Halar®

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Halar® ECTFE

Design & Processing Guide

SPECIALTY POLYMERS

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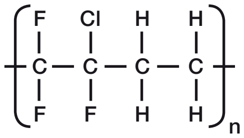
Chemistry

Composition and Structure – Property Relationships

Halar® ECTFE is a semi-crystalline and melt-processable fluoropolymer from Solvay Specialty Polymers manufactured at its ISO-certified plant in Orange, Texas.

The ethylene chlorotrifluoroethylene (ECTFE) polymer is obtained by the polymerization of the two monomers ethylene and chlorotrifluoroethylene, and corresponds to the following chemical formula:



A 1:1 alternating polymer

Because of its chemical structure, Halar® ECTFE offers a unique combination of properties:

1. chemical resistance and high thermal rating given by the extremely strong dissociation between carbon and fluorine atoms

2. very good mechanical properties given by strong interchain interactions due to hydrogen bonding.

One of the principal advantages of Halar® fluoropolymer is the ease with which it can be processed. Halar® fluoropolymer is a true thermoplastic that can be handled by conventional techniques of extrusion as well as by blow, compression, injection, roto and transfer molding. Powder coating methods are also applicable. Halar® ECTFE polymer is available in a wide range of melt viscosities to suit virtually every processing technique.

Purity of the Polymer

Halar® ECTFE is an extremely pure polymer. Static soak testing in ultra-pure water and high purity chemicals shows extremely low levels of metallic and organic extractables. Additional dynamic rinse data validates Halar® ECTFE as suitable for high purity systems in the semiconductor, biotech, and pharmaceutical industries. Furthermore, Halar® ECTFE exhibits very low fluoride ion leach out.

For this reason, Halar® ECTFE is used as a lining and coating for ultra-pure water systems in the semiconductor industry. FM 4922 approved exhaust duct systems use Halar® ECTFE coated stainless steel.

Comparison to Other Fluoropolymers

The most distinctive properties of the main melt-processable fluoropolymers are depicted in Table 1. As seen in Table 1, among Fluoroplastics, Halar® ECTFE features an intermediate behavior. For instance, ECTFE has wider chemical resistance and a higher thermal rating compared to polyvinylidene fluoride (PVDF), while it features better mechanical properties with respect to ethylene tetrafluoroethylene (ETFE) and perfluorinated polymers. Thus, Halar® ECTFE embodies an excellent trade-off among general properties, offering high chemical and mechanical resistance combined with easy processing of the polymer.

In addition, Halar® ECTFE exhibits much lower surface roughness when compared to most other plastics. This is extremely important in high purity applications as it helps to limit foreign particle trapping.

Physical Properties

Table 1: Comparison to other fluoropolymers: general properties

	Units	PVDF	Halar® ECTFE	ETFE	FEP	PFA	PTFE
Average properties							
Density	g/cm³	1.78	1.68	1.72	2.15	2.15	2.17
Melting point	°C (°F)	160 – 172 (320 – 342)	242 (468)	262 (504)	270 (518)	305 (581)	330 (626)
Chemical resistance (comparative behavior)		good (pH 1 – 12)	very good (pH 1 – 14)	very good (pH 1 – 14)	excellent	excellent	excellent
Tensile properties at 23 °C (73 °F)							
Yield strength	MPa (psi)	50 (7,250)	30 (4,300)	25 (3,600)	12 (1,700)	16 (2,300)	10 (1,450)
Stress at break	MPa (psi)	40 (5,800)	54 (7,800)	40 (5,800)	22 (3,200)	30 (4,300)	30 (4,300)
Elongation at break	%	20 – 100	250	250	300	300	350
Modulus of elasticity	MPa (kpsi)	2,000 (290)	1,655 (240)	1,000 (145)	550 (80)	550 (80)	750 (109)
Shore D hardness	-	78	75	68	57	62	57
Deflection temperature under load of 1.82 MPa (264 psi)	°C (°F)	100 (212)	65 (149)	70 (158)	54 (129)	50 (122)	56 (133)
Thermal conductivity	W/(mK)	0.20	0.20	0.20	0.20	0.22	0.25
Coefficient of linear thermal expansion	$K^{-1} \cdot 10^{-6}$	130	100	90	110	120	130
Volume resistivity	Ω·cm	≥ 10^{14}	10^{16}	10^{14}	10^{18}	10^{17}	10^{18}

Thermophysical Properties

Halar® ECTFE polymers offer a wide use temperature range from –80 °C to 150 °C in non load-bearing applications.

The maximum service temperature can be affected by the presence of system stresses and chemical environment. Stress cracking for standard grades may appear in the 125 – 150 °C range, especially for high-MI grades.

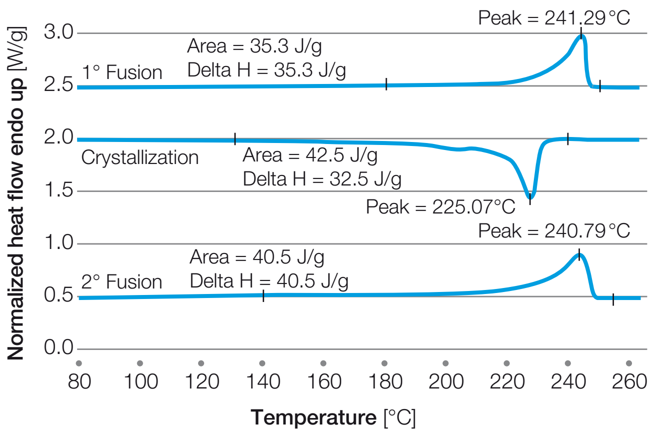
Differential Scanning Calorimetry (DSC)

The crystalline melting temperatures and corresponding heats of fusion ΔH_f of Halar® ECTFE polymers, recorded by DSC under defined operating conditions (ASTM D 3418), are presented in Table 2. Figure 1 depicts the relative heat flux curves of Halar® ECTFE as a function of temperature during heating, cooling and subsequent re-heating (second fusion). Crystallization occurs about 20 °C (36 °F) below the melting temperature.

Table 2: Thermophysical data recorded by DSC for Halar® ECTFE

	Halar® ECTFE	Units
Melting Point, T_f	242 (468)	°C (°F)
Heat of Fusion, ΔH_f	42 (18)	J/g (Btu/lb)
Crystallization Point, T_χ	222 (432)	°C (°F)
Crystallization Heat, ΔH_χ	40 (17)	J/g (Btu/lb)

Figure 1: DSC curves for Halar® ECTFE



Heat Deflection Temperature (HDT) (ASTM D648)

The heat deflection temperature under load (HDT) indicates the short-term thermal behavior of a material under a certain applied load. The HDT for a polymer is determined by the following test procedure outlined in ASTM D648: the test specimen is loaded in three-point bending in the edgewise direction under a flexural stress of either 0.456 MPa (66.7 psi) or 1.82 MPa (264 psi), and the temperature is increased at 2 °C/min until the specimen deflects 0.25 mm. Table 3 presents the HDT values for Halar® ECTFE.

