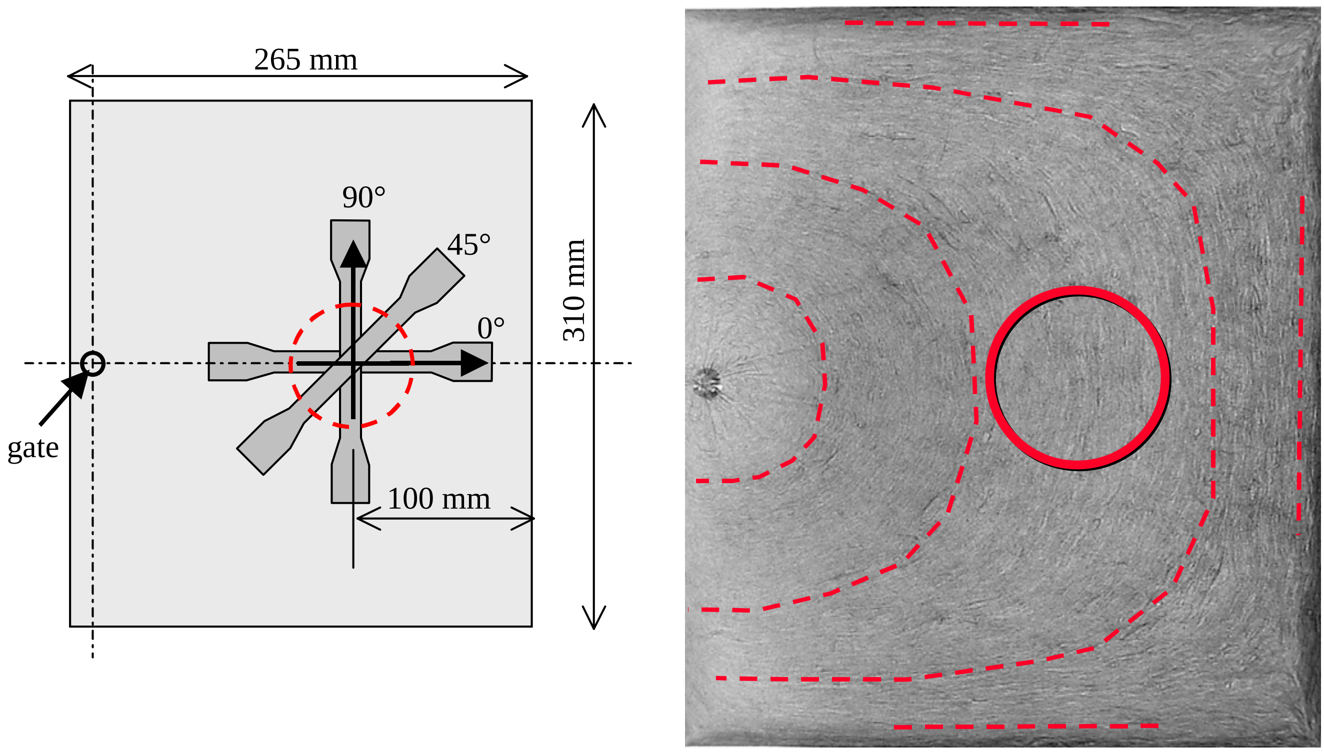

Figure 6, Example of acceptable plate dimensions and core orientation made visible by X-Ray.

An example of unacceptable fibre orientation is shown in Figure 7 below, where specimen positions are plotted over the X-Ray image.

