Introduction and Applications

of

Speciality Fluoropolymers

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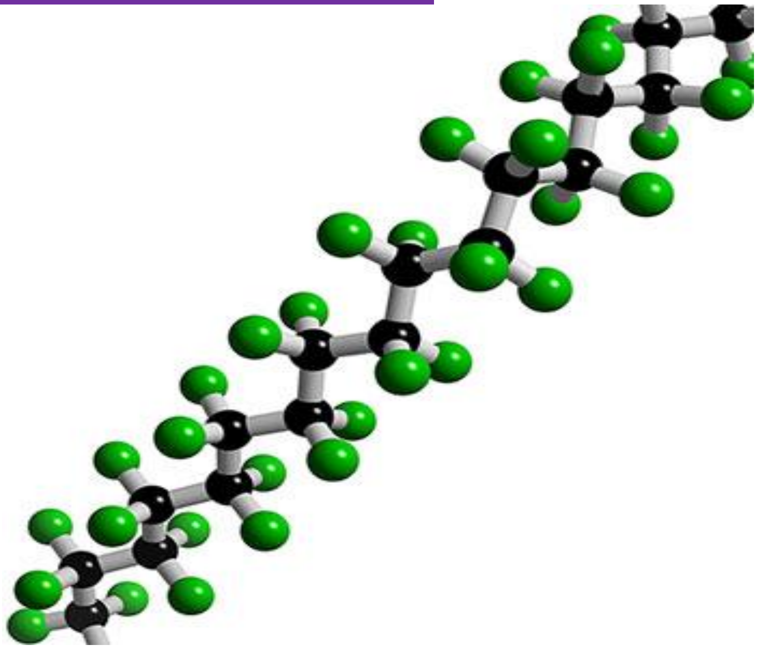
3M India Ltd.



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- **Synthesis of TFE Monomer**
- **Polymerization Techniques**
- **Processing Technique for fluoro polymers**
- **Properties of Fluoropolymers**
- **Applications of Fluoropolymers**
- **3M Fluoro polymer grades / FDA status / Specialty of Modified PTFE (i.e., 3M Dyneon TFM)**

Synthesis of TFE Monomer

HF preparation:

$$CaF_2 + H_2SO_4 \rightarrow 2HF + CaSO_4$$

Chloroform preparation:

$$CH_4 + 3Cl_2 \rightarrow CHCl_3 + 3HCl$$

Chlorodifluoromethane preparation:

$$CHCl_3 + 2HF \rightarrow CHClF_2 + 2HCl$$

TFE Synthesis:

$$2CHClF_2 \rightarrow CF_2{=}CF_2\text{(TFE)} + 2HCl$$

Other Monomers:

$CF_2{=}CFOC_3F_7$(PPVE), $CF_2{=}CFOCF_3$(PMVE)

$CF_2{=}CF{-}CF_3$(HFP), $CH_2{=}CF_2$(VDF), $CH_2{=}CH_2$(ETHY.)

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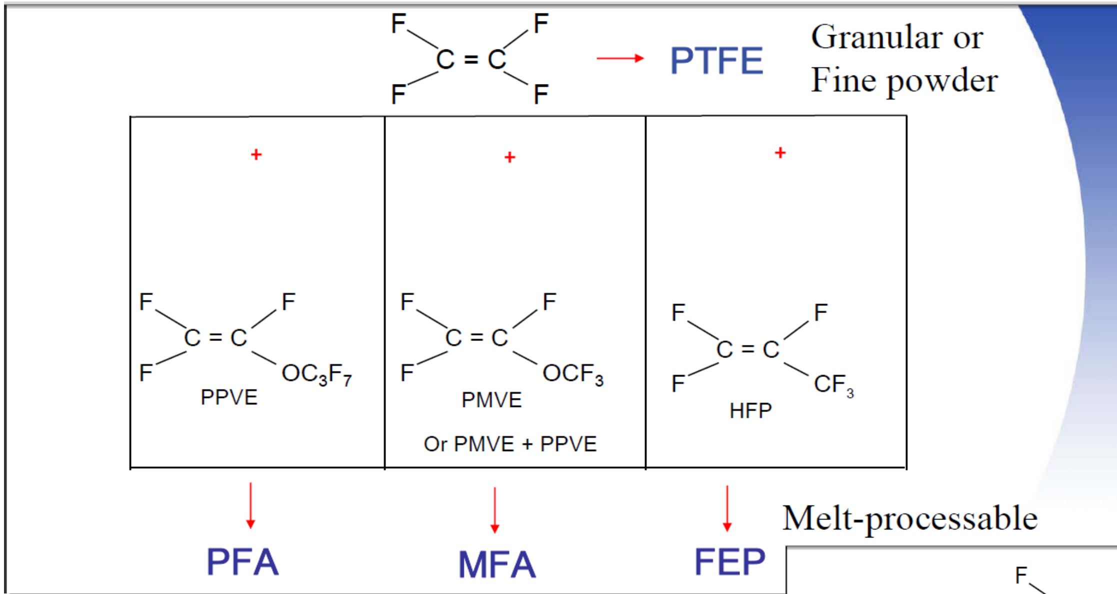
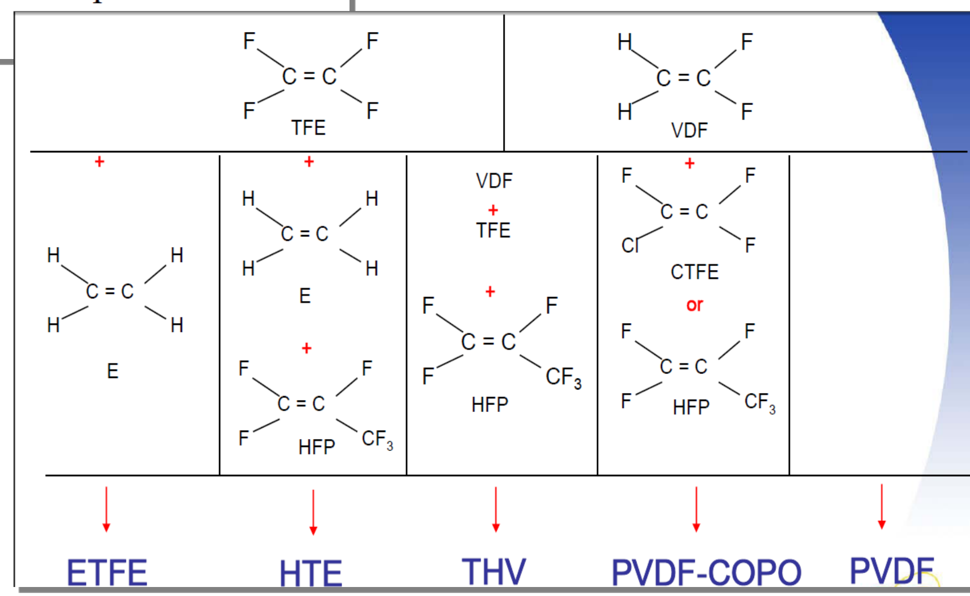
Polymerization Techniques

- Suspension polymerization
  - Monomer, Initiator, Medium
  - Product in stringy and irregular shape
  - Converting into Free flow or fine cut