Table 3: HDT values for Halar® ECTFE (4 mm thick samples)

	Heat Deflection Temperature	
	Load 0.46 MPa [°C (°F)]	Load 1.82 MPa [°C (°F)]
Halar® ECTFE	90 (195)	65 (150)

Coefficient of Linear Thermal Expansion

The coefficient of thermal expansion describes how the size of an object changes with a change in temperature. Specifically, it measures the fractional size variation per degree variation in temperature at a constant pressure and is defined by the formula:

$$\alpha_L = \frac{1}{L}\frac{dL}{dT}$$

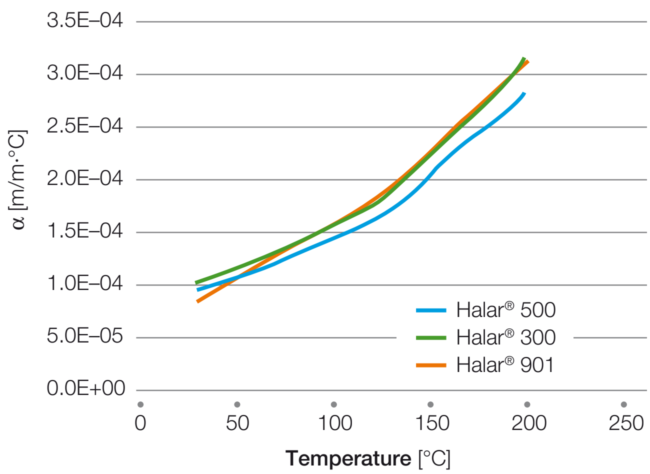
where *L* is the linear dimension (e.g. length) and *dL/dT* is the rate of change of that linear dimension per unit change in temperature. If the variation of the linear expansion coefficient with temperature is not very large, an average linear thermal expansion coefficient α_L over a temperature range is normally reported. The average thermal expansion coefficient over several temperature indicator ranges is shown in Table 4. Figure 2 shows linear expansion increase as a function of temperature.

Table 4: Coefficient of linear thermal expansion

Temperature Range	in/in·°F	m/m·°C
–30 to 50 °C (–22 to 122 °F)	$4.4 \cdot 10^{-5}$	$8 \cdot 10^{-5}$
50 to 85 °C (122 to 185 °F)	$5.6 \cdot 10^{-5}$	$10 \cdot 10^{-5}$
85 to 125 °C (185 to 257 °F)	$7.5 \cdot 10^{-5}$	$13.5 \cdot 10^{-5}$
125 to 180 °C (257 to 356 °F)	$9.2 \cdot 10^{-5}$	$16.5 \cdot 10^{-5}$

In addition, in Figure 2 the coefficient of thermal expansion is presented as a function of the temperature for three different Halar® ECTFE grades.

Figure 2: Thermal expansion curves for Halar® ECTFE measured by thermomechanical analysis (TMA)



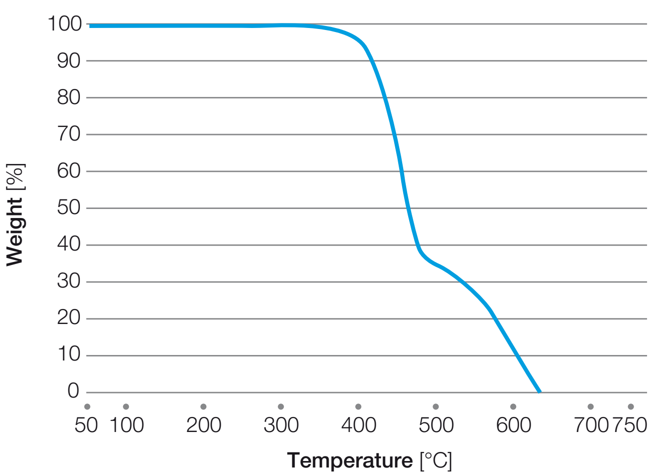
Thermo-Gravimetric Analysis (TGA)

Short-term thermal stability

A very common method to evaluate the thermal stability of a polymeric material is thermogravimetric analysis (TGA), a test that records the changes in weight of a sample heated according to a given temperature program in a controlled atmosphere, usually air or an inert gas like nitrogen. Figure 3 shows the result of thermogravimetric analysis under air for Halar® ECTFE, performed at a heating rate of 10 K/min. The curves for these products are very similar and exhibit major weight loss, which indicates material degradation, at about 400 °C (752 °F). ECTFE polymers are generally processed at temperatures around 270 °C (520 °F), well below the decomposition temperature. It is imperative not to exceed a temperature of 350 °C (662 °F) to avoid rapid polymer degradation.

The decomposition of ECTFE can be sharply accelerated by the presence of certain contaminations, even in low quantities. It is recommended to consult Solvay Specialty Polymers before adding any fillers or pigments to Halar® ECTFE.

Figure 3: Thermogram under air for Halar® ECTFE



Stress Cracking Temperature

Although ECTFE polymers are characterized by very good stability within a wide range of environmental conditions, they may be susceptible to slow, brittle failure when exposed to prolonged mechanical stress at elevated temperatures. This failure mode is usually referred to as thermal stress cracking and can be observed and measured by utilizing Fed. Spec. L-P-390C Class H, a test procedure originally designed for polyethylene. In this test 6.35 mm (0.25 in) wide strips of a 1.3 mm (0.05 in) thick sheet are wrapped around a 6.35 mm (0.25 in) diameter mandrel and exposed to various temperatures in forced-draft ovens. The calculated strain (elongation) of the strip wrapped on the mandrel is about 16 %. The temperature at which Halar® ECTFE polymer will stress crack appears to be predominantly a function of molecular weight and molecular-weight distribution. Based on the results from the above test, the following grades of Halar® ECTFE polymer have the indicated stress-cracking temperatures.

Table 5: Stress cracking temperature

Halar® ECTFE Grade	Melt Index [g/10 min]*	Stress Cracking Temperature [°C (°F)]
300	2	150 (302)
500	18	140 (284)

** Melt index at 275 °C (527 °F) under 2.16 kg load*

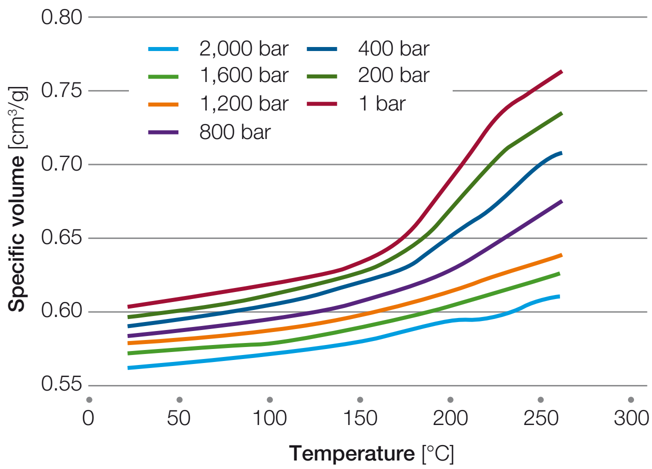
Specific Volume – pvT Curves

The specific volume v=V/m [cm^3/g], where V is the volume of a sample of a certain material and *m* its mass, is an intrinsic property of the material. The specific volume was measured under different pressures and at different temperatures, leading to the curves presented in Figure 4.

This diagram (containing pvT curves, meaning pressure-volume-temperature curves) is obtained by measuring the volume occupied by a known mass of material introduced into a cylindrical space and then heated to fusion and finally cooled under various pressures between 1 and 2,000 bar.

These curves are of special interest for injection molding, because they are a powerful tool for the optimization of the holding phase cycle in injection molding processes.

Figure 4: pvT curves for Halar® ECTFE



Rheological Properties

In the following plots the typical rheological curves for Halar® ECTFE polymers are found. Figure 5 and 6 report the viscosity η and the storage modulus G' against shear rate γ at 275°C (527 °F). Both were measured with a parallel plate rheogoniometer.