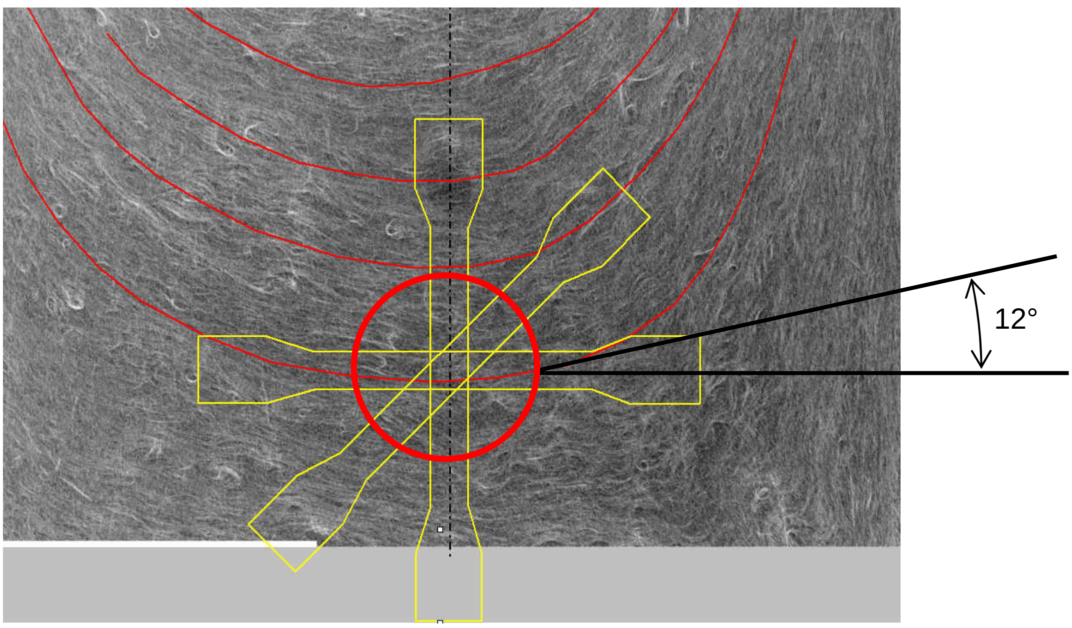

Figure 7, Example of just unacceptable fibre orientation variation within a specimen.

Plate thickness will have only minor influence on isotropic properties. A thickness typical for industrial applications is recommended, which will usually lay between 2 and 3 mm.
If only one thickness is chosen for testing, 3 mm is recommended.

2.2.2 Plate manufacture

Moulding conditions should represent a typical material quality as would be found on larger scale industrial applications. Alternatively a set of moulding conditions can be used, yielding sets of material properties for each particular moulding condition.

All plates should be moulded under equilibrium conditions, at a constant cycle time, taking care to discard sufficient plates during start-up.
It is preferred to use an automatic machine cycle operation.

It is advised to record all relevant process parameters, including plastification times.

3 Tensile properties, isotropic

3.1 Specimen positions

3.1.1 Compression moulding

The specimen position according to the fibre orientation is defined in Figure 8. Specimens are to be taken in 0°, 45° and 90° with respect to the flow direction (0° direction).
The dark grey areas indicate the allowable positions for the specimens. Specified distances can be larger if the plate geometry does allow it.
More than one specimen can be taken from one plate.

