Industrieverband
langlebige Kunststoffprodukte
und Mehrwegsysteme e.V.

pro-K Floropolymergroup

Technical brochure 04

Quality requirements and test guidelines for PTFE-products

Industrieverband
langlebige Kunststoffprodukte
und Mehrwegsysteme e.V.

Preamble

The completely fluorinated polymer PTFE is the most widely used fluoropolymer and based on its unique properties established as an undispensible construction material in modern industries.

The extraordinary properties of PTFE are due to its resistence to most chemicals, its broad service temperature range, the excelent electrical properties, the persisting to embrittlement, the ageing resistance and its high purity.

This technical brochure informs about the quality requirements and test conditions, which are necessary to assess semi – finished goods made from PTFE resins, which are essential for high quality PTFE products.

This brochure replaces in parts respectively augments the brochure „quality requirements, test guidelines and tolerances“ for PTFE products edited in 1993 by the „Gesamtverband Kunststoffverarbeitende Produkte (GKV).

This technical brochure is edited by the Fluoropolymergroup of pro-K and provides the present state of knowledge as by January 2020. It replaces the brochure from July 2014, September 2017 and March 2019.

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1. Field of application

These quality requirements and test conditions help to assess semi-finished goods made from PTFE. The requirements listed below define the minimum requirements that are necessary for high quality PTFE products.

In the practical experience you can sometimes find so called „mean values“ whose variations of deviance may be quite high. In such cases it is necessary to define limits for the requested tolerance to be able to compare physical properties.

The tests described below shall be carried out in an air-conditioned test room. The specifications as described in DIN EN ISO 20568-2 (Plastics - Fluoropolymer dispersions and moulding and extrusion materials) apply.

To determine the density, the prefabricated test pieces are preconditioned for at least 4 hours at a laboratory temperature of 23 °C ± 2 °C in accordance with ISO 291. The other tests do not require preconditioning. The density is determined at a laboratory temperature of 23 °C ± 2 °C.

Since fluoropolymers do not absorb moisture, it is not necessary to maintain a constant humidity during testing. A humidity monitoring of the test room is not obligatory. For tests on preconditioned samples in powder or pellet form, however, it must be ensured that no condensation of air humidity occurs on the surface of the powder or pellet particles.

The determination of the melt flow index and the crystalline melt peak temperature is performed under standard laboratory conditions.

2. Specific gravity

2.1 Test method

DIN EN ISO 1183-1 Plastics - Methods for determining the density of non-cellular plastics - Part 1: Immersion method, liquid pycnometer method and titration method with the following requirements:

- Test specimen as compact as possible, mass preferably 1 g to 50 g
- Balance accuracy for test specimens:
  a) 0,1 mg at ≤ 10 g
  b) 1 mg at > 10 g
- Use of a suitable liquid: preferably a PTFE wetting liquid or distilled water with no more than 0.1 % of a wetting agent
- Temperature of the immersion liquid 23 ± 2 °C
- Determination on at least 3 test specimens with output of the mean value with a maximum of 3 decimal places in g/cm³

2.2 Minimum and maximum requirements

specific gravity for unfilled PTFE and modified PTFE: 2,12 - 2,20 g/cm^3

2.3 Comments

Semi-finished goods made from low molecular PTFE show a higher specific gravity than those made from PTFE of higher molecular weight. This means that the influence of the used resin has to be taken into account when the PTFE-resin is evaluated.
Low specific gravity indicates a low crystalline and hence flexible material. 2,12 g/cm^3 define low compression and a high porosity.
High specific gravity is typical for a high crystalline and hence stiff material. Values above the tolerance indicate changed physical properties, that may be caused either by the used resin or from “over-sintering“ which may be due to a thermal degradation.

Compounds which contain glass, graphite, carbon or bronze show specific gravities that are defined by the used filler.

3. Tensile strength and elongation at break

3.1 Test method

DIN EN ISO 527-1:1996-04 Plastics – determination of tensile strength - part 1: general principles

The determination of the tensile strength according to the above mentioned ISO standard is used to evaluate the behaviour of fluoropolymers during elongation in one dimension. The test is performed on specific specimen under defined conditions for the preparation, the test climate and the speed of elongation. This test method is mainly used for quality control purposes using the following parameters:

- Use of the SPI standard FD-105 (Fig. 1) as a test specimen
- Test speed: 50 mm/min
- Thick specimens: 0.5 - 3 mm; preferably 1 mm for PTFE and 1.5 mm for PTFE compounds
- clamping length: 22 mm
- It is recommended to use an extensometer with a measuring length of 10 mm
- A 500 N size load cell is recommended
- It is recommended to apply a preload to determine the zero point according toTable 1.