Figure 5: Melt viscosity at 275 °C (527 °F) of different Halar® ECTFE grades

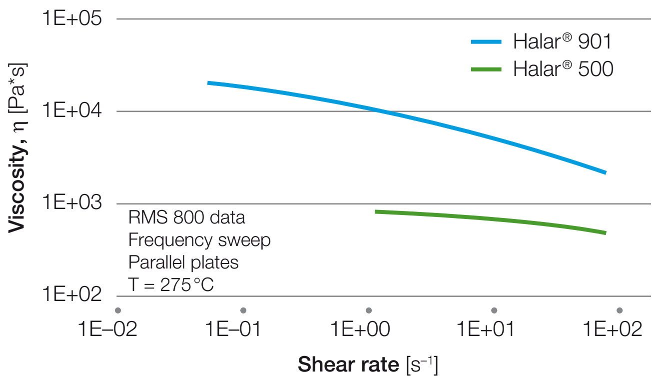
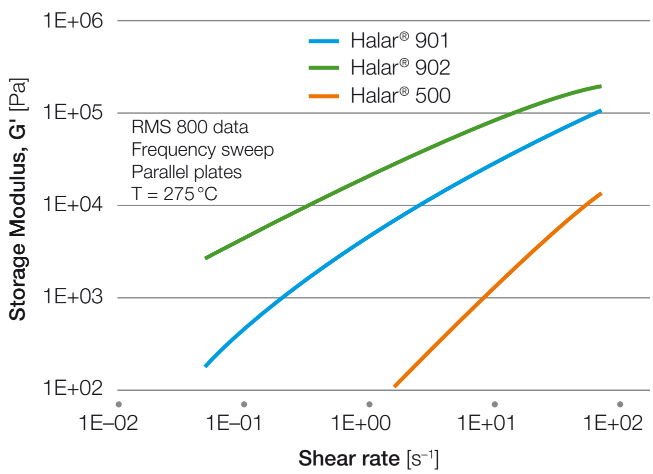


Figure 6: Storage modulus of the melt at 275 °C (527 °F) of various Halar® ECTFE grades



Surface Properties

Angle of Contact and Surface Tension

The angle of contact θ of a drop of liquid on a material and the critical wetting surface tension γ_S for a solid provide indications of the surface wettability for this material: if the angle θ is high and the surface tension is low, the material is not easily wettable.

Halar® ECTFE polymers have a critical surface tension of wetting comparable to that of the ethylene and chlorotrifluoroethylene polymers that make up the Halar® polymer. Halar® ECTFE is not wetted by water but oils and hydrocarbons readily spread on its surface, thus the material can be regarded as hydrophobic. The wettability of Halar® ECTFE can be markedly enhanced by etching with sodium-based etchants normally employed for PTFE.

Table 6 reports the values of the contact angle θ for water and for hexadecane, a non-polar solvent, as well as the critical surface tension values of various polymers compared to those of Halar® ECTFE. Measurements were made at 20 °C (68 °F).

Table 6: Angle of contact with water and hexadecane, and critical surface tension of Halar® ECTFE and other thermoplastics (at 20 °C (68 °F))

	Angle of Contact		Critical Surface Tension γ_C*
	Water [Degrees]	Hexadecane [Degrees]	[mN/m]
Halar® ECTFE	99	11	32
PVDF	80	41	25
PFA/MFA®	105	54	–
PTFE	110	45	18
PCTFE	84	36	31
HD-PE	88	< 5	31
PET	76	–	43
PA 6.6	72	–	46

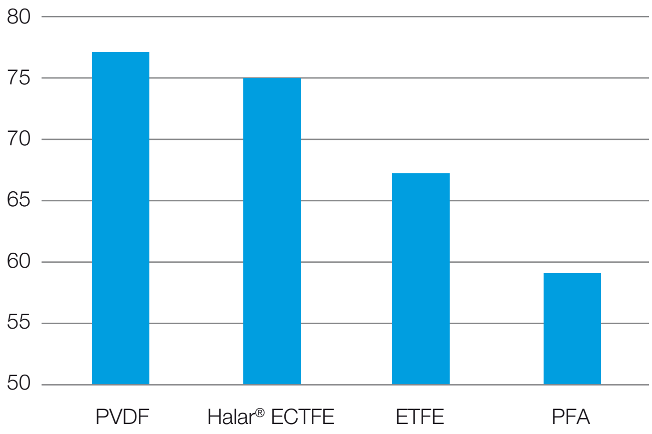
[*Zisman method, values taken from technical literature]

Hardness

Hardness is the material's resistance to indentation (penetration by a hard object). It is normally measured with a Shore durometer, which measures the depth of indentation achieved with a standard indenter for a given time under a given load, according to the ASTM D2240 testing method. Different Shore scales are defined depending on the material's hardness: for hard polymers like Halar® ECTFE the Shore D scale is normally used.

Shore D hardness values for the most common fluoropolymers are reported in the following diagram.

Figure 7: Typical average Shore D hardness for common fluoropolymers

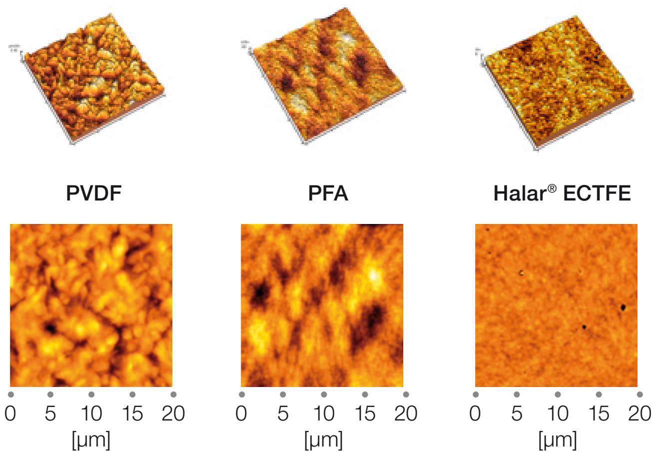


Surface Smoothness

Halar® ECTFE is distinguished from all other fluoropolymers by its exceptional surface smoothness which facilitates the shedding of particles, avoids particle trapping and helps significantly reduce the formation of biorganic films and bacterial colonies.

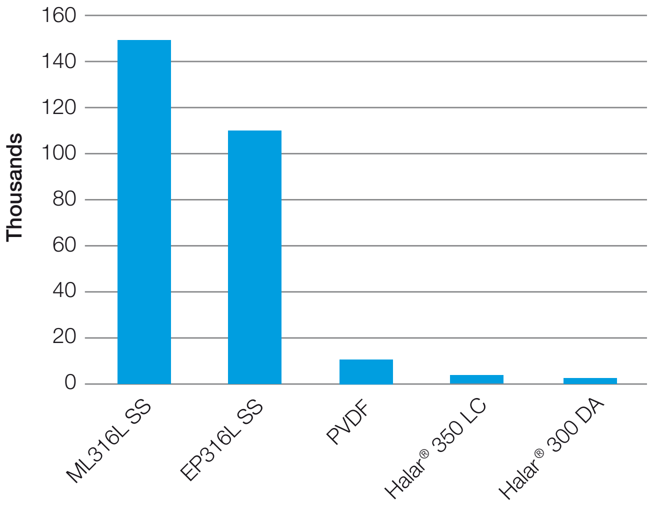
Three-dimensional images of the surface of items extruded using Halar® ECTFE and other fluoropolymers obtained by Atomic Force Microscopy (AFM) are shown in Figure 8. Typical roughness values can be extrapolated by numerical elaboration demonstrating the exceptional smoothness of Halar® ECTFE in comparison with other fluorinated materials.