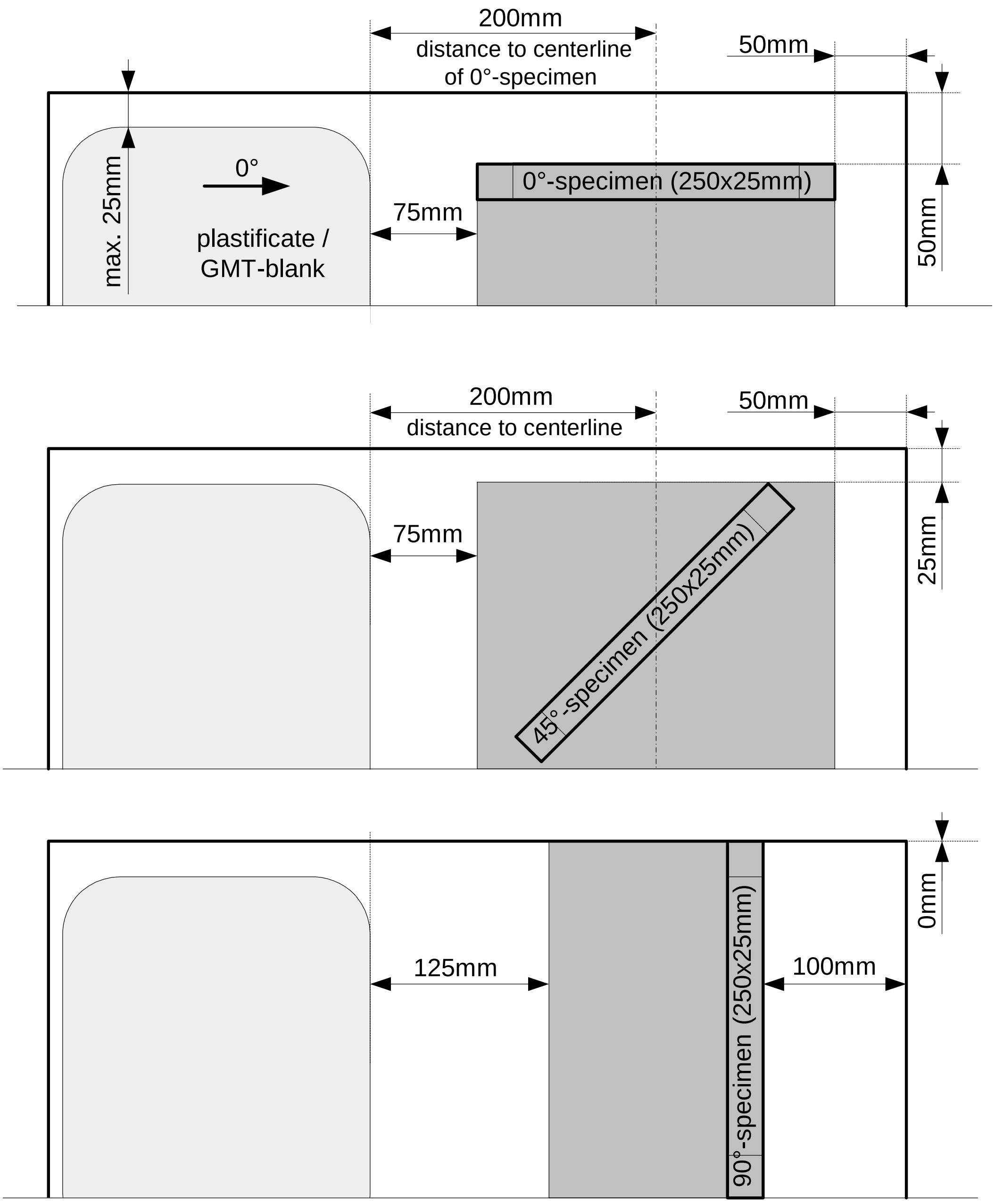

Figure 8: Compression moulding plate dimension details. The dark grey areas mark where the specimens of different fibre orientation can be taken. In bright grey the extrudate/plastificate inlay is shown.

The ends of the 90°-specimens (clamping area) may be placed closer to the plate-edge as defined in Figure 2, because this part is used for clamping the samples into the tensile test machine.

3.1.2 Injection moulding

The specimen positions for injection moulded plates are linked to the chosen plate geometry as described under chapter 2.2.1

3.2 Specimen geometry, preparation

3.2.1 Compression moulding

Tensile specimens shall have dimensions 250 x 25 mm. (ISO 527/4, type 2 specimen)
If 250mm long specimens aren't feasible, the minimum length should be at least 200mm. For tensile tests the distance between grips has to be 150mm.

Several machining options like milling, sawing or water jet cutting may be used as long as care is taken to ensure a good quality finish to the specimen edge. Laser cutting or stamping are not recommended, as they will affect edge quality.

3.2.2 Injection moulding

Specimen type is defined by ISO 527/1B.

Specimens may be machined from plate or cut by water jet, taking care to obtain a smooth specimen edge. Laser cutting or stamping are not recommended, as they will affect edge quality.

3.3 Tensile testing

Tests are to be carried out according ISO527 part 4.
Test speed for the complete test will be 5 mm/min. (Thus the test speed applies both to E-modulus and tensile strength measurement)

10 specimens per orientation have to be tested.
If there is a small variation between individual specimen results, characterised by a standard deviation < 5% of the average E-modulus value result for a particular orientation, the minimum number of specimens per orientation may be reduced to 5. In all other cases a reduced number of specimens shall be noted on the test protocol.

The specimens for each orientation are to be taken out of a minimum of 5 different plates. (This will be automatically the case for plates manufactured by injection moulding, as only one specimen per plate is taken there.)

