Industrieverband
Halbzeuge und Konsumprodukte
aus Kunststoff e.V.

pro-K Floropolymergroup

Technical brochure 04

Quality requirements and test guidelines for PTFE-products

March 2013

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 March 2013.

Important note

March 2013

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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 have to be performed at a so called „normal climate“¸ this means at a temperature of 23 °C
(± 2°C), and an air humidity of 50 % (± 10%) (according to DIN EN ISO 291 "Plastics –normal climate for conditioning and testing").

These requirements are valid including chapter 6 for all products made of pure/unfilled PTFE.

2. Standard Specific gravity

2.1 Test method

DIN EN ISO 12086 Plastics - Fluorpolymerdispersions, and moulding and extrusion materials - part 2: Preparation of test specimen and determination of properties (Subclause 8.4).

2.2 Requirements

Figures: 2,12 - 2,20 g/cm^3

2.3 Comment

Semi-finished goods made from low molecular PTFE show a 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-product is evaluated.

Low specific gravity indicates a low crystalline and hence flexible material. Figures below 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

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 at break according to the above mentioned ISO standard is used to evaluate the behaviour of fluoropolymers during elongation in one axis. 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 recommended specimen parameters:

Thickness: 1,0 mm for PTFE und 1,5 mm for Compounds.
Elongation speed: 50 mm/min
Specimen for the testing of skived films and plates :

For skived films of 0,5 to 3 mm thickness: specimen according or similar to SPI-Standard FD-105 (picture 1).

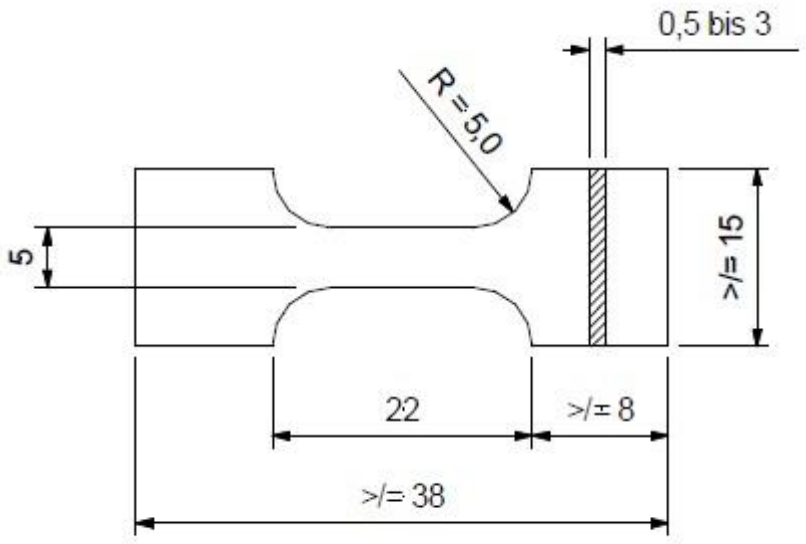


Figure 1: Specimen according to SPI-Standard FD-105.

For skived films 0,5 to 3 mm thickness: Specimen according to Standard FD-105 (picture 1) or DIN EN ISO 527-2 (figure 2).