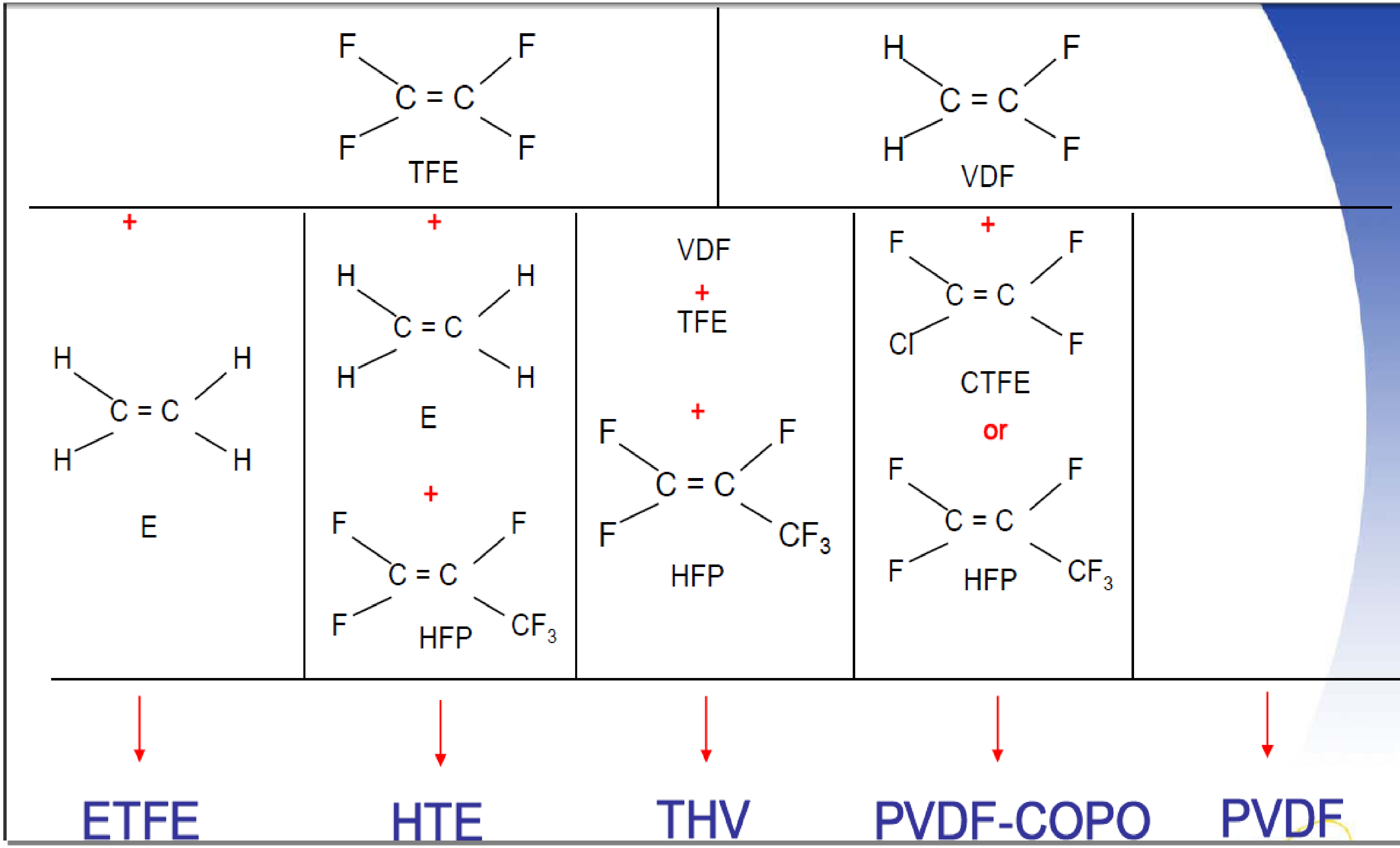
- Dispersion polymerization
  - Monomer, Initiator, Surfactant and Medium
  - Product in Fine Powder or Thick liquid

Chemical structure and copolymer





Chemical structure and copolymer



Processing Techniques for Fluoropolymers

- **Non meltable PTFE (Suspension grade):**
  - **Preform formation i.e., billet moulding**
  - **Sintering and skiving**
  - **Ram extrusion**

- **E-PTFE (Dispersion grade):**
  - **Paste Extrusion and/or spray coating**

- **Isostatic moulding and Automatic moulding**

- **Melt processable Fluoropolymers:**
  (FEP, PFA, ETFE, PVDF,THE,THV)

  - **Extrusion, Injection molding, Transfer moulding**

Fluoro polymers factsheet

POLYMER	MONOMER UNIT	MELT POINT °C[1]	MELT VISCOSITY, Pa.Sec.[2]	MELT FLOW RATE, dg/min.	FABRICATION TECHNIQUE
Granular PTFE	$-CF_2-CF_2-$	327	$10^{10}-10^{12}$ (3)		Compression Molding
Fine Powder PTFE	$-CF_2-CF_2-$	327	$10^{10}-10^{12}$ (3)		Paste Extrusion
Dispersion PTFE	$-CF_2-CF_2-$	317-337	$10^{10}-10^{12}$ (3)		Coating Methods
FEP	$-CF_2-CF_2-C(CF_3)F-CF_2-$	260-282	$10^{4}-10^{5}$ (4)	0.8-18 (5)	Melt Processing
PFA	$-CF_2-CF_2\ C(O-R_f)F-CF_2-$	302-310	$4\times10^{3}-3\times10^{4}$ (4)	1-18 (6)	Melt Processing
ETFE	$-CF_2-CF_2-CH_2-CH_2$	254-279	$0.7-10\times10^{3}$ (7)	3.7-16 (8)	Melt Processing
PCTFE	$-CClF-CF_2-$	210-215	1-10 (9)		Compression Molding/ Melt Processing
ECTFE	$-CClF-CF_2-CH_2-CH_2-$	240	-	1-50	Melt Processing
PVDF	$-CF_2-CH_2-$	155-192	$0.2-17\times10^{3}$	5-180 (10)	Melt Processing
PVF	$-CHF-CH_2-$	190	-		Melt Extrusion of Dispersion in Latent Solvent
Soluble Fluoropolymers	$-[CF_2-CF_2]_{n-m}-[C_f]_m$ (11)	305	-	-	Latex Application Methods

Properties of PTFE & Fluoroplastics

- Excellent chemical resistance against solvents, chemicals and even corrosive agents like acids, bases, cleaning solutions, oxidizing agents at high temperatures and pressures
- Superior resistance to damage and changes in performances by thermal shock during temperature cycling over a wide range
- Low dielectric constant which remains almost constant with wide range of frequency and temperature change
- Unusual stability against weathering and fire
- Resistance to property changes with time & temperature
- Low friction and self-lubricity
- Non-stick property
- Excellent sealing property
- Most fluoro polymers do not require additives to enhance performance, and therefore offer a clean surface without any contamination to the contacting liquids or other materials
- Due to low surface energy and smooth surface finish in comparison to alternate material solutions, PTFE and /or Fluoroplastics are best material suited for easy cleanability of Bio Film inside the liners. Due To low surface energy water will not stick to surface and will prevents / slow down the growth of Bio film inside the liner.

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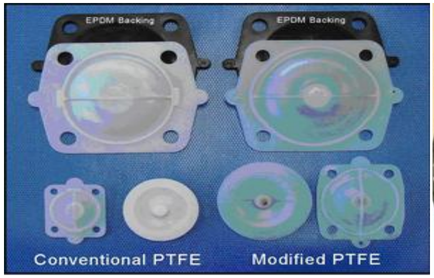
Applications of Fluoropolymers in CPI

- **Chemical Industry:**
  -Lining for vessels, columns, pipes, pumps, valves
  -Seals, O-rings, gaskets, packing materials for mechanical seals and other rotating parts
  -Internal components for fractionating columns and other equipment
  -Heat exchangers, Hoses and expansion joints

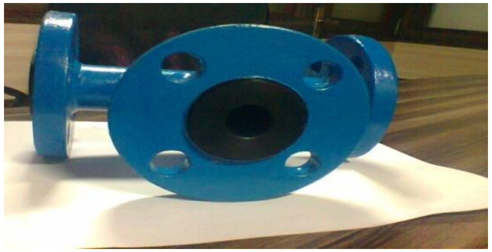
- **Pharma and Laboratory:**
  -Hose chemical lining for pipe, valves and fitting, reactor lining, agitator, injector, addition devices
  -Fluoro polymer based Tubes, Bottles, Beakers, Vacuum-DK, Stirrers, Sheets, Sampling device, Adopter, Joints, Head bush etc.