3.4 Isotropic properties calculation

Isotropic properties can be calculated from the 0°, 45° and 90° test results as follows:

Any anisotropic material state, such as present in the tested plate, can be described by the following material parameters: E_1, E_2, G_{12}, ν_{12} and ν_{21}, which are respectively: E-moduli in 1 and 2 direction, Shear modulus and Poisson's ratios in 1 and 2 direction. (first 4 parameters are independent, ν_{21} is a dependent parameter)
All these properties may be measured directly, and with the results, using laminate theory, the isotropic E-modulus can be calculated.
In a similar way, the isotropic thermal expansion coefficient can be calculated. For this the anisotropic material parameters α_1 and α_2, coefficients in 1 and 2 direction, are needed. These coefficients can be directly measured.
Alternatively, with very good approximation, values for shear modulus may be estimated from 0°, 45° and 90° tensile bars (see ref.1). In that case E_1 and E_2 are measured directly as 0° and 90° results, and the shear modulus is derived indirectly from the 45° tensile result, while the Poisson's ratio ν_{12} is estimated. For this calculation any laminate theory program can be used, or e.g. the Excel sheet that can be downloaded from www.eatc-online.org

Result of these calculations will be the following isotropic properties:
E (isotropic modulus)
G (isotropic shear modulus)
ν_{12} (isotropic Poisson's ratio)

Ref.1 "Characterisation and performance of long fibre materials", W.Schijve, 8. Internationale AVK-TV Tagung Baden-Baden, September 2005

3.5 Presentation of tensile test results

The test report shall include information on used plate dimensions and manufacturing method.

Test results shall include:

- Results per orientation (0, 45 and 90°): Modulus, Tensile strength, Failure elongation.
  including average value and standard deviation.
- Isotropic calculation result: Modulus, Shear Modulus, Poisson's ratio, Tensile strength.
  including method of calculation, e.g. SABIC Excel sheet, version 1, or company internal calculation method.
- Any deviation from the described test procedure shall be mentioned.

3.6 Further notes on tensile test

As tensile test samples are not always perfectly flat, and fibre orientation is not always perfectly in line with the test specimen direction, attention should be paid to proper specimen clamping and placement of strain transducers on the specimen. Non-optimal test conditions may influence the value of the E-modulus measured. To check the correct execution of a test, the stress strain curve should be checked as shown in Figure 9. A so-called S-curve shape is an indication for a poorly executed test and will yield a too low E-modulus value.

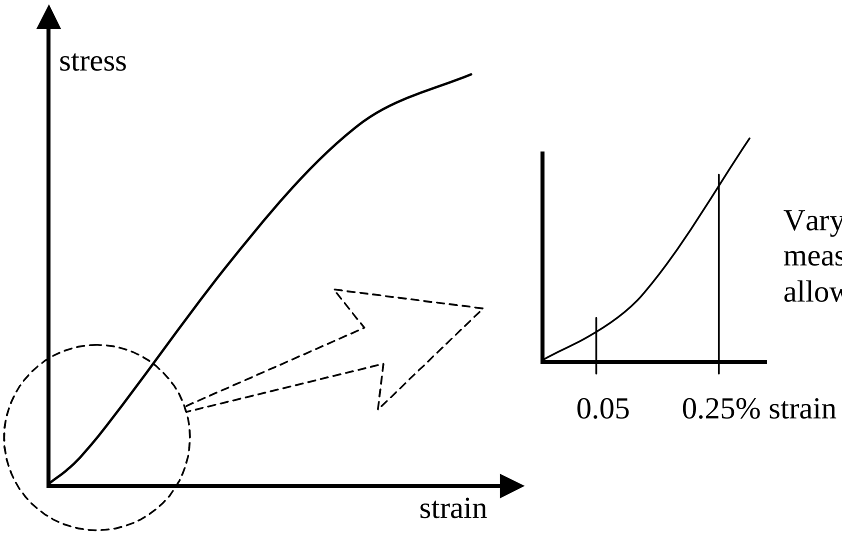

Figure 9, Example of stress-strain curve showing "S-curve" shape, yielding incorrect E-modulus.

4 Falling dart impact properties

Because fibre orientation homogeneity and orientation are much less important for falling dart impact testing, there is no difference in testing between compression and injection moulding. The total penetration energy measured in the falling dart impact test has proven to be a good measure for material quality, including fibre length.

4.1 Specimen positions

Specimens should be taken close to the region where tensile specimens are taken, to ensure that the same material quality is tested. This is indicated in Figure 2 and Figure 4.

4.2 Specimen geometry and preparation

Specimen type is ISO 6603/2, specimen size is 60x60mm (square or circular).

Specimen thickness is the same as used for determining tensile properties.
Note: Even though the result may be expressed as penetration energy in Joules/mm thickness, this result is not a pure material property, but it is still strongly dependant on thickness.

Cutting method may be chosen freely.