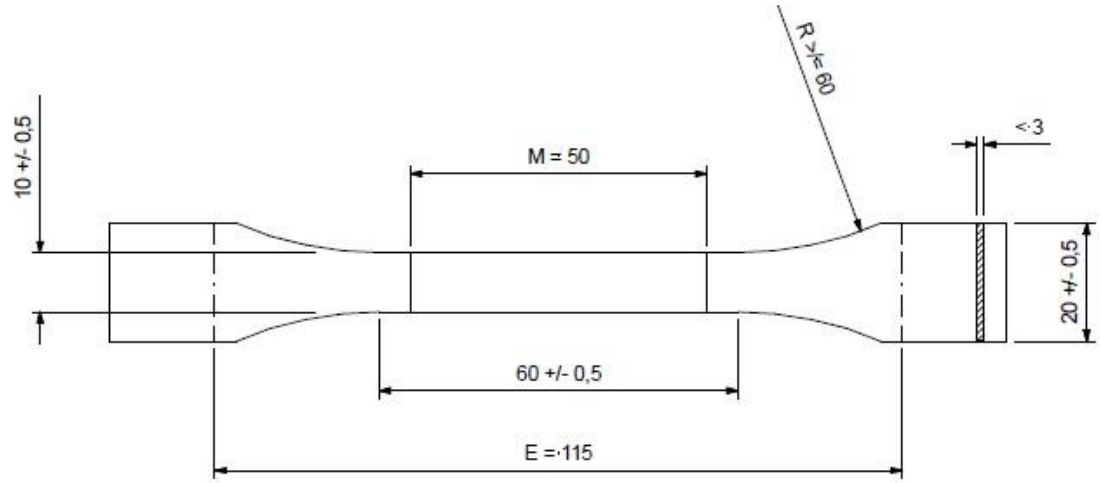


Figure 2: specimen 1B according to DIN EN ISO 527-2.
M = length of measurement
E = length in the test device

3.1.1 Machining of test specimen

Please use the specifications of DIN EN ISO 2818:1996, Plastics – Preparation of test specimen by machining.

3.2 Tensile strength and elongation – Minimum requirements

Note: pro-K Fluoropolymergroup recommends to use the following thickness for specimen :
1,0 mm for PTFE and 1,5 mm for Compounds.

	Films and plates moulded PTFE		Ram-extruded PTFE						Paste extruded PTFE (measured Across to Extrusion direction)
			virgin PTFE-powder		Pre-sintered PTFE-powder		Regrind		
	free flowing granules	non free flowing powder	extrusion direction		extrusion direction		extrusion direction		
			along	across	along	across	along	across	
Tensile strength N/mm²	23,0	28,5	22,0	25,5	19,0	20,0	11,0	13,0	26,0
Elongation %	260	300	230	265	190	210	90	110	275

4. Ball hardness H 132/30 and Shore-hardness D

4.1 Test method

Ball indentation hardness **according to** ISO 2039
Thickness of specimen: minimum 4 mm; distance to edge above 10 mm. test time: 30 sec.

Shore-hardness D according to DIN 53505:2000-08

Determination of plastics - test of hardness according to Shore D
Thickness of specimen: minimum 6 mm. test time: 3 sec.

Requirement: The following values are valid only for unlaminated samples.
Thickness of specimen: minimum 4 mm
Distance to edge : above 10 mm
Ball hardness: minimum 22,0 N/mm^2
Shore-hardness D: minimum 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 Requirement (based on experience):

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

(please use only complete figures)

5.1.3 Please note

The thickness of films should not be less than the average particle size of the used resin.

5.1.4 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.

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 Requirement

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

6.3 Please note

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

7. Semi-finished goods made of filled PTFE

For semi-finished goods made of filled PTFE the following minimum parameters are required in addition to chapter
2. (specific gravity), 3. (tensile and elongation) and 4. (ball indentation hardness). The results for carbon compounds refer to a filler called „soft carbon“. For reasons of comparison the following table only contains results made of free flowing resins.