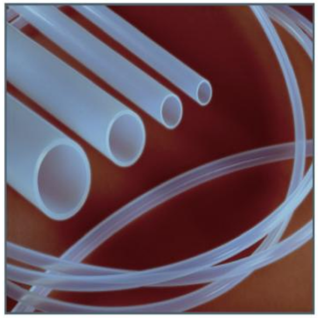
Diaphragm : Compression Molded

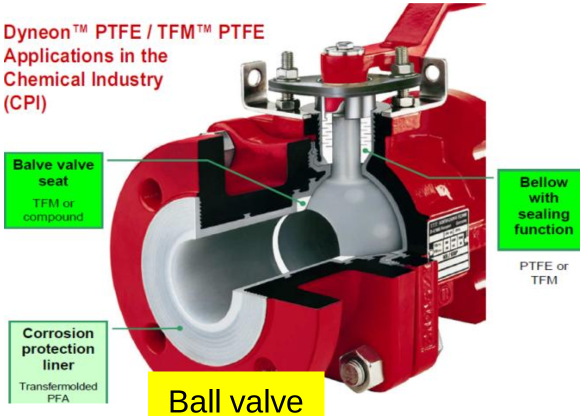


TFM liners

Antistatic PFA







Ball valve

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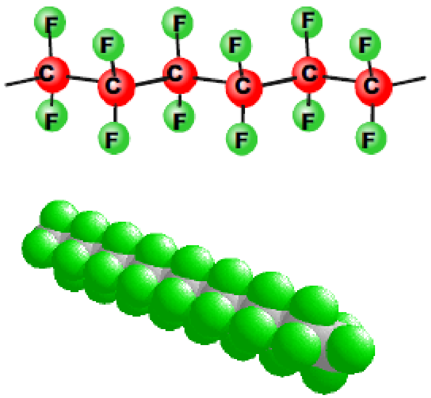
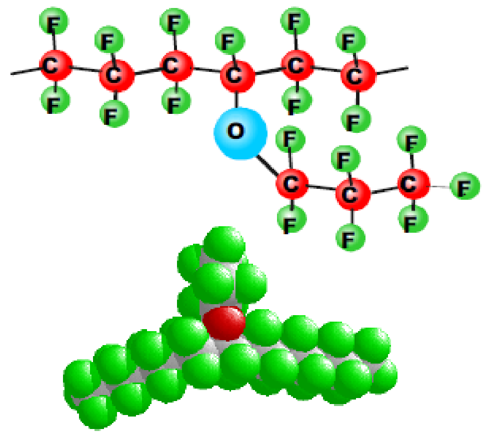
Molecular Structure – PTFR / TFM / PFA

Dyneon™ PTFE



Molecular weight: ca. 100 Mio
Melt viscosity: 10^{12}
Comonomer PPVE: ---
Melt-/gel point: 347 / 327 ℃

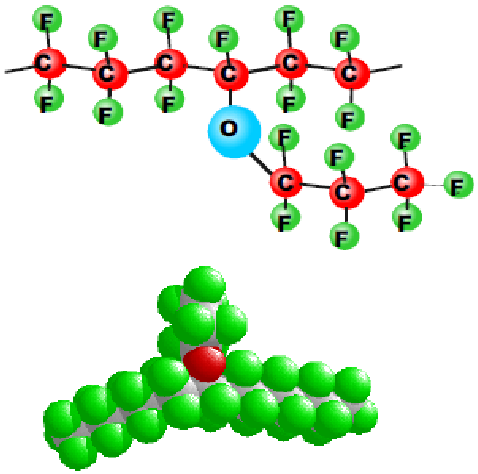
Dyneon™ TFM™ PTFE



Molecular weight: ca. 20 Mio
Melt viscosity: 10^{9}-10^{10}
Comonomer PPVE: <1%
Melt-/gel point: : 347 / 327 ℃

PTFE and TFM are not melt processable fluoroplastics

Dyneon™ PFA



Molecular weight: ca. 1 Mio
Melt viscosity: 10^{8}
Comonomer PPVE: 3-4%
Melting point: 290-308 ℃

PFA is a melt processable fluorothermoplastic

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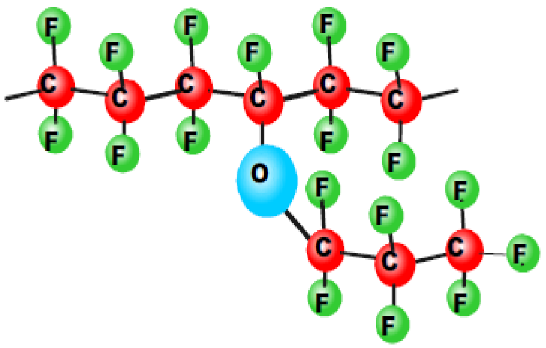
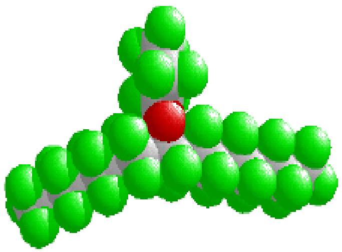
TFM - Molecular structure and Properties



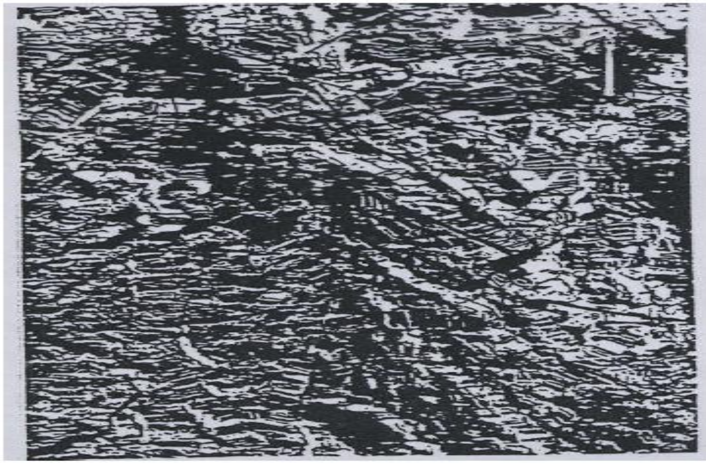


Molecular weight:	ca. 20 mio g/mol
Melt viscosity:	10^9-10^{10} Pas
Comonomer PPVE:	<1%
Melt-/gel point:	347 / 327 °C

- Large Service temperature range: -200 to 260 ^{0}C
- High purity /free of additives
- No embrittlement or aging
- Excellent Mechanical properties:
  - High elongation at break
  - Reduced SVI,
  - Low deformation under load,
  - Good recovery after deformation
- Dense polymer structure due to improved particle fusion
- Very low pore content and reduced permeation
- Improved surface quality and easy cleaning
- Good weldability by special welding technique

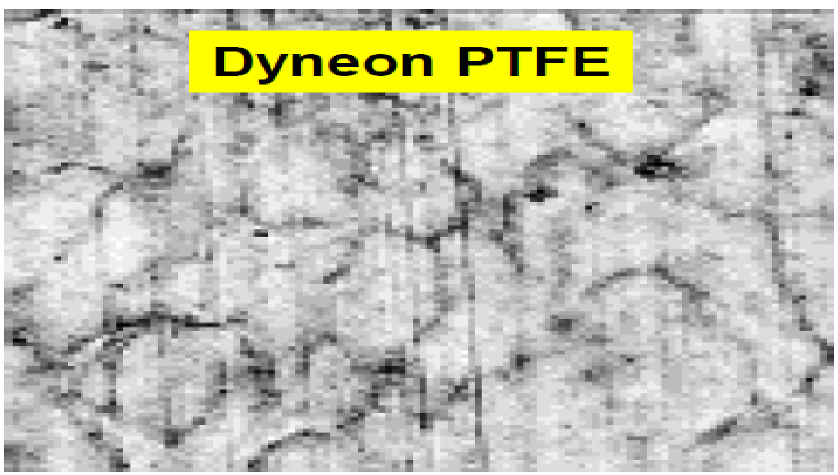
Crystallite structure of PTFE and TFM and Particle Fusion in TFM

SEM photographs of fractured surfaces (-196 °C)