Tabelle 1 preload at different temperatures for PTFE and PTFE compounds

test temperature [°C]	preload [N/mm²]	preload for FD-105 1 mm thickness [N]	preload for FD-105 1,5 mm thickness [N]
23	0,4	2	3
38	0,3	1,5	2,25
100	0,15	0,75	1,13
150	0,10	0,50	0,75
200	0,05	0,25	0,38
250	0,05	0,25	0,38

The material flow have not be hindered within the clamping length. The use of an extensometer results in deviations for the elongation at break compared to testing without an extensometer (traverse measurement). The elongation at break is usually higher for PTFE compounds when using an extensometer, depending on the compound. This is taken into account in the overview in chapter 7, table 3, by a correction factor elongation at break k_r.
When exceeding the above mentioned range of thickness of test specimen, the test specimen heats up significantly during the tensile test and thus leads to a incorrect, reduced tensile strength.

The terms tear strength and elongation at break common in PTFE processors are in agreement with the terms breaking stress and elongation at break used in DIN EN ISO 527-1.

A separate test program must be used to determine the modulus of elasticity. At the beginning of the stress-strain curve in the range 0 - 0.1 % strain, which is used to determine the modulus of elasticity, the test speed is 1 mm/min and is increased to 50 mm/min in the further course.

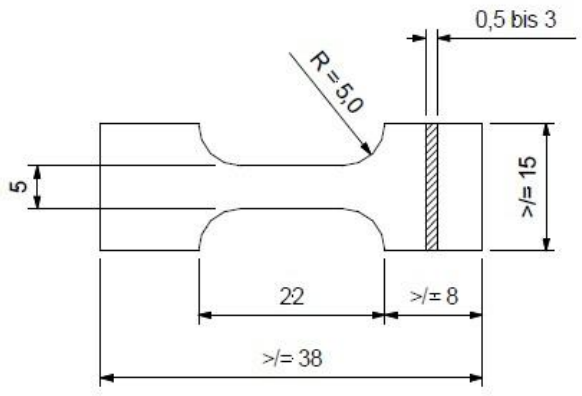

Figure 1: Specimen according to SPI-Standard FD-105.

3.2 Mechanical machining of test specimen

The requirements of DIN EN ISO 2818: 1996, plastics - production of test specimens by mechanical processing apply. In particular, if there are transverse grooves on the surface of the test specimen, lower values for tensile strength and elongation at break must be expected due to the notch sensitivity of the PTFE.

3.3. Minimum requirements

Table 2 Minimum values for PTFE products

	films plates moulded PTFE		**non pre sintered** PTFE-powder		**Ram-extruded PTFE** **Pre-sintered** PTFE-powder		**Repro PTFE**		**Paste extruded PTFE**	
	non free flowing PTFE powder	**free flowing** PTFE granules	extrusion direction		extrusion direction		extrusion direction		extrusion direction	
			along	across	along	across	along	across	along	across
Tensile strength [N/mm²]	28,5	23,0	22,0	25,5	19,0	20,0	11,0	13,0	-	26,0
Elongation at break [%]	300	260	230	265	190	210	90	110	-	275

4. Ball hardness and Hardness Shore D

4.1 Test methods

Although the hardness Shore D is used as a widely used quality criterion, due to the very pointed indenter, this measuring method does not do justice to the relatively soft PTFE. The so-called ball indentation hardness, which is determined by pressing a spherical measuring body into the surface of the material, is much more meaningful about the product quality. It is therefore recommended to use the ball indentation hardness as a standard method for determining hardness on PTFE and PTFE compound components. The average must be given in each case.