4.3 Falling dart impact testing

Testing is carried out according ISO 6603/2, using a 40 mm diameter specimen clamping and a 20 mm diameter striker with spherical tip.
The number of specimens required is at least 10 divided over at least 5 plates

By deviating from what is mentioned in ISO 6603/2, the total penetration energy should be measured until the striker has penetrated the specimen 20 mm, see Figure 10. In this way the total energy for fracturing the complete specimen is always measured. The cut-off is less sensitive to vibrations in the measured force signal, which might drop too early below the 50% maximum force level as was mentioned in the original ISO 6603/2 text. Further, the total energy has proven to be the most reliable indicator for fibre length, and an early cut-off would reduce this reliability.

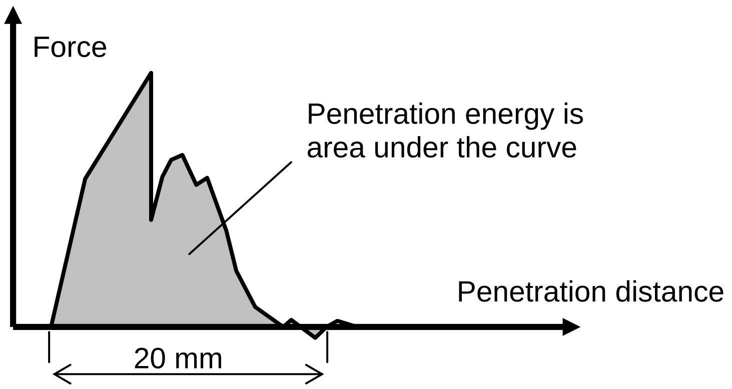

Figure 10, Total penetration energy, based on 20 mm penetration criterion.

It is also good practise to check that the force level returns to approximately zero, when the penetration has passed.

4.4 Presentation of the results

Result shall include the maximum force, and total penetration energy as defined above, expressed in J/mm. Both average and standard deviation shall be reported.
The results shall also include the actual measured thickness. This is to avoid confusion when comparing material data coming from different sources, measured on different thickness specimens, which cannot be compared with each other.

5 Ash content

Specimens should be taken close to the region where tensile specimens are taken, to ensure that the same material quality is tested. This is indicated in Figure 2 and Figure 4.
When taking specimens out of injection moulded plates (or parts), do not use the end of flow position (edge of the plate), as this may show a slightly higher glass content. For most, but not all materials, exclusion of the last 10 mm of the part will generally be sufficient.
Specimens shall be taken from at least 2 different plates

Specimens should be at least 2500 mm^2 in area, e.g. 50x50 mm or larger.
As an alternative to square specimens, the already tested (and broken) tensile specimens may be used.

Ash content is determined according method ISO 1172.

Result shall include average value and individual values, or average and standard deviation when the number of specimens tested is at least 5.

6 Density

Specimens should be taken close to the region where tensile specimens are taken, to ensure that the same material quality is tested. This is indicated in Figure 2 and Figure 4.
Specimens shall be taken from at least 2 different plates

Density is determined according method ISO 1183.

Result shall include average value and individual values, or average and standard deviation when the number of specimens tested is at least 5.

7 Other properties - optional tests

Other properties may be optionally measured.
They will not be described in detail here. However, to ensure some uniformity in testing methods, preferred methods are listed below.

Properties, that can be measured, if property is essential for final application

Material properties as delivered				
Property	**Standard**	**Specimen**	**Unit**	**Test conditions and special recommendations**
Oxidation Induction Time (OIT)	EN 13677	100 mm x 100 mm	min	time to mass loss > 2 % at 230 °C, annex F
Blank Size	EN 13677	blank as delivered	mm2	see annex D
Blank Weight	EN 13677	blank as delivered	g	see annex D
Flow Properties / Energy	DIN EN ISO 12115 or 12114		J	3 blanks 190 mm x 190 mm in centre of mould
Flow Properties / Pressure	DIN EN ISO 12115 or 12114		bar s	3 blanks 190 mm x 190 mm in centre of mould
Melting Temperature	ISO 3146		°C	10°C/min under nitrogen, second heating
Recrystallisation Temperature	ISO 3146		°C	10°C/min under nitrogen, after first heating