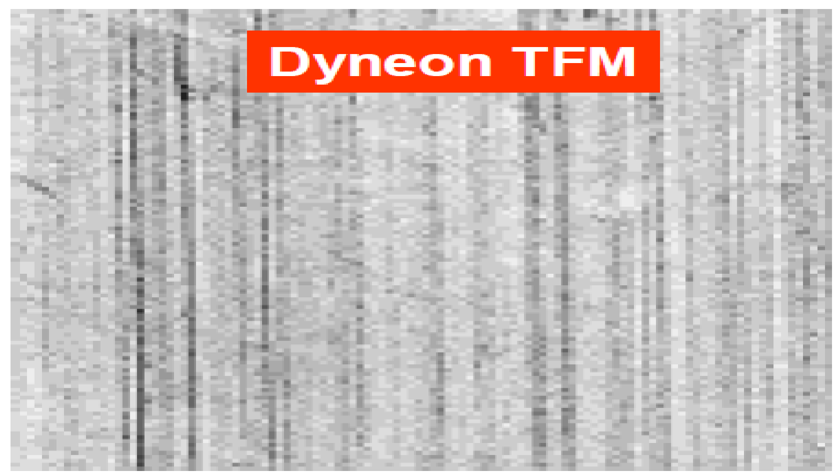


Dyneon PTFE

Dyneon TFM



Visible particle structure



Homogeneous particle structure

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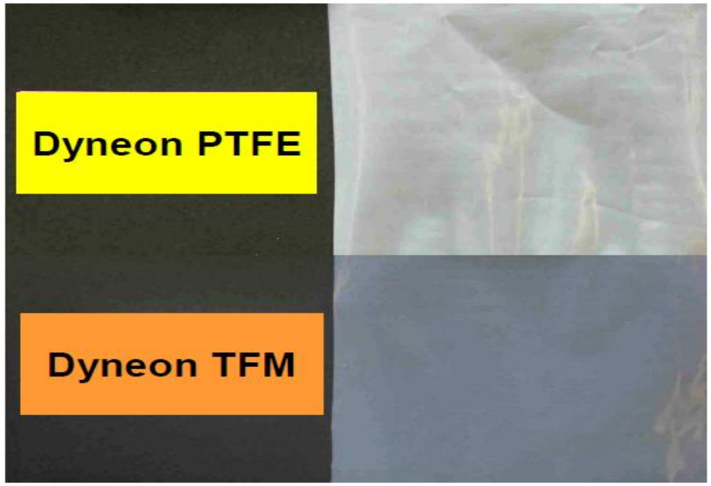
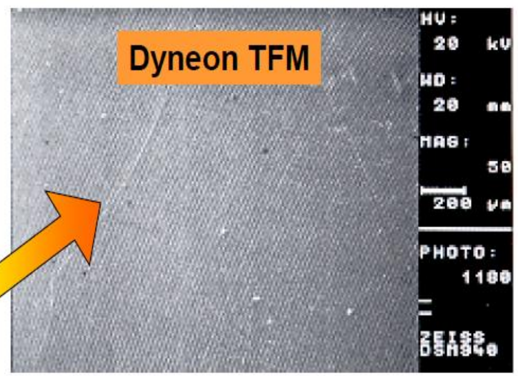
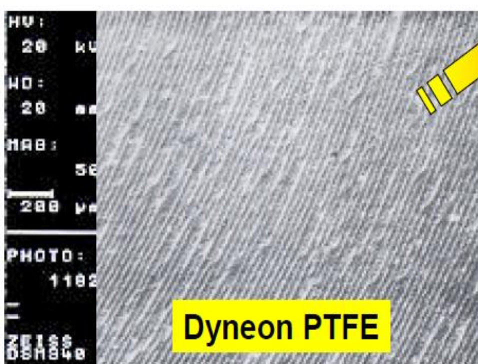
TFM – Improved Transparency and Surface Quality



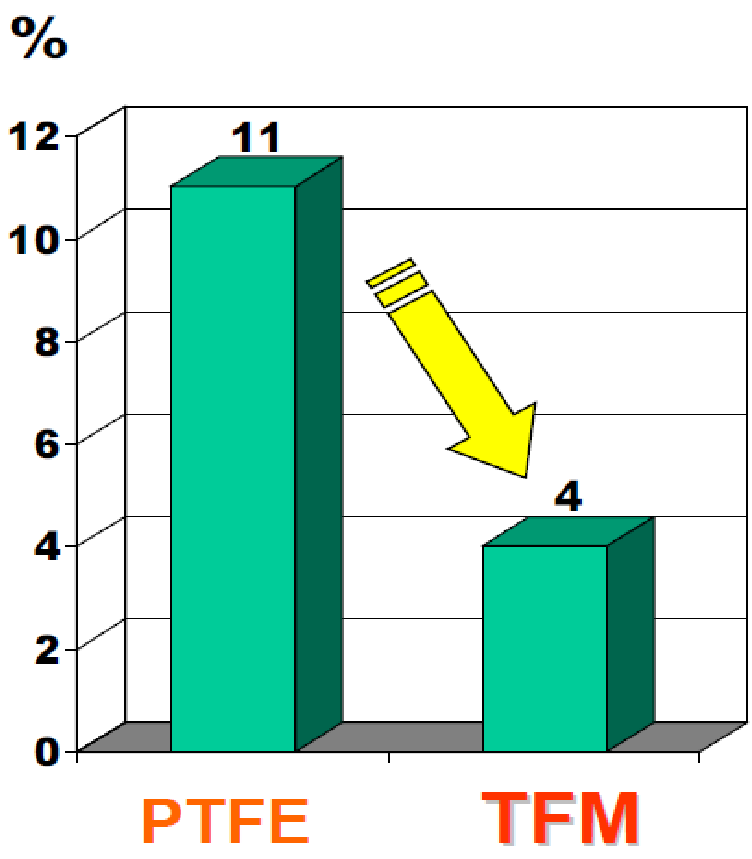


Reduced surface roughness after machining



(magnification 50x)

TFM - Improved Cold Flow property



- Increased stability
- For sealing applications
  - -Reduced loss of surface pressure
  - -Improved recovery behavior at alternating load
  - -Better sealing vs. time
- Assembly
  - -Shorter assembly time
  - -Reduced effort for tightening
- <u>Conditions:</u>

  Load: 15 MPa

  Duration: 100 h

  Remaining deformation after 24 h recovery

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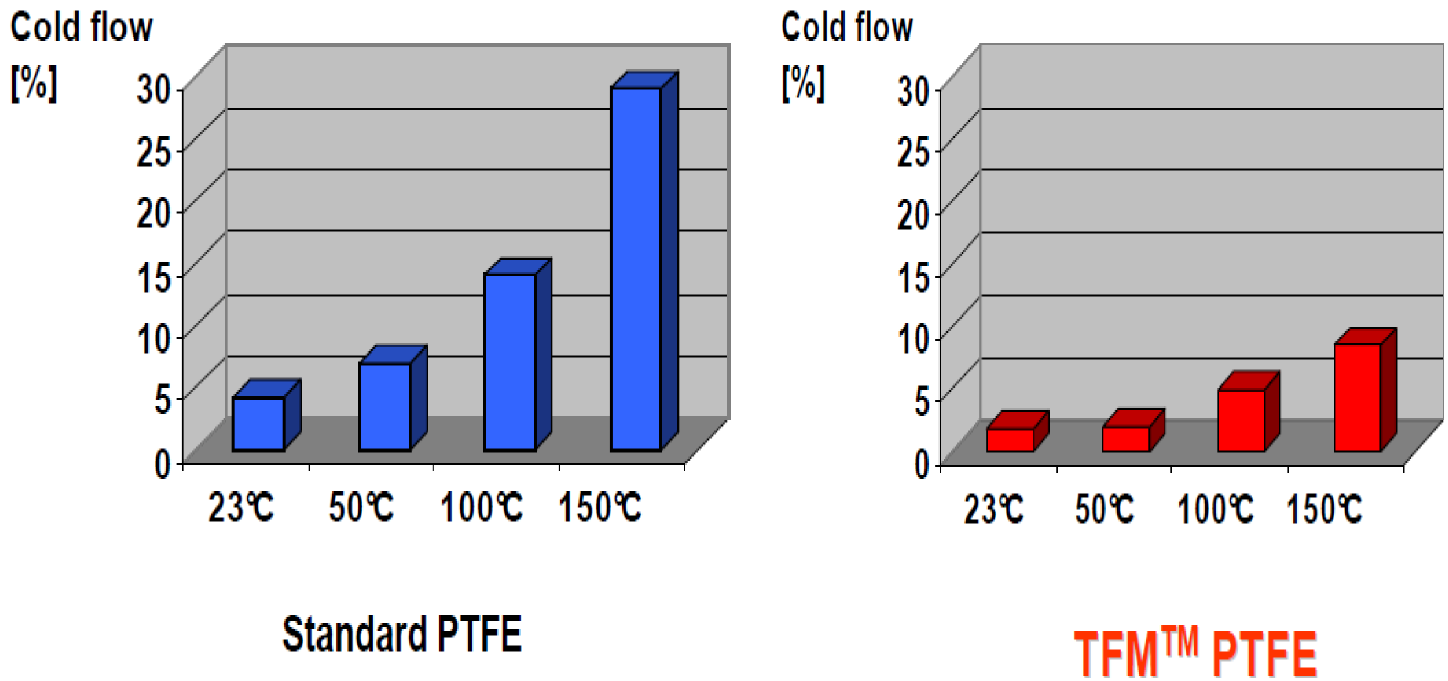
TFM - Improved Cold Flow property

Deformation under load (cold flow) at various temperatures



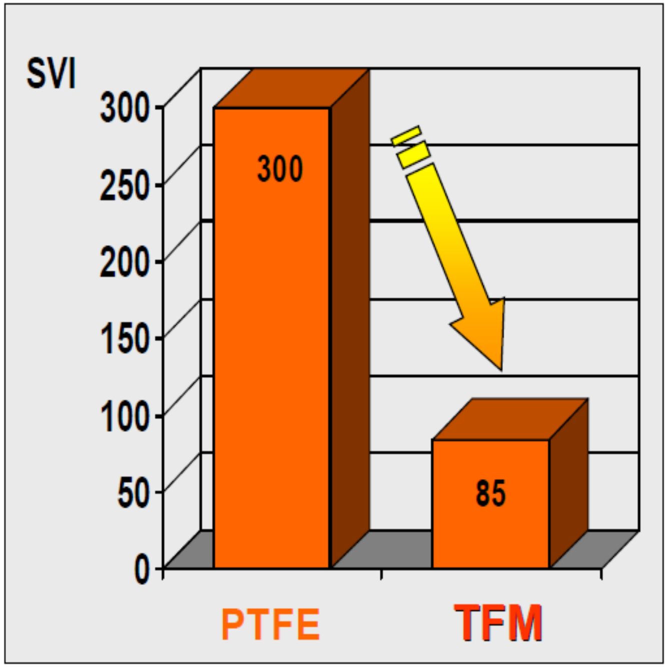
<u>Conditions:</u>

Load: 10 MPa

Duration: 100 h

Remaining deformation after 24 h recovery

TFM – Improved SVI property



SVI = Index for void formation during stretching of PTFE

The better particle coalescence reduces the stretch void index (SVI) of TFM PTFE by a factor of more than 3.