Figure 8: Atomic Force Microscopy topographies of the inside surface of fluoropolymer extruded pipes



Furthermore, as shown in Figure 9, Halar® ECTFE pipes exhibit a low incidence of microbial bio-fouling, making it ideal to use in ultrapure water (UPW) applications.

Figure 9: Average direct cell count/cm^2 for some Halar® ECTFE grades in comparison with stainless steels and polyvinylidene fluoride



Coefficient of Friction

The coefficient of friction is strongly influenced by parameters such as surface roughness, sliding rate, contact pressure, lubrication, etc. According to the ASTM D1984 method, the coefficients μ_0 (static) and μ (dynamic) are evaluated under a load of 2 N (0.45 lbf) and a displacement rate of 150 mm/min. The values are reported in Table 7. Halar® ECTFE, thanks also to its particularly smooth surface, does not require any structural or surface modification to achieve a low coefficient of friction.

Table 7: Coefficient of friction for Halar® ECTFE

	Friction Coefficient	
	Static (μ_0)	**Dynamic** (μ_0)
Halar® ECTFE	0.2	0.2

Abrasion Resistance

The abrasion resistance was determined using a TABER abrasion test, which measures the wear of a material by friction on an abrasive substance. The specimen is fixed to a turning plate and in contact with an abrasive disk loaded with a weight of 9.81 N (2.21 lbf). The abrasion resistance is given by the weight lost by the specimen after a certain number of revolutions. Table 8 presents the results for Halar® ECTFE in comparison with other materials.

Table 8: Abrasion resistance of Halar® ECTFE in comparison with other materials (TABER test).

Material	Abrasive Disk	Weight Loss [mg/1,000 rev.]
Halar® ECTFE	CS-17	25 to 35
PVDF (homopolymers)	CS-10	5 to 10
	CS-17	7 to 10
PP (homopolymers)	CS-10	15 to 20
	CS-17	18 to 28
PTFE	CS-10	8 to 12

Optical Properties

Refractive Index

The refractive index of Halar® 500 at 21 °C (72 °F) for 589 nm light is n = 1.44.

Absorption Spectra

Absorption spectra in the visible, UV and IR ranges, measured on Halar® ECTFE films, are given in Figures 10 and 11.

Figure 10: Absorption spectra of Halar® 500 in UV and visible ranges

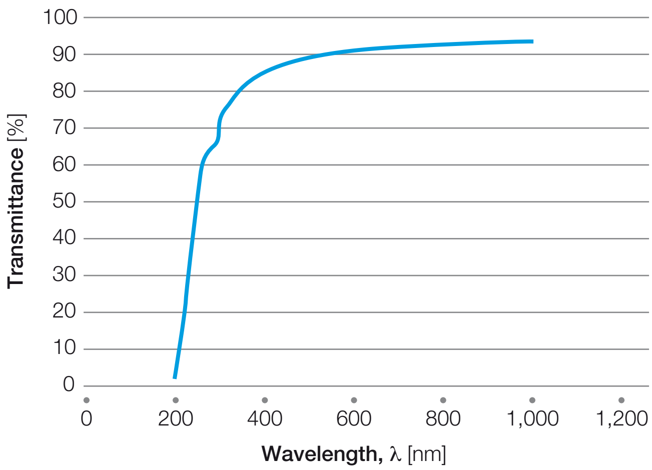
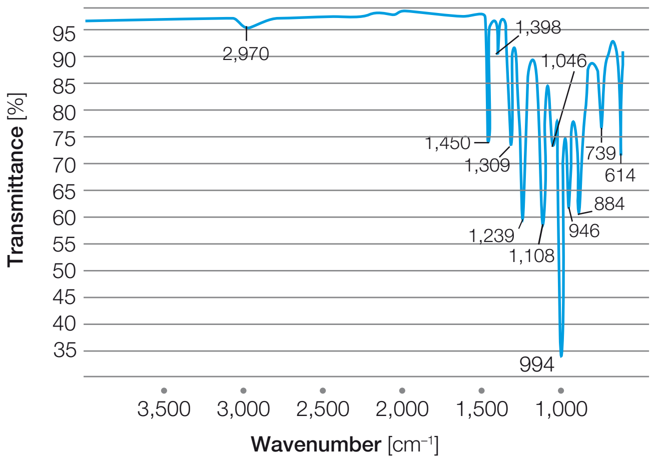


Figure 11: ATR-IT (attenuated total reflection-infrared) spectrum of Halar® ECTFE 500 thin film (transmission).



Transparency, Haze and Gloss

The Halar® ECTFE films produced for photovoltaic applications are UV stable, optically clear (with 95 %* total light transmission and 3–4 % Haze*, gloss** 110 gu at 60°)

** measured for a 50 µm film in accordance with ASTM D1003 in air*
*** measured for a 50 µm film in accordance with ASTM D2457*
(ASTM D1746, ASTM D1003 and ASTM D2457)
The optical properties in white light are measured under various aspects:

1. *Total light transmission through the object*
2. *Transparency or fraction of the transmitted light deflected by more than 0.1° of solid angle*
3. *Haze or fraction of the transmitted light deflected by more than 5° of solid angle*
4. *Gloss or luminosity depend on the processing conditions, surface quality, etc., as well as the film thickness.*

Mechanical Properties

Halar® ECTFE is a strong, hard, tough, abrasion resistant, highly impact-resistant material that retains its useful properties over a broad range of temperatures. Its low-temperature properties, especially those related to impact, are particularly outstanding. Halar® ECTFE also has good tensile, flexural and wear resistant properties. Mechanical property information is provided in the table and figures below.

Table 9: Typical mechanical properties

Properties	Halar® ECTFE	Unit	Test Method
Tensile stress at yield	30–32 (4.3–4.6)	MPa (kpsi)	ASTM D638
Tensile stress at break	40–57 (5.8–8.3)	MPa (kpsi)	
Elongation at yield	3–5	%	
Elongation at break	250–300	%	
Tensile Modulus	1,400–2,100 (203–304)	MPa (kpsi)	
Flexural strength	45–55 (6.5–8.0)	MPa (kpsi)	ASTM D790
Flexural modulus	1,600–1,800 (232–261)	MPa (kpsi)	
IZOD impact, notched at 23 °C (73 °F)	no break	J/m	ASTM D256
IZOD impact, notched at –40 °C (–40 °F)	50–110	J/m	ASTM D256
Hardness, Shore D	70–75	–	ASTM D2240
Hardness, Rockwell R	90	–	ASTM D785
Abrasion resistance	5	mg/1000 rev	TABER
Friction coefficient: static/dynamic	0.1–0.2/0.1–0.2	–	ASTM D1894

Short-term Stresses

Tensile Properties

Tensile properties are determined by clamping a test specimen into the jaws of a testing machine and separating the jaws at a specified rate in accordance with ASTM D638. The force required to separate the jaws divided by the minimum cross-sectional area is defined as the tensile stress. The test specimen will elongate as a result of the stress, and the amount of elongation divided by the original length is the strain. In Figure 13 the tensile curves of Halar® ECTFE at different temperatures are reported.