4.1.1 Ball hardness (according to DIN EN ISO 2039-1)

- Sample thickness: ≥ 4 mm
- Distance to the edge: ≥ 10 mm
- Distance impressions: ≥ 10 mm
- Test time: 30 seconds
- required valid measurements: 10

4.1.2 Hardness Shore D (according to DIN EN ISO 868:2003)

- Sample thickness: ≥ 4 mm
- Distance to the edge: ≥ 12 mm
- Distance indentation: ≥ 6 mm
- Test time: 15 Sek.
- required valid measurements: 5

4.2 Typical Values

Ball hardness:	28 N/mm^2	(values only valid for un layered samples)
Hardness Shore D:	54	

5. Voids

5.1 Test method

5.1.1 Stabilized DC-voltage*

The testing is performed with a suitable device for detecting voids with stabilized DC-voltage. For this purpose pro-K re-commends to use brush like electrodes. The test voltage depends on the thickness of the specimen.
For thicknesses between 0,4 mm and 4,2 mm it should be calculated according to the below algorithm: Thickness (in mm) time 2,5 kV plus a bias of 1,5 kV.

*In agreement with the user the test can be performed also with AC-voltage.

Please note: For a thickness of 0,4 mm the testing voltage would account to 1,5 kV + 0,4 x 2,5 kV = 2,5 kV, for 1mm thickness accordingly 4,0 kV. See also VDE-standards and ATEX-guidelines.

5.1.2 Indicator for voids

To detect voids and/or cracks the complete surface is cleaned respectively decreased with a cleaning agent. After drying the surface is treated with a commercial penetrating colour by spraying or dipping. After 5 minutes this substance is removed by wiping or rinsing. As soon as the surface is dry it is detected for voids or cracks.

5.2 Maximum limits

$$\text{max. Voids per m}^2 = \frac{1{,}0}{\text{thickness (mm)} \times 2}$$

(Partial values are rounded up to full numbers.)

Note: The minimum thickness of foils should not be less than the average grain diameter of the PTFE pressed powder used.

6. Dielectric strength, volume resistivity and surface resistivity

6.1 Test method

DIN EN ISO 12086
Plastics - Fluoropolymer dispersions and moulding and extrusion materials.
Part 2: Subclauses 8.1.1, 8.1.2 and 8.1.3

IEC 60093
Test method for electric insolator, volume resistivity and surface resistivity of solid, electrically isolating construction materials.

The test results shall contain the shape of the used electrodes.

The dielectric strength shall additionally contain the thickness of the test specimen used for the measurement.

Due to the excellent insolating properties of PTFE products the dielectric strength used to be very high. To avoid ambiance the tests shall be performed with specimen of a thickness >0,5 mm in a halocarbon medium.

6.2 Minimum Requirements

Minimum 50 kV/mm (measured with a specimen thickness of minimum 0,5 mm).

Note: The value of dielectric strength decreases significantly with increasing thickness of the film.

7. Semi-finished goods made of filled PTFE (PTFE Compounds)

In addition to sections 2. (specific gravity), 3. (tensile strength and elongation at break) and 4. (ball hardness and hardness Shore D), the following applies to semi-finished products made of PTFE and filled PTFE (PTFE compounds) for tensile strength and elongation at break etc. Minimum values. Table 3 shows mean values for the ball indentation hardness and the hardness Shore D. For reasons of comparability, only free-flowing material is listed in the table below.
The data for elongation at break refer to a test without an extensometer (so-called traverse measurement), determined on semi-finished products created under laboratory conditions. For products where the tensile yield strength is higher than the tensile fracture strength, the value for the tensile fracture strength was always used in Table 3, see also DIN EN ISO 527-1.

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Table 3: Minimum requirements for semi finished parts of PTFE and PTFE-Compounds