- **The smaller the SVI-value, the lower the void formation during stretching**
- **Materials with low SVI-values (Dyneon TFM PTFE) have low void content, even in the stretched condition: i.e. the product is more dense and less permeable !**

SVI = (USG – SSG) X 1000

USG = unstrained spec. gravity

SSG = strained spec. gravity

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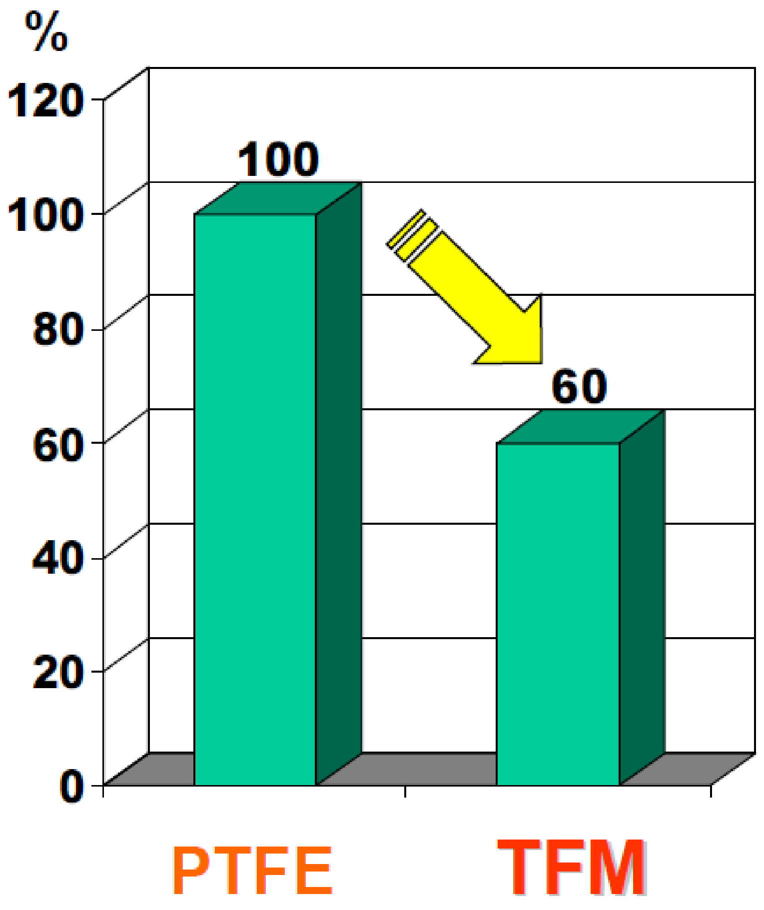
TFM - Improved Permeation property



- Improved barrier properties
- Reduced emission at same wall thickness
- Reduced wall thickness at same emission
- Reduced permeation also after shaping (SVI effect)

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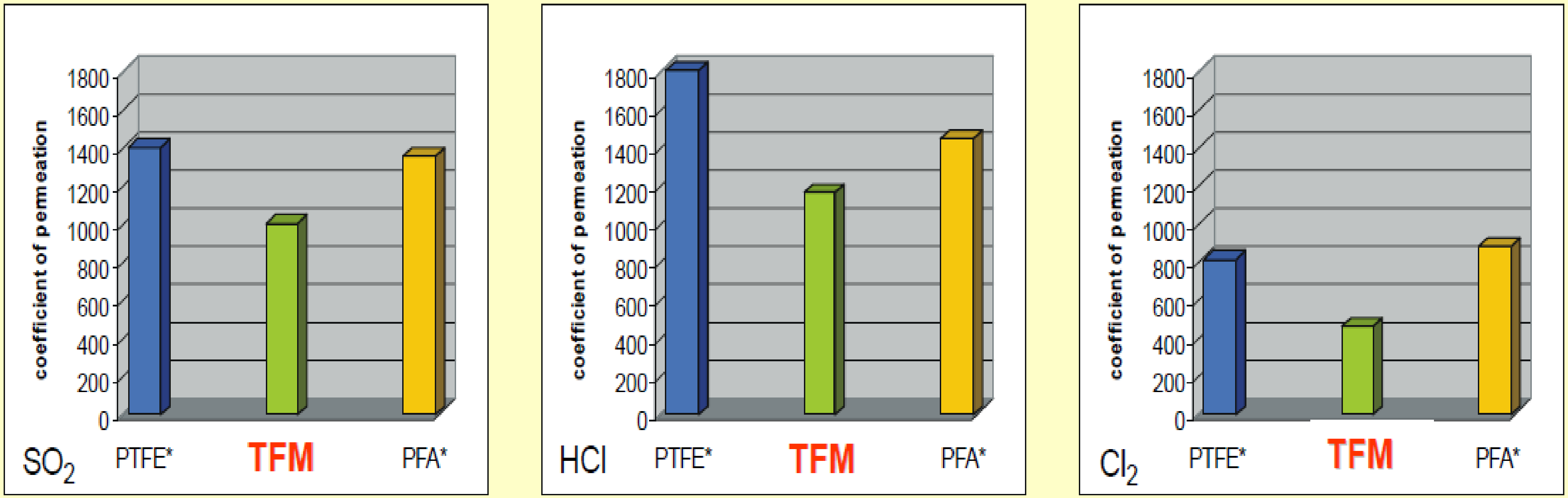
TFM - Improved Permeation property

Permeation of SO_2, HCl and Chlorine through fluoropolymers (100 ^{0}C – 1mm film)