In addition, the other figures hereby reported, illustrate the behavior of important mechanical parameters with the temperature, as the tensile modulus (defined as the ratio of the uniaxial stress over the uniaxial strain in the range of stress in which Hooke's Law holds), the stress at yield (defined as the stress at which a material begins to deform plastically) and the stress at break (defined as the stress at which the failure or rupture occurs).

Figure 12: Tensile curves for Halar® ECTFE at various temperatures

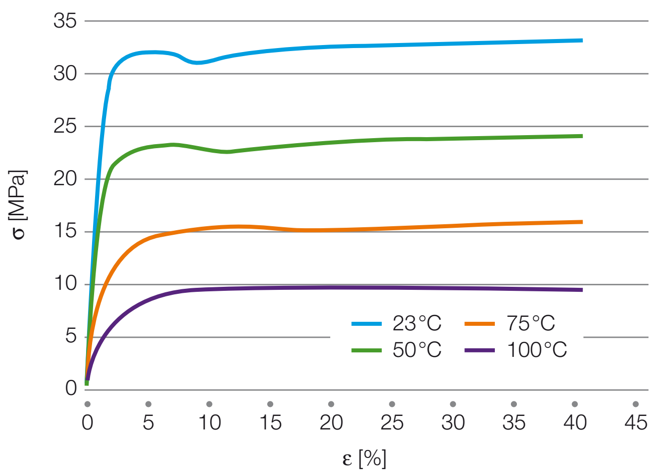


Figure 13: Tensile modulus vs. temperature for Halar® ECTFE

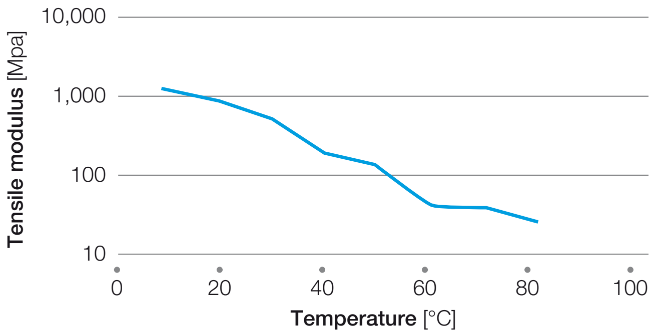
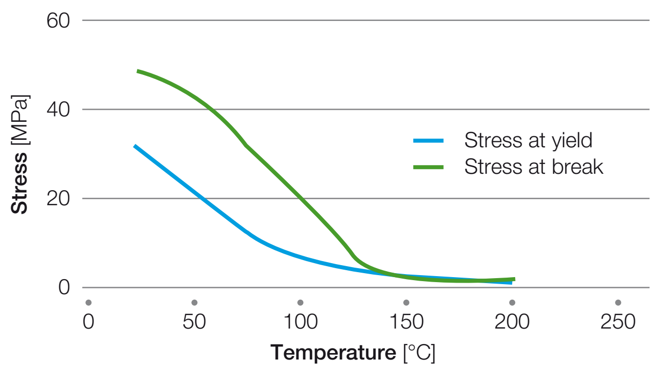


Figure 14: Stress at yield and stress at break vs. temperature for Halar® ECTFE



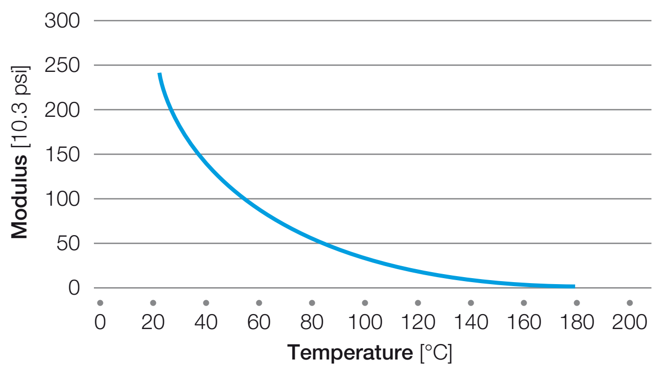
Flexural Properties

Flexural properties were determined in accordance with ASTM D790 using the three-point loading method. In this method the test specimen is supported on two points, while the load is applied to the center. The specimen is deflected until rupture occurs or the strain reaches five percent.

Flexural testing provides information about a material's behavior in bending. In this test, the bar is simultaneously subjected to tension and compression.

The mechanical flexural characteristics of Halar® ECTFE are presented in Figure 15.

Figure 15: Flexural modulus of Halar® ECTFE vs. temperature



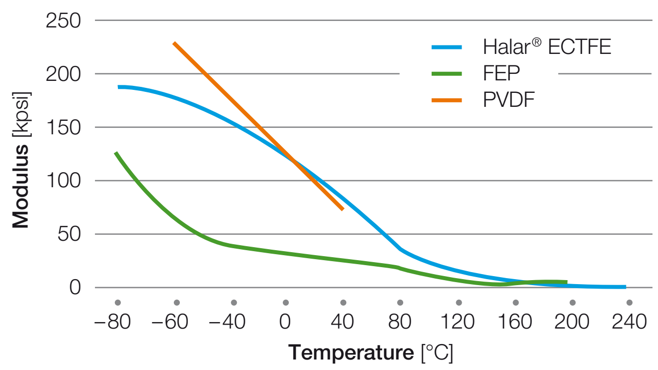
Compressive Properties

Compressive modulus was measured on a test specimen placed between parallel plates. The distance between the plates is reduced while the load required for pushing the plates together and the plate-to-plate distance is monitored.

The maximum stress endured by the specimen (this will usually be the load at rupture) is the compressive strength, and the slope of the stress/strain curve is the compressive modulus.

The behavior of the compressive modulus with temperature is reported for Halar® ECTFE in comparison with other fluoropolymers in Figure 16.