Material properties of moulded plaques; default thickness 2mm, optional 3 or 4 mm				
Property	**Standard**	**Specimen**	**Unit**	**Test conditions and special recommendations**
Flexural Modulus RT & 80°C	ISO 178	type II, 80 mm x 25 mm	MPa	5 mm/min, secant 0,05 - 0,25 %
Flexural Strain RT & 80°C	ISO 178	type II, 80 mm x 25 mm	%	5 mm/min
Flexural Strength RT & 80°C	ISO 178	type II, 80 mm x 25 mm	MPa	5 mm/min
Charpy Impact Strength	ISO 179/2fn	type II, 15mm width	kJ/m²	flatwise testing
Charpy Impact Strength -30°C	ISO 179/2fn	type II, 15mm width	kJ/m²	flatwise testing
FPI Energy at Fmax	ISO 6603	60 mm x 60 mm or round, d = 60 mm	J	4,4 m/s; measure and record thickness
Tensile Modulus 80°C	ISO 527/4	Injection moulding: ISO 527-1B; Compression moulding: ISO 527-4/Typ2 (250 (min. 200) mm x 25 mm)	MPa	5 mm/min
Tensile Strength 80°C	ISO 527/4	Injection moulding: ISO 527-1B; Compression moulding: ISO 527-4/Typ2 (250 (min. 200) mm x 25 mm)	MPa	5 mm/min
Elongation at Break 80°C	ISO 527/4	Injection moulding: ISO 527-1B; Compression moulding: ISO 527-4/Typ2 (250 (min. 200) mm x 25 mm)	%	5 mm/min
Compression Modulus	ISO 14126	as defined in norm		1 mm/min, secant 0,05 - 0,25 %
Compression Strength	ISO 14126	as defined in norm		1 mm/min
CTE normal / parallel	ISO 11359	as defined in norm	10-6 1/K	
Water Absorption	ISO 62/C		%	weight gain after 24 hours
LTHA	EN ISO 4577		h	
Burning Behaviour	ISO 3795		mm/min	identical to MVSS 302
HDT	ISO 75-2A		°C	testing 0° and 90°

Further comments on preferred optional tests:

Glass length
Glass length distribution can be measured in many ways, and is not standardized yet. It should be noted that at this moment of time, different laboratories will give very different average glass length results, mainly due to differences in taking a representative sample of glass fibres. Differences may easily vary a factor of 2!
A standardized method to overcome this problem is still to be developed.

Coefficient of linear thermal expansion, CLTE
On exactly the same locations as the 0 and 90° tensile bars, take 0 and 90° CLTE specimens. At least 2 specimens per direction. One specimen per plate.
Test method of CLTE in accordance with EATC norm proposal.
Record physical and technical CLTE as function of temperature (physical is CLTE value at a given temperature, technical is average CLTE between two temperatures, e.g. 23 and 80°C, usually used for simple calculations or datasheet values.)
Physical CLTE values at a given temperatures can be used together with 0, 45 and 90° tensile specimen results at that temperature to calculate an isotropic CLTE at that temperature. See method of calculating isotropic properties.

Flexural, Charpy, IZOD
All these properties are highly orientation dependent and also sensitive to variations in glass concentrations over the thickness of the sample. These variations in turn may be highly sensitive to variations in process conditions. As a result quite arbitrary values may be obtained when just taking specimens e.g. in two directions from moulded products.
Therefore it is not recommended to test these values for injection moulded materials. If still needed to do so, the same method as used for isotropic tensile properties should be used in obtaining isotropic values. And results are to be treated with care.

Flow properties
Flow properties, as necessary for e.g. a mould filling simulation are not treated in this norm yet.
In general a characterisation would include viscosity, pVT, thermal conductivity and thermal capacity measurements. Small size capillaries are not recommend for long fibre materials and for thermal conductivity fibre orientation and dispersion should be taken into account. Also it should be noted that especially viscosity is depending on fibre length.

Fibre orientation development
Parameters that define fibre orientation development as function of flow type and length are not treated in this document. Typically these parameters would include fibre interaction coefficients such as used in mould filling finite element simulations. The needed parameters can usually be obtained via the material suppliers.

Shrinkage
A shrinkage test standard is not specified in this document.
In general it applies that for anisotropic shrinkage determination, sufficient flow length should be available in a test plaque to yield a sufficient orientation level. (Length over width of plate should be higher than 3.) Shrinkage should be determined for various processing conditions and wall-thicknesses. Also here it applies that shrinkage is highly dependent on fibre length.
Near square plates, film-gate are not suitable for determining anisotropic shrinkage levels, as film gates will not change very much the initial orientation state towards a more aligned one.