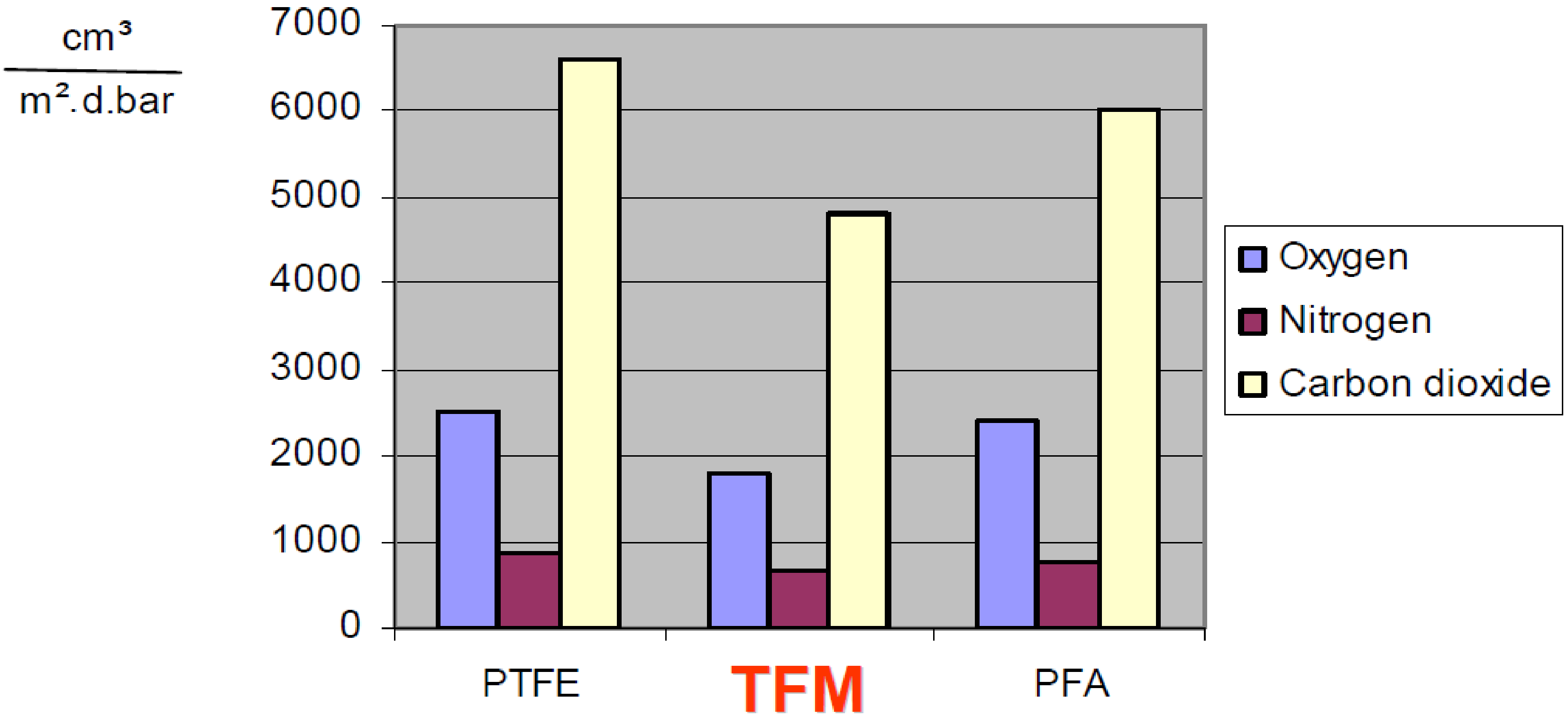


results in $\frac{cm^3 \bullet 1000\,\mu m}{m^2 \bullet d \bullet bar}$

* PTFE = Dyneon™ TF 1750 PTFE
* TFM = Dyneon™ TFM™ 1700 PTFE
* PFA = Dyneon™ PFA 6502 N

TFM - Improved Permeation property

Permeation of inert gases through fluoropolymers
(20 ^{0}C – 100 micron film)



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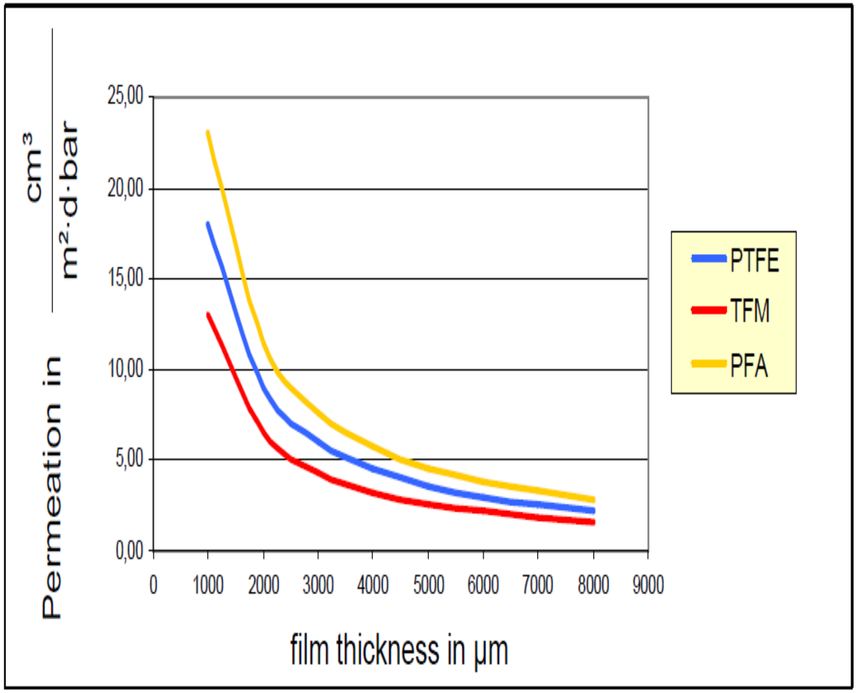
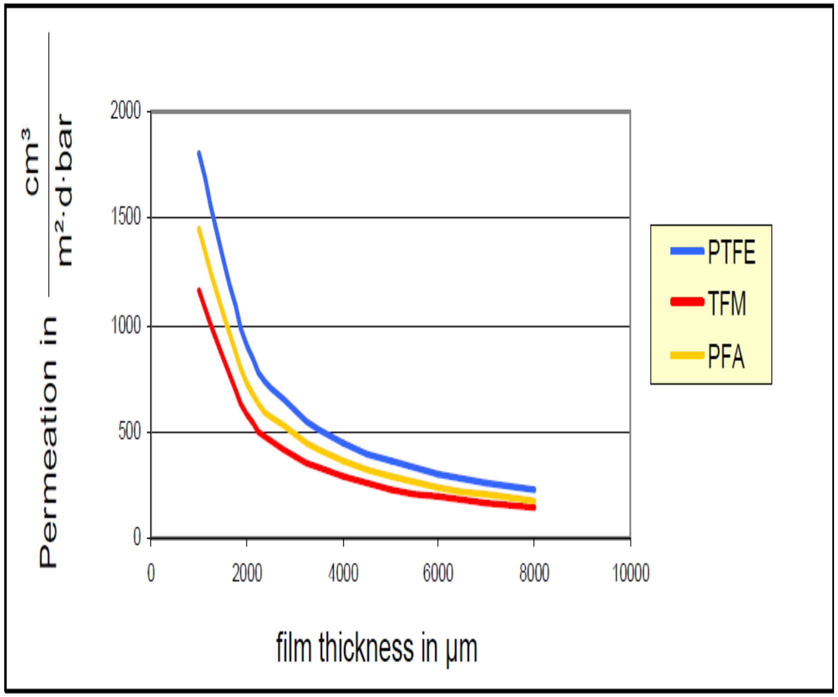
TFM - Improved Permeation property

Permeation of water vapor Vs. film thickness (100 ^{0}C)



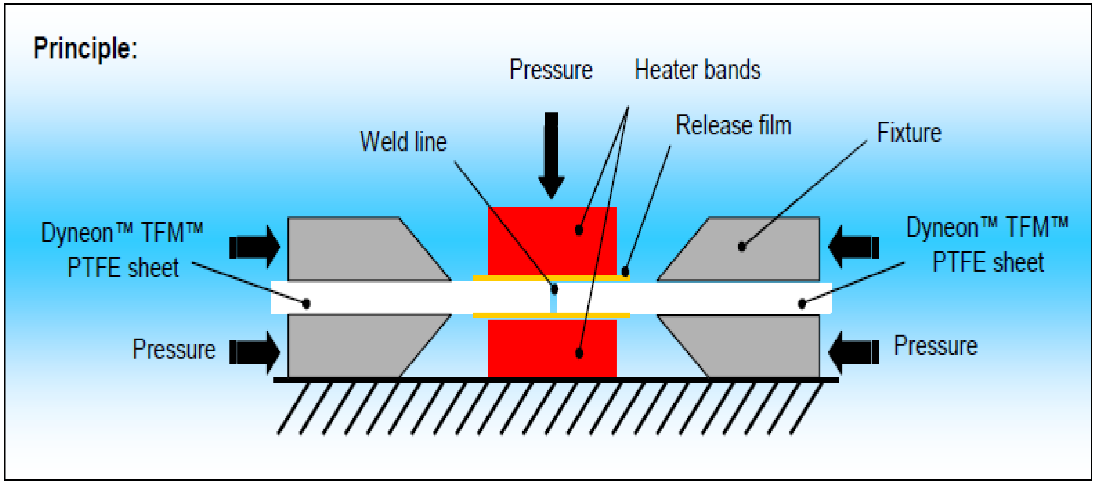
PTFE = TF 1750
TFM = TFM 1700
PFA = PFA 6502 N

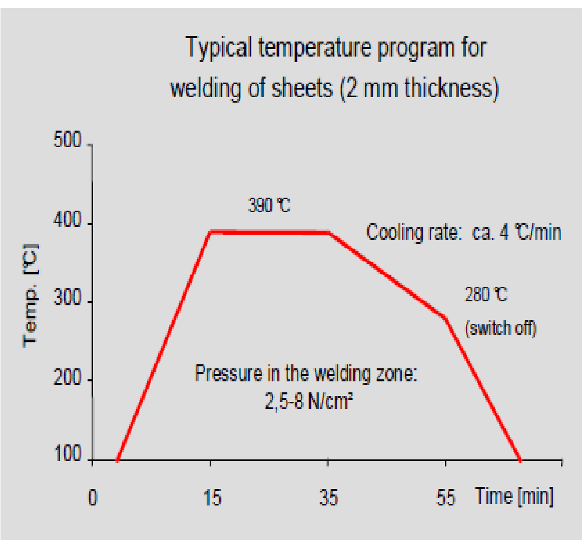
Permeation of HCl gas Vs. film thickness (100 ^{0} C)