Figure 16: Compressive modulus for Halar® ECTFE and other fluoropolymers



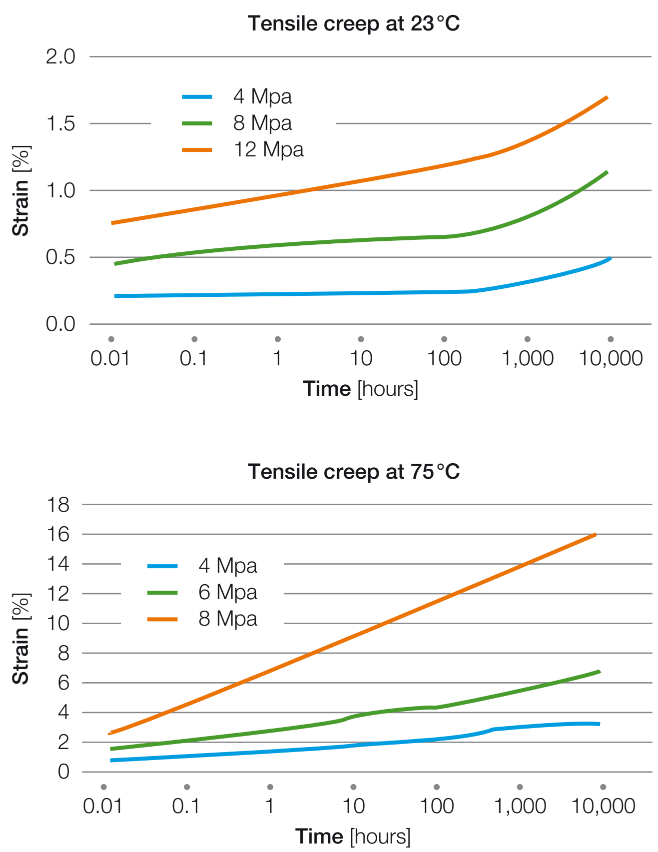
Long-term Static Stress

Creep and Stress Relaxation

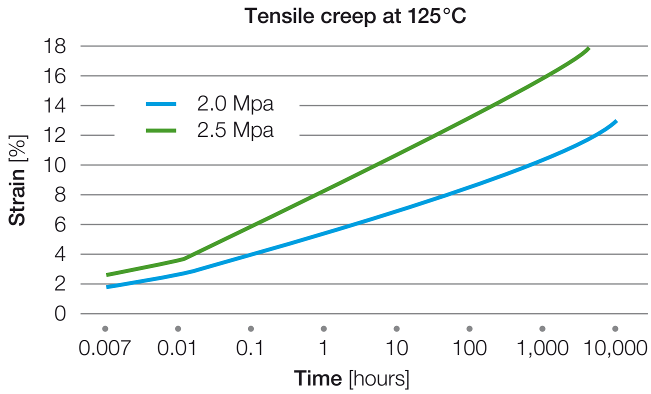
When a bar made of a polymeric material is continuously exposed to a constant stress, its dimensions will change in response to the stress. This phenomenon is commonly called "creep". When samples are measured in simple tension, the test specimen will elongate as a function of time under stress. The term "strain" is used for the amount of length increase divided by the initial length.

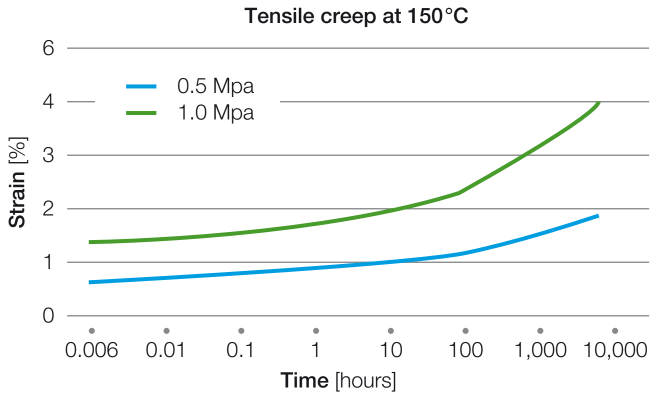
Creep can also be observed and measured in a bending or flexural mode, or in a compressive mode. The creep information presented in this manual was developed using the tensile mode.

Figures 17 and 18: Tensile creep of Halar® ECTFE at various temperatures and under different stresses



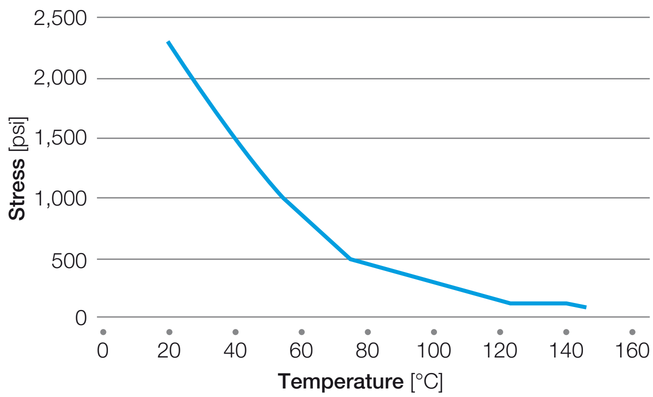
Figures 19 and 20: Tensile creep of Halar® ECTFE at various temperatures and under different stresses





Stress relaxation is defined as the reduction in strain needed to maintain a constant stress. This physical phenomenon is a uniquely measured form of creep.

Figure 21: Stress relaxation after 1,000 hours in Halar® ECTFE specimens deformed by 2 % as function of temperature



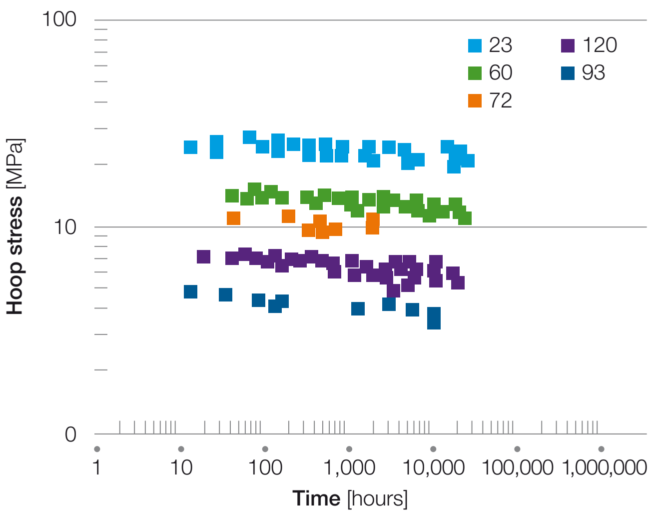
Tests on Pipe – Long-term Hoop Stress Using IPT Equipment

The long-term behavior of Halar® ECTFE pipes subjected to internal pressure has been studied lately using IPT test equipment (IPT Service GmbH, Germany). The IPT tests are generally conducted on medium diameter pipes (D = 32 mm or more), in a neutral environment (water). The pipes are maintained at test temperature with forced hot air circulation. A regulation device maintains the temperature and internal pressure constant. Any loss of water due to permeation is compensated automatically from a pressurized tank. Each test station, comprising several pipes under pressure, is equipped with a timer which is automatically cut out at each pipe failure. Figure 22 below shows the bursting hoop stress of Halar® 901 pipes vs. time in the IPT test equipment.

Usually, ductile-to-brittle transition in long-term pipe testing is associated with a change of slope in the Log (stress) vs. Log (time) plot ("knee"). No evident knee could be found in the figure based on current burst pressure data.

However, it is necessary to be extremely cautious when using this data and its regression out of the ductile failure area, as previous experience on other fluoropolymers has shown that the presence of a knee may depend on experimental testing conditions.

Figure 22: Long-term hoop stress of Halar® 901 pipes using IPT test equipment (ISO 10931-2).



Values for pipes in water service only. For any other fluid a thorough chemical resistance analysis should be carried out.

Dynamic Loading

Alternating Low Amplitude, Short-term Stresses DMTA (Dynamic Modulus, ASTM D4065)

ECTFE, like all thermoplastics, behaves as a viscoelastic material. Under the effect of a stress, the response (deformation) includes an elastic component and a viscous component.