				Compression moulded parts		**Ram-extruded parts**				
Property [Dimension] Test method	**Specific gravity [g/cm³]** DIN EN ISO 1183-1	**Deformation under load 15N/mm² 100h [%]** corresponds to. pro-K*		**Tensile strength [MPa]** DIN EN ISO 527	**Elongation at break [%]** DIN EN ISO 527	**Tensile strength [MPa]** DIN EN ISO 527	**Elongation at break [%]** DIN EN ISO 527	**Ball hardness MW ± 6 [N/mm²]** DIN EN ISO 2039	**Shore D** MW ± 5** DIN EN ISO 868	**Correction factor elongation at break k_r for measurements with extensometer**
Test temperature [°C]		**23**	**100**							
PTFE virgin	2,16 ± 0,04	18	33	23	260	19	190	28	54	0,96
PTFE modified virgin	2,16 ± 0,04	9	23	26	360	20	300	29	54	0,96
PTFE + 10 % carbon	2,14 ± 0,04	14	19	22	220	19	170	32	57	1,11
PTFE + 15 % carbon	2,13 ± 0,04	10	14	20	200	16	140	34	59	1,14
PTFE + 25 % carbon	2,10 ± 0,04	8	22	13	60	11	80	41	61	1,81
PTFE modified + 25 % carbon	2,10 ± 0,04	4	-	12	30	11	35	43	61	1,94
PTFE + 33 % carbon	2,08 ± 0,06	6	15	9	40	6	20	47	63	1,65
PTFE modified+ 33 % carbon	2,08 ± 0,06	3	-	10	8	6	15	53	60	1,33
PTFE + 15 % graphite	2,16 ± 0,04	11	-	16	130	13	110	35	58	1,27
PTFE + 10 % glass	2,19 ± 0,04	19	26	23	220	16	200	30	55	1,11
PTFE + 15 % glass	2,21 ± 0,04	14	30	19	200	19	190	33	57	1,13
PTFE + 20 % glass	2,22 ± 0,04	16	21	15	180	15	170	34	57	1,16
PTFE + 25 % glass	2,23 ± 0,04	12	30	14	160	14	150	39	57	1,25
PTFE modified + 25 % glass	2,23 ± 0,04	6	-	12	280	18	200	35	56	1,07
PTFE + 40 % bronze	3,10 ± 0,10	13	26	23	190	20	180	35	60	1,24
PTFE + 60 % bronze	3,85 ± 0,15	8	20	14	80	13	100	44	63	1,73
PTFE + 55 % bronze + 5 % MoS_2	3,85 ± 0,15	-	-	12	50	15	100	44	62	2,09
PTFE + 50 % stainless steel	3,35 ± 0,10	4	-	13	130	15	100	42	61	1,62
PTFE + 20 % PEEK	1,92 ± 0,04	5	-	13	50	11	80	43	61	1,95
PTFE + 10 % aromatic Polyester	2,07 ± 0,04	11	-	18	230	14	150	34	55	1,14
PTFE + 20 % aromatic Polyester	1,95 ± 0,04	-	-	14	160	13	120	38	56	1,30
PTFE + 10 % carbon fibre	2,09 ± 0,04	-	-	18	180	19	210	31	58	1,16
PTFE + 20 % carbon fibre	1,98 ± 0,04	-	-	10	70	13	120	32	59	1,48
PTFE + 7 % Polyimide	2,06 ± 0,04	-	-	22	250	20	240	30	57	1,11
PTFE + 10 % Polyimide	2,03 ± 0,04	-	-	16	220	15	150	35	56	1,05

* according to ASTM D 621 the remaining deformation is detected after 100 h under load without relaxation time.

** test time: 15 s. If test time is reduced to 3 s: the value of Shore D hardness equals the listed value plus 2

8. Detection of Melting Point, Crystallinity and filler content for PTFE and PTFE-compounds

8.1 Differential scanning calorimetry (DSC)

The DSC analysis of PTFE compounds is based on ISO 11357-3: 1999: Plastics differential scanning calorimetry. Recommended amount of sample: 5 - 10 mg. The investigation begins under a nitrogen inert gas atmosphere.

In the DSC method, the sample is first heated to 390 ° C in a first heating process, PTFE is charactericed by melting point at approx. 327 °C (sintered PTFE) or 345 °C (unsintered PTFE) The temperature range of the melting curve (°C) and the melting enthalpy (J/g) are determined. Special features of the product or the sintering during processing are recognized. The sample is then cooled with a defined cooling rate below the crystallite melting point. During the second melting, permanent product changes, e.g. due to oversintering, can be detected.

To reduce the analysis time required, the temperature program can be run at a higher heating and cooling rate, e.g. 20 K/min. This procedure results in a systematic shift of the measured values.

8.2 Thermal Gravimetric Analysis

The following method describes one of the most common methods for determining the filler content in PTFE compounds based on e.g. glass, coal, graphite or metals.

It is recommended to use the TGA method (**TGA** = **T**hermal **G**ravimetric **A**nalysis) to determine the filler content using inert and oxidizing atmospheres. The TGA method is typically used following a DSC analysis (**DSC** = **D**ifferential **S**canning **C**alorimetry).

During the second melting, the heating is continued up to 650 °C, whereby both PTFE and thermally degradeable fillers can be broken down.