				Moulded semi finished goods		Ram extruded semi finished goods			
	Standard Specific gravity	**Deformation under load 15 N/mm² 100h**		**Tensile strength**	**Elongation**	**Tensile strength**	**Elongation-**	**Ball-indentation hardnesse**	**Shore D hardness**
Test method	**DIN 53479**	**equivalent pro-K***		**DIN EN ISO 527**	**DIN EN ISO 527**	**DIN EN ISO 527**	**DIN EN ISO 527**	**ISO 2039**	**DIN 53505**
Dimension	**[g/cm³]**	**[%]**	**[%]**	**[N/mm²]**	**[%]**	**[N/mm²]**	**[%]**	**[N/mm²]**	
		23° C	**100° C**						
PTFE virgin	2,16 ± 0,04	18	33	23	260	19	190	22	54
PTFE modified virgin	2,16 ± 0,04	9	23	22	360	18	300	23	56
PTFE + 10 % carbon	2,14 ± 0,04	14	19	16	180	14	160	26	61
PTFE + 15 % carbon	2,13 ± 0,04	10	14	14	150	12	130	27	62
PTFE + 25 % carbon	2,09 ± 0,04	8	22	13	100	11	90	34	63
PTFE modified + 25 % carbon	2,09 ± 0,04	4	-	9	45	8	35	34	63
PTFE + 33 % carbon	2,09 ± 0,04	6	15	9	25	6	15	35	65
PTFE modified + 33 % carbon	2,09 ± 0,04	3	-	7	5	6	15	35	65
PTFE + 15 % graphite	2,16 ± 0,04	11	-	20	200	16	120	-	63
PTFE + 10 % glass	2,19 ± 0,04	19	26	17	210	16	200	23	57
PTFE + 15 % glass	2,21 ± 0,04	14	30	15	200	14	180	25	58
PTFE + 20 % glass	2,22 ± 0,04	16	21	14	180	12	160	26	58
PTFE + 25 % glass	2,23 ± 0,04	12	30	14	160	11	140	27	59
PTFE modified + 25 % glass	2,23 ± 0,04	6	-	16	220	18	200	-	59/54
PTFE + 40 % bronze	3,10 ± 0,10	13	26	13	150	10,5	140	27	63
PTFE + 60 % bronze	3,85 ± 0,15	8	20	12	120	9,5	100	30	65
PTFE + 55 % bronze + 5 % MoS_2	3,85 ± 0,15	-	-	14	55	-	-	-	72/68
PTFE + 50 % stainless steel	3,35 ± 0,10	4	-	16	200	20	200	-	69/65
PTFE + 20 % PEEK	1,92 ± 0,04	5	-	18	200	16	180	-	68/62
PTFE + 10 % aromatic Polyester	2,07 ± 0,04	11	-	24	340	20	270	-	63/57
PTFE + 20 % aromatic Polyester	1,95 ± 0,04	-	-	18	200	-	-	-	64/60
PTFE + 10 % carbon fibrer	2,09 ± 0,04	-	-	22	260	23	250	-	65/60
PTFE + 20 % carbon fibrer	1,98 ± 0,04	-	-	14	140	14	140	-	67/63
PTFE + 7 % PI	2,06 ± 0,04	-	-	22	250	26	280	-	60/54
PTFE + 10 % PI	2,03 ± 0,04	-	-	16	250	-	-	-	68/60

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

8. Detection of filler content in PTFE-compounds

The following test method describes one of the most common procedures to determine the filling content of PTFE-compounds made of glass, carbon, graphite or metals
It is recommended to use the TGA-method (**TGA** = **T**hermal **G**ravimetric **A**nalysis) applying inert or oxydizing atmosphere.
The use of the TGA-method typically applies after a DSC-analysis (**DSC** = **D**ifferential **S**canning **C**alorimetry).
The performance of the DSC-Analysis of PTFE-compounds applies according to ISO 11357-3:1999: (Plastics differential scanning calorimetry).

Recommended sample size: 5 – 10 mg.
The analysis starts under a Nitrogen inert atmosphere.

When using the DSC-method the specimen is first heated to 390 °C. Doing so the melting behaviour at about 327 °C (sintered PTFE) resp. 345 °C (unsintered PTFE) characterizes the base resin. By this the melting tmeperature and the melting enthalpy (J/g) are determined. During this procedure the specific behaviour of the resin respectively of the sintering process can be recognized.
Afterwards the sample is cooled down at a defined cooling-rate below the melting point. Melting the sample a second time provides information about remaining changes of the product properties as they occur by oversintering.
When melting the sample the second time the temperature is increased up to 650 °C which causes the PTFE and some fillers to decompose.
Based on the detected weight loss the amount of PTFE and the amount of thermally non oxidizable fillers is determined.
Starting from 650 °C the atmosphere is switched to oxygen heating up to 850 °C. Based on the detected weight lossfound now the amount of oxydizable fillers is determined.
Oxidizable , thermically not decomposable fillers, e.g. bronze, can be recognized by an increase in weight due to oxydation.

Example for a combined DSC/TGA- procedure:

1. 1. Heating: 30 °C - >390 °C (10 K/min)
2. Isothermic segment 390 °C (5 min)
3. Cooling 390 °C - >150 °C (5 K/min)
4. Isothermic segment (5 min)
5. 2. Heating 150 °C - >650 °C (10 K/min)
6. Switch to O_2,-atmosphere heating 650 °C - >850 °C (10 K/min)

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

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 4) 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 (see chapter 1, 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. In case the temperature is different please correct the pressure appropriately.
The deformation is measured at (23 ± 2) °C and at (100 ± 2) °C; different temperatures have to be recorded.