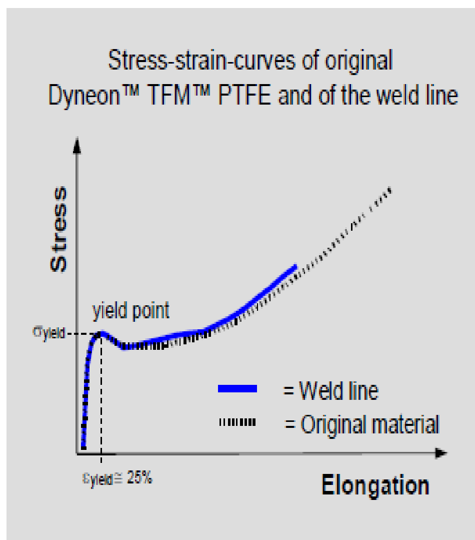


PTFE = TF 1750
TFM = TFM 1700
PFA = PFA 6502 N

Welding techniques for TFM PTFE







Butt welding by heater band

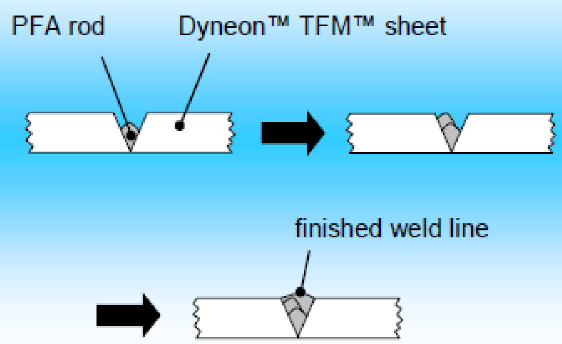
- **Applications:** Welding of Sheets, Pipes or Tubes

-Results:
Excellent weld strength
Best welding technique for TFM-TFM

Welding techniques for TFM PTFE

Hot air welding with PFA rods

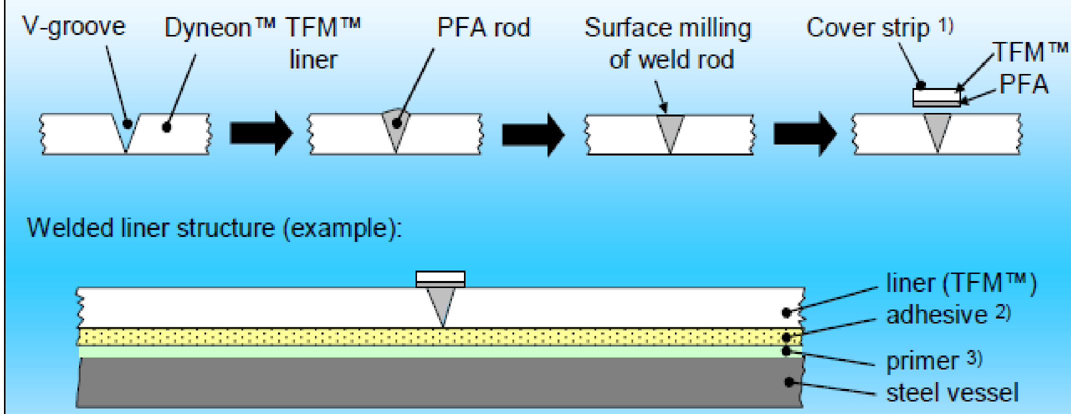
Principle:



Applications:
Sheet welding of Dyneon™ TFM™ PTFE for lining applications in the chemical processing industry (CPI). Quality control by dielectric strength measurement.

Cover strip welding with PFA

Principle:



1) Typical film thicknesses are 1 mm for the Dyneon™ TFM™ PTFE film and 0,2 mm for the PFA film. Both films are joined by compressing at ca. 340 ℃.
2) Adhesive selection is determined by requirements for service temperature and chemical resistance. Typically, 2K-epoxy adhesives (higher strength and chemical resistance) or 2K-PU systems (higher elasticity) are used.
3) Alternatively also sandblasting

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Chemical resistivity of Dyneon Fluoro products

		Chemicals	Concen. %	Temp. °C	PTFE & TFM™ PTFE	PFA	FEP	ETFE	THV 500	HTE 1705
Acids	Organic	Trichloroacetic Acid		23	+	n/a**	+	+	n/a**	n/a**
		Trichloroacetic Acid		50	+	n/a**	+	•	—	•
		Trichloroacetic Acid		100	+	+	n/a**	n/a**	n/a**	n/a**
	Inorganic	Hydrochloric Acid	36	23	+	n/a**	+	+*	+	+
		Hydrochloric Acid	36	100	+	—	n/a**	•*	+	+
		Hydrochloric Acid	36	155	+	—	•	n/a**	n/a**	n/a**
		Sulfuric Acid	96	23	+	n/a**	+	+*	n/a**	n/a**
		Sulfuric Acid	96	100	+	+	n/a**	+*	+	+
		Sulfuric Acid	96	155	+	+	+	n/a**	n/a**	n/a**
		Phosphoric Acid	conc.	100	+	+	n/a**	+*	+	+
		Phosphoric Acid	conc.	165	+	+	•	n/a**	n/a**	n/a**
		Hydrofluoric Acid	40	100	+	+	+	n/a**	•	+
		Hydrofluoric Acid	40	155	•	+	n/a**	n/a**	n/a**	n/a**
		Hydrofluoric Acid	49	35	+	n/a**	+	n/a**	+	+
		Hydrofluoric Acid	49	155	•	+	n/a**	n/a**	n/a**	n/a**
		Nitric Acid	conc.	23	+	n/a**	+	+*	n/a**	n/a**
		Nitric Acid	conc.	100	+	n/a**	n/a**	+*	+	•
		Nitric Acid	conc.	155	+	•	•	n/a**	n/a**	n/a**
		Fluorosulfonic Acid		100	•	•	n/a**	+*	n/a**	n/a**
		Chlorosulfonic Acid		100	+	+	n/a**	—*	n/a**	n/a**
		Chromosulfonic Acid		100	+	+	n/a**	•*	n/a**	n/a**
Alcohols		Ethylene Glycol		100	+	+	n/a**	n/a**	n/a**	n/a**
		Methanol		23	+	+	+	+	•	+
		Methanol		50	+	+	n/a**	+	•	+
Amides		Dimethylacetamide		50	+	n/a**	+	—*	—	—
		Dimethylacetamide		100	+	+	n/a**	•*	n/a**	n/a**
		Dimethylformamide		23	+	n/a**	+	n/a**	n/a**	n/a**
		Dimethylformamide		50	+	n/a**	+	+*	—	—
		Dimethylformamide		100	+	•	n/a**	+*	n/a**	n/a**
		N-methylpyrrolidone		23	n/a**	n/a**	+	n/a**	—	+
		N-methylpyrrolidone		50	+	n/a**	n/a**	n/a**	—	n/a**
		N-methylpyrrolidone		100	+	+	+	n/a**	—	—
Amines		Benzyl Amine		23	+	n/a**	n/a**	+*	n/a**	n/a**
		Benzyl Amine		100	+	n/a**	n/a**	•*	n/a**	n/a**
		N-Butyl Amine		100	+	n/a**	n/a**	•*	n/a**	n/a**