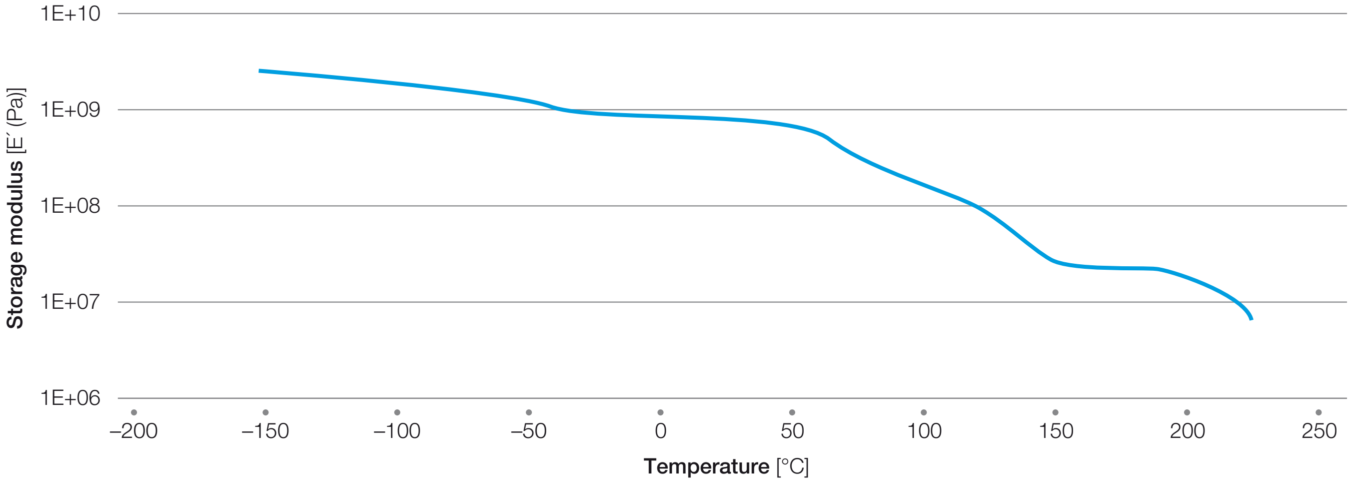
Under a forced harmonic stress system, the amplitude and the phase displacement of the resulting deformation are measured. When it is performed over a wide range of temperatures, this method of evaluation makes it possible to identify the thermomechanical spectrum of the material at a given frequency, characterized by:

- The temperature variation of the elastic modulus E' (real or purely elastic component of the complex modulus E*, where E* = E' + iE")
- The variation of the mechanical damping (or loss) tg δ as a function of the temperature. tg δ is the ratio of the viscous (E") and elastic (E') components:

$$tg\,\delta = \frac{E''}{E'}$$

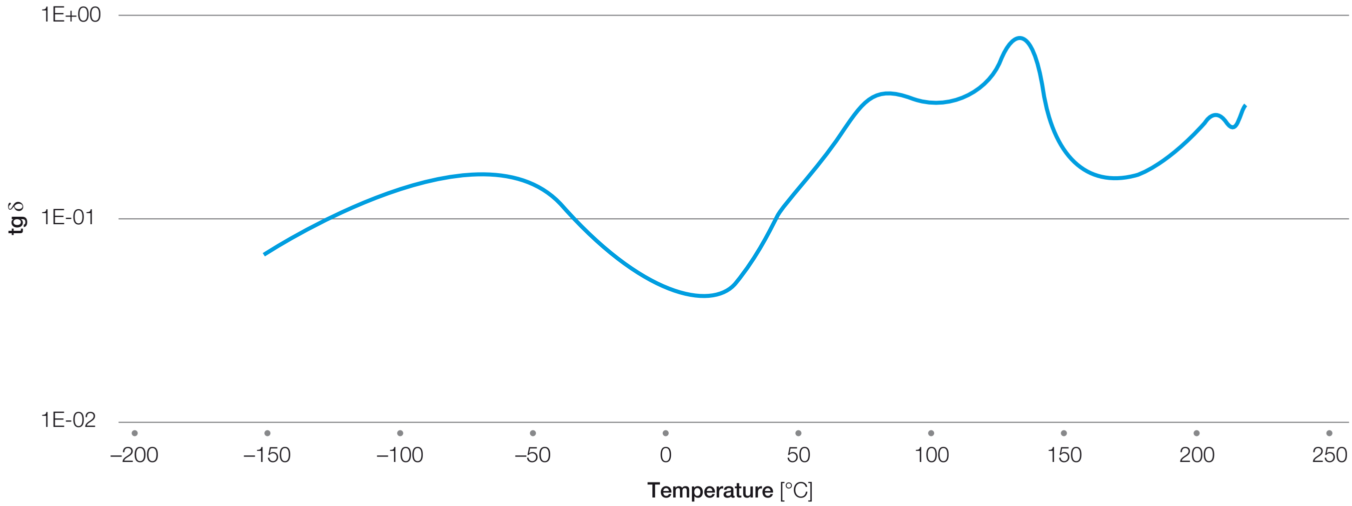
The curve of tg δ displays peaks which correspond mainly to the second-order transitions, the most important of which is the glass transition (due to the amorphous phase). These transitions are the result of progressive liberations of movements of molecular segments (greater or smaller depending on the transition) when the temperature rises (thermal agitation). The DMTA (dynamic mechanical thermo-analysis) technique was used to characterize the Halar® ECTFE samples, in torsion rectangular geometry with a frequency of 1 Hz. Figures 23 and 24 show the curves of E' and damping (tg δ) respectively of some Halar® polymers.

Figure 23: Storage modulus E' of Halar® ECTFE vs. temperature (DMTA)



ARES data, torsion rectangular, dynamic temperature ramp (2°C/60 s), frequency (const.) = 6.28 Hz

Figure 24: Damping (or tg δ) of Halar® ECTFE vs. temperature (DMTA)



ARES data, torsion rectangular, dynamic temperature ramp (2°C/60 s), frequency (const.) = 6.28 Hz

Impact Strength

Several methods are used to measure the impact resistance of plastics, such as the Izod and Charpy tests presented below. These impact tests allow designers to compare relative impact resistance under controlled laboratory conditions and, consequently, are often used for material selection or quality control.

Izod Impact Strength

The notched Izod test (ASTM D256) is one of the most widely employed methods for comparing polymeric materials. In this test, a notched specimen is clamped at one end ("cantileverbeam") and then struck at the other end by a swinging pendulum. At the point of impact, the striker has a known amount of kinetic energy. The impact energy is calculated from the excess energy remaining in the pendulum after breaking the specimen.

Almost all the Halar® ECTFE grades tested under the standard conditions of the Izod impact test (notched V indented specimens 10 mm) presented no break at room temperature and an impact strength of 207 J/M (2.0 ft·lbf/in) at a temperature of –40 °C (–40 °F).

Brittleness Temperature

This test method covers the determination of the temperature at which plastics exhibit brittle failure under impact conditions specified in the ASTM D746 standard. To determine the brittleness temperature, specimens secured to a specimen holder are immersed in a bath containing a heat transfer medium that is cooled. The specimens are struck at various temperatures at a specified linear speed and then examined. The brittleness temperature is defined as the temperature at which 50 % of the specimens fail.

The brittleness temperatures measured on 2 mm pressed sheets of various Halar® ECTFE grades are below –76 °C (below –105 °F).

Tear Resistance of Films

The tearing resistance, i.e. the resistance of a material to be broken apart by force, without the aid of a cutting tool, is important particularly in thin film applications.

Tearing initiation resistance was measured for Halar® ECTFE extruded films in the machine and in the transversal direction; and compared with other fluoropolymers used in thin film applications by means of two different standard conditions, reported below.

The results are presented in Table 10 and 11.