The PTFE content and the proportions of the thermally non-oxidatively degradable fillers are determined on the basis of the determined temperature loss. The changeover to oxygen atmosphere takes place at 650 °C. The mixture is then heated to 850 °C. The proportion of oxidatively degradable filler components is determined on the basis of the further weight loss.
Oxidizable, thermally non-degradable fillers, e.g. bronzes, can be recognized by the oxidation-related increase in weight.

Please perform these measurements only under appropriate safety precautions like an efficient exhaustion.

8.3 Sample for temperature regime

Example of performing a DSC analysis separately:

- 1st heating 30 °C bis 390 °C (10 K/min)
- Isothermal segment 390 °C (5 min)
- Cooling 390 °C bis 150 °C (10 K/min)
- Isothermal segment (5 min)
- 2nd heating 150 °C bis 390 °C (10 K/min)

Example of performing a TGA analysis separately:

- Heating from 30 °C bis 650 °C (10 K/min) under nitrogen atmosphere
- Optional isothermal segment 650 °C (5 min)
- Switch to oxygen O_2, heating 650 °C bis 850 °C (10 K/min)

The test can also be carried out on a combination device.
Example of a combined DSC/TGA program:

- 1st heating 30 °C bis 390 °C (10 K/min)
- Isothermal segment 390 °C (5 min)
- Cooling 390 °C bis 150 °C (10 K/min)
- Isothermal segment (5 min)
- 2nd heating 150 °C bis 650 °C (10 K/min)
- Switch to oxygen O_2, heating 650 °C bis 850 °C (10 K/min)

9. Deformation under load

The determination of the deformation under load is not part of the general quality control. Therefore a special agreement between supplier and customer is necessary.

9.1 Test method

A cylindrical specimen with the following dimensions (diameter 10 mm, height 10 mm), which is free of internal stress, is fixed in the test-device (see figure 2) which is in the test-chamber. Please make sure the thickness is not above 10 mm. The specimen, which has been conditioned at normal climate (ASTM D621/64), has to be fixed between the both pressure pistons. Be sure the lateral face is plane. At the normal climate the test-pressure accounts to 15 ± 0,5 N/mm^2.
The deformation is measured at (23 ± 2) °C and at (100 ± 2) °C; different temperatures have to be recorded.

The determination has to be done after 100 h under load and subsequent 24 h of relaxation.

9.2 Analysis

The deformation under load is calculated as follows:

$$\frac{A - B}{A} \text{ X } 100 \text{ [\%]}$$

Please not (exactness amounts to 0,01 mm):
A = original height of specimen
B = height of test specimen after test cycles

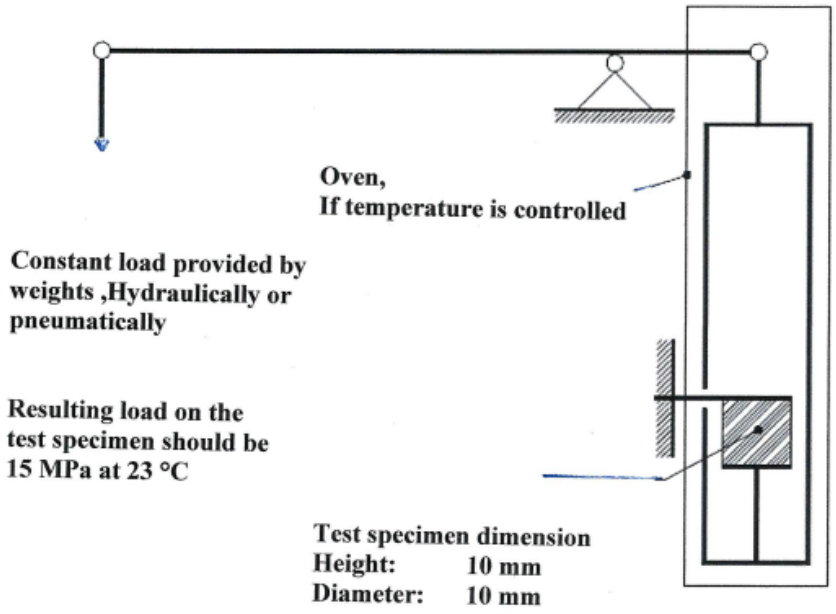

Fig. 2: Test device for measurement of deformation under load (schematic design).

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