Chemical resistivity of Dyneon Fluoro products

		Chemicals	Concen. %	Temp. °C	PTFE & TFM™ PTFE	PFA	FEP	ETFE	THV 500	HTE 1705
Bases		Ammonia	25	23	+	+	+	n/a**	—	+
		Sodium Hydroxide	25	23	+	n/a**	+	n/a**	n/a**	n/a**
		Sodium Hydroxide	50	23	+	+	+	n/a**	+	+
		Potassium Hydroxide	50	23	+	+	+	n/a**	+	+
		Potassium Hydroxide	50	100	+	+	n/a**	—*	n/a**	n/a**
Esters		Ethyl Acetate		23	+	n/a**	●	●	—	—
		Ethyl Acetate		50	+	n/a**	n/a**	n/a**	—	—
		Ethyl Acetate		100	+	n/a**	n/a**	●	n/a**	n/a**
Ethers		Tetrahydrofuran		23	+	+	+	—	—	—
		Tetrahydrofuran		50	n/a**	●	—	—	—	—
Halogens		Chlorine gas		23	+	●	n/a**	+*	+	+
		Iodine		23	+	+	n/a**	n/a**	n/a**	n/a**
		Bromine		23	+	+	n/a**	+*	n/a**	n/a**
Halogenated Hydrocarbons		1,1 - Dichloroethane		23	●	n/a**	n/a**	+*	n/a**	n/a**
		1,1 - Dichloroethane		50	●	n/a**	n/a**	+*	n/a**	n/a**
		1,2 - Dichloroethane		23	●	n/a**	n/a**	+*	n/a**	n/a**
		1,2 - Dichloroethane		50	●	n/a**	n/a**	+*	n/a**	n/a**
		1,2 - Dichloroethane		100	●	n/a**	n/a**	+*	n/a**	n/a**
		1,4 - Dichlorobutane		23	+	n/a**	n/a**	+*	n/a**	n/a**
		1,1,2 - Trichloroethane		23	●	n/a**	n/a**	●*	n/a**	n/a**
Hydrocarbons	Aliphatic	Iso Octane		23	+	+	+	+	+	+
		Iso Octane		50	+	+	n/a**	n/a**	+	+
		Hexane		23	+	n/a**	●	n/a**	+	n/a**
	Aromatic	Toluene		23	+	+	+	●	●	+
Ketones		Acetone		23	+	+	+	+*	—	—
		Acetone		50	+	n/a**	n/a**	n/a**	—	—
		Acetone		100	+	n/a**	n/a**	●*	n/a**	n/a**
		Methyl Ethyl Ketone		23	+	n/a**	+	n/a**	—	—
		Methyl Ethyl Ketone		50	+	n/a**	n/a**	—	n/a**	—
		Methyl Ethyl Ketone		80	+	+	n/a**	n/a**	n/a**	n/a**
Other Heterocycles		Benzyl Chloride		23	●	n/a**	+	n/a**	n/a**	n/a**
		Pyridine		23	+	n/a**	+	—*	n/a**	●

3M

Chemical resistivity of Dyneon Fluoro products

	Test Method	Unit	Dyneon™ PTFE	Dyneon™ TFM™ PTFE	Dyneon™ PFA[1]	Dyneon™ FEP[1]	Dyneon™ ETFE[1]	Dyneon™ THV 500[2]	Dyneon™ HTE 1705[2]
Physical Properties									
Specific Gravity	ASTM D792	g/cm^3	2.16	2.16	2.15	2.15	1.73	1.98	1.76
Melting Point	ASTM D4591	°C	327	327	310	260	267	165	210
Service Temperature									
minimum		°C	-200	-200	-200	-200	-190	-50	n/a*
maximum		°C	260	260	260	205	150	130	n/a*
Mechanical Properties	ISO 12086 50mm/min at 23°C								
Tensile Strength at Break									
@23°C		MPa	33	34	30	30	46	28	49
@100°C		MPa	22	25	24	14	20	12	17
Elongation at Break									
@23°C		%	360	580	380	350	425	500	450
@100°C		%	520	590	440	320	600	590	800
Tensile Modulus									
@23°C		MPa	540	630	530	600	1150	240	980
@100°C		MPa	210	255	160	85	280	27	60
Yield Strength									
@23°C		MPa	12.4	13.2	16	14	26	8	20
@100°C		MPa	7.5	8.2	7	6	10	3	4
Flexural Modulus	ASTM D790 3pt bend	MPa	n/a*	n/a*	550	580	1100	210	1040
Deformation Under Load after 100h stress & 24h recovery	ASTM D621								
@23°C / 15 MPa		%	11	4	2	n/a*	n/a*	n/a*	n/a*
@150°C / 8.8 MPa		%	34.5	20	10	n/a*	n/a*	n/a*	n/a*
@205°C / 5.2 MPa		%	18.5	5.5	14.5	n/a*	n/a*	n/a*	n/a*
Hardness	ASTM D2240/DIN 53505	ShD	56	59	60	57	66	54	66
Surface Roughness			0.9[3]	0.3[3]	0.3[4] (UHP grades <0.1)	0.04[4]	n/a*	n/a*	n/a*
Electrical Properties									
Dielectric Constant	ASTM D150 @ 1MHZ		2.1	2.1	<2.15	<2.15	2.58	4.82	2.21
Dissipation Factor	ASTM D150 @ 1MHZ	10^{-4}	0.7	0.7	<5	<9	<10	100	<60
Dielectric Strength	ASTM D149	kv/mm	60	70	75	63	59	48	69
			(0.20 mm film)	(0.20 mm film)	(0.25 mm film)	(0.25 mm film)	(0.25 mm film)	(0.25 mm film)	(0.20 mm film)
Flame Resistance									
Limiting Oxygen Index	ASTM D2863	%	96	96	>95	>95	>30	>75	33
Flammability Rating	UL 94		V-0	V-0	V-0	V-0	V-0	V-0	n/a*
Permeation Properties									
HCl	DIN 53380 Part 4.1.2	(cm^3•1000µm/m^2•d•bar)							
@23°C			630	445	160	n/a*	190	90	180
@100°C			1600	1150	1400	n/a*	2700	5600	4250
Water Vapor	DIN 53122 Part 2	(g•1000µm/m^2•d)							
@23°C			0.02	0.017	0.018	n/a*	0.11	0.17	0.11
@100°C			3.5	2.43	4.54	n/a*	7.00	33.0	5.00

Approvals

Regulatory Compliance Status of Dyneon Fluoroplastics for Food Contact

The following Dyneon products are in compliance with FDA 21 CFR 177.1550: Dyneon PFA 6900N – Dispersion; Dyneon PFA 6502N/PFA X 6502 UHP – Granular; Dyneon PFA 6505N/PFA X 6505 UHP – Granular; Dyneon PFA 6510N – Granular; Dyneon PFA 6515N/PFA X 6515 UHP – Granular; Dyneon FEP X 6300 – Dispersion; Dyneon FEP X 6301 – Granular; Dyneon FEP X 6303 – Granular; Dyneon FEP X 6307 – Granular

The following Dyneon products are in compliance with FDA 21 CFR 177.2600: Dyneon THV X 310 – Agglomerate, Granular; Dyneon THV 500 – Agglomerate, Granular; Dyneon THV X 610 – Agglomerate, Granular; Dyneon THV 510D – Dispersion

Compliance with regulations: It is the responsibility of the user to determine whether its specific formulation and intended use comply with applicable laws and are suitable for its intended application. For food contact applications, fillers and ingredients utilized must be in compliance with applicable regulations for repeated food contact use.

[1]Nominal value for extrusion grade resin.

[2]Dyneon manufactures a variety of THV and HTE materials which differ in composition, melt flow index, and properties. Please consult us for further information on additional grades.

[3]Roughness measured on machined parts without polishing.

[4]Roughness measured on the inside of extruded tubes.

*Information not available

Approvals

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3M recommended grades and FDA Status

-Normal PTFE:

-TF-1750,TF-1620, TF-1641, TF-1645

-Modified PTFE:

-TFM-1700, TFM-1705, TFM-1600

- PTFE/TFM based compound:

-various composition available e.g., 25% carbon, 25% Glass & 23%carbon+2% graphite ect.

-PTFE Paste extrusion:

-TF-2021, TF-2025, TFM-2033 , TF-2029, TF-2071/72/73 ect.

-PTFE Dispersion:

-TF-5135, TF-5050, TF-5060, TF-5070

-Fluoro Thermoplastics:

- PFA - 6502TZ, 6515TZ, 6525TZ (UHP grades)
- FEP – 6301Z, 6303Z, 6307Z, 6309Z, 6322Z
- ETFE, THV, THE, PVDF

1000 Series	FDA	3A	USP	2000 Series	FDA	3A	USP	9000 Series	FDA	USP
TF 1750	Y	P	P	TF 2021	Y	P	P	TF 9201	Y	NT
TF 1620	Y	P	P	TF 2025	Y	P	P	TF 9202	Y	NT
TF 1641	Y	P	P	TF 2029	Y	P	P	TF 9205	N	NT
TF 1645	Y	P	P	TF 2071	Y	P	P	TF 9207	Y	NT
TFR 1105	Y	P	NT	TF 2072	Y	P	P			
TFR 1502	Y	P	NT	TFM 2001	Y	P	P			
TFM 1700	Y	P	P							
TFM 1705	Y	P	P							
TFM 1600	Y	P	P							
TFM 1605	Y	P	P							
TFM 1610	Y	P	P							
TFM 1635	Y	P	P							

Legend: Y = Compliant | N = Not compliant |
P = Representative Samples Tested and Passed | NT = Not Tested

3M

Thanks