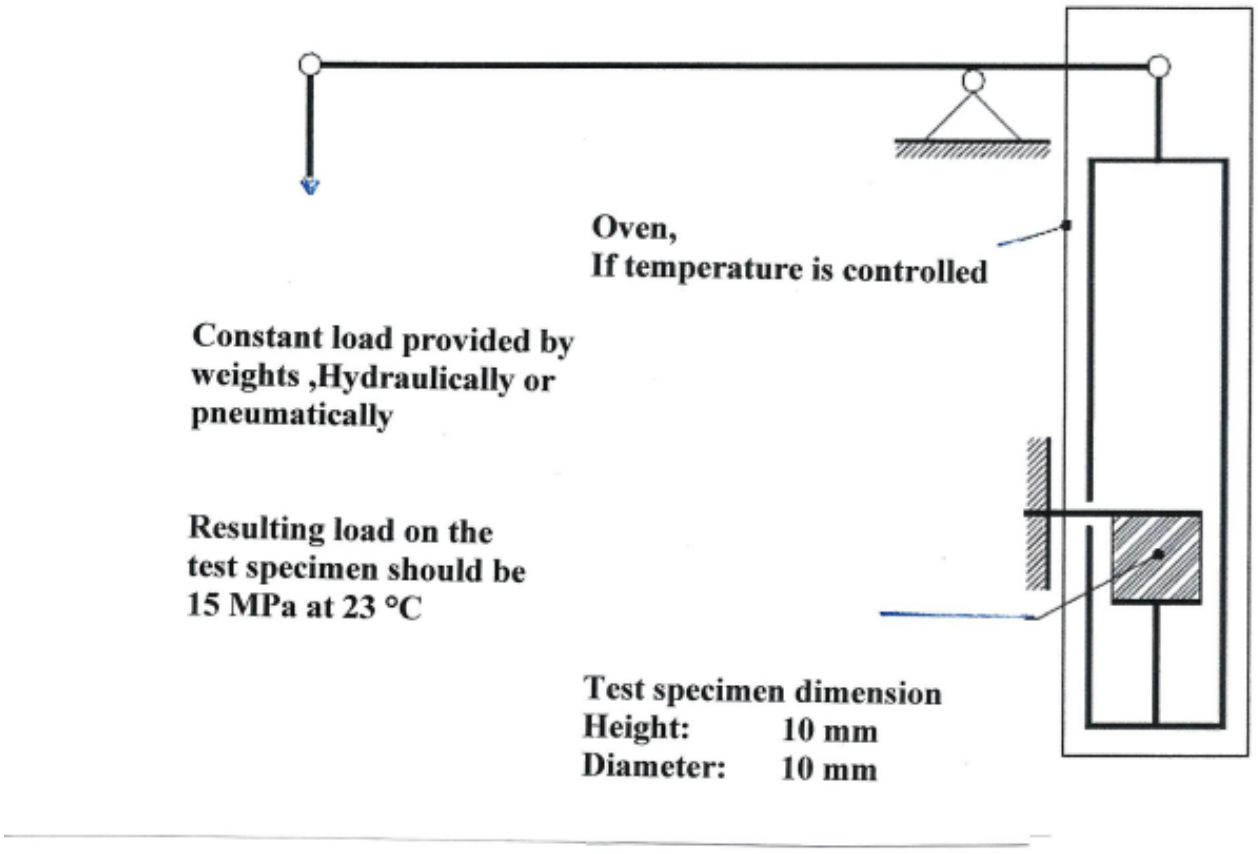
The determination can be done after 100 h under load or at 100h under load and subsequent 24h of relaxation.

9.2 Analysis

The deformation under load is calculated as follows:

$$\frac{B-A}{B} \quad X\ 100\ (\%)$$

Please not the exactness amounts to 1/100 mm:
A = Height of test specimen after test cycles
B = original height of specimen



Picture 4: Test device for the determination of deformation under load.

The following companies contributed to this brochure:

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