Table 10: Tearing resistance of Halar® ECTFE and ETFE polymer (ethylene tetrafluoroethylene) in 100 µm extruded films at room temperature, ASTM D 624 die C, speed 500 mm/min, grip distance 60 mm)

		Load/Thickness [N/mm]	
Material (Extruded Films)	Thickness [µm]	Machine Direction	Transversal Direction
Halar® 500	100	213.0	222.5
ETFE	100	158.0	170.5

Table 11: Tearing resistance of Halar® ECTFE and PVF polymer (Polyvinyl fluoride) in extruded thin films at room temperature, ASTM 1004, speed travel 500 mm/min

			Load/Thickness [N/mm]	
Material (Extruded Films)	Test Temperature [°C (°F)]	Thickness [µm]	Machine Direction	Transversal Direction
Halar® 500	23 (73)	8	190	199
PVF	23 (73)	12	118	175

Electrical Properties

General Characteristics

Halar® ECTFE standard and modified polymers exhibit high bulk and surface resistivities, high dielectric strength, low dielectric constant, and moderate dissipation factor; which make them suitable for applications requiring electrical insulators.

The dissipation factor varies slightly for frequencies above 1 kHz. The dielectric constant of Halar® ECTFE is stable across broad temperature and frequency ranges. Halar® ECTFE can be used as jacketing of plenum rated cables in more demanding applications. Its excellent electrical properties simplify the design of high-performance cables. The very low moisture absorption properties of Halar® ECTFE and the temperature insensitivity ensure that cables utilizing Halar® ECTFE jackets maintain their electrical performance under a wide variety of environmental conditions. PVC jacketed cables have been shown to deteriorate significantly in electrical performance due to moisture absorption during aging. The low temperature ductility of Halar® ECTFE allows installation in cooler temperatures without cracking and splitting.

The typical average electrical properties of Halar® ECTFE are reported in Table 12.

Table 12: Halar® ECTFE typical electrical properties

Properties	ASTM	Halar® ECTFE
Volume resistivity (Ω·cm)	D 257	> 10^{15}
Surface resistivity (Ω)	D 257	> 10^{14}
Dielectric strength at 1mm thickness (kV/mm)	D 149	30–35
Relative dielectric constant	D 150	
at 1 kHz		2.5
at 1 MHz		2.6
Dissipation Factor	D 150	
at 1 kHz		0.0016
at 1 MHz		0.015

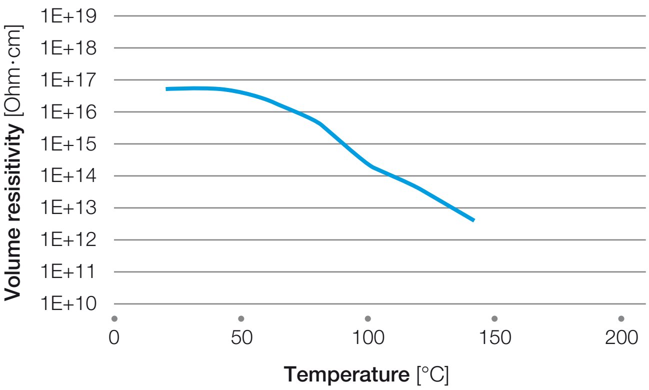
Many applications for thermoplastic polymers depend upon their ability to function as electrical insulators. Several tests have been developed to provide the designer with physical parameters that help to predict how well a particular polymer can perform that function.

Volume Resistivity

Volume resistivity is defined as the electrical resistance offered by a material to the flow of current, multiplied by the cross sectional area of current flow per unit length of current path. The volume resistivity test is run by subjecting the material to 500 volts for 1 minute and measuring the current. The higher the volume resistivity, the more effective a material will be in electrically insulating components.

Figure 25 shows the trend of volume resistivity versus temperature for Halar® ECTFE.

Figure 25: Volume resistivity vs. temperature for Halar® ECTFE

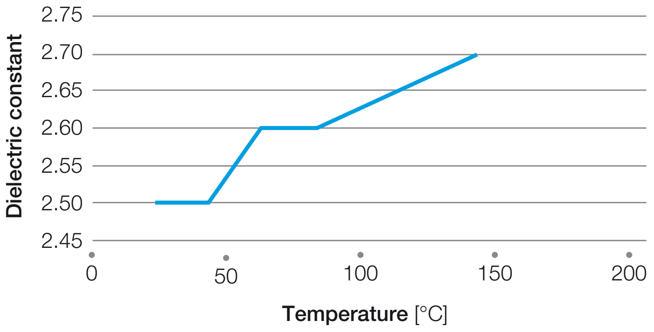


Dielectric Constant

Dielectric constant is defined as the ratio of the capacitance of a condenser using the test material as the dielectric versus the capacitance in air. Insulating materials are used in two very distinct ways: (1) to support and insulate components from each other and from being grounded, (2) to function as a capacitor dielectric. In the first case, it is desirable to have a low dielectric constant. In the second case, a high dielectric constant allows the capacitor to be physically smaller.

Figure 26 depicts the trend of the dielectric constant with temperature for Halar® ECTFE.

Figure 26: Dielectric constant vs. temperature for Halar® ECTFE

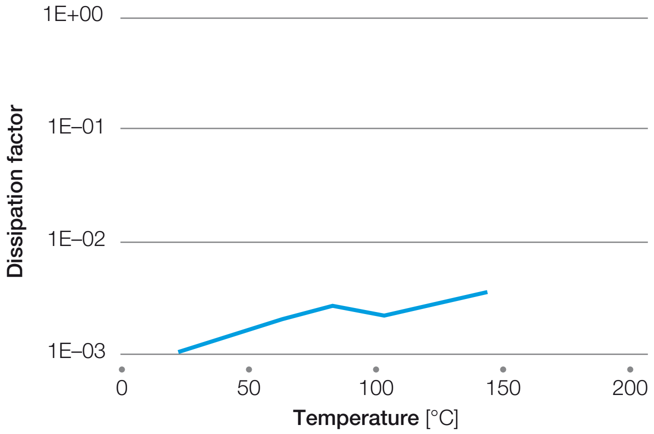


Dissipation Factor

Dissipation factor (also referred to as loss tangent or tan δ) is a measure of the amount of heat (energy) dissipated by a material under alternating voltage. Low dissipation factors are desirable in most cable applications; especially with communication LAN copper wires.

The dissipation factor as a function of temperature is presented in Figure 27 for Halar® ECTF.

Figure 27: Dissipation factor vs. temperature for Halar® ECTFE



General Chemical Resistance Properties

Halar® ECTFE demonstrates excellent overall chemical resistance. In general only a few substances are known to chemically attack Halar® ECTFE and only a limited number of chemicals can significantly swell the polymer leading to a worsening of the performance of the material.

Halar® ECTFE fluoropolymer is especially resistant to:

- Strong and weak inorganic acids and bases
- Weak organic acids and bases
- Salts
- Aliphatic hydrocarbons
- Alcohols
- Strong oxidants
- Halogens

However, Halar® ECTFE, particularly at high temperatures, can significantly swell in contact with some:

- Esters
- Aromatic hydrocarbons
- Ethers
- Ketones
- Amides
- Partially halogenated solvents

Halar® ECTFE can be attacked by amines, molten alkali metals, gaseous fluorine, and certain halogenated compounds such as ClF_3.

Chemical attack and swelling are very complex phenomena. The known factors affecting chemical suitability of Halar® ECTFE or any other plastic for a chemical application, not listed in order of priority, are the following:

- Specific chemical or mixture composition
- Temperature and temperature variation
- Concentration of the attacking chemical which may be a compound that is completely different from the individual components
- Exothermic energy or heat of reaction or mixing pressure, due primarily to the effect of pressure on the concentration of a reactive gas
- Time of exposure
- Stress levels
- Velocity
- Suspended solids
- Thickness
- Electromagnetic force (EMF) potential of the supporting metal compared to the ground potential

The recommended procedure to determine suitability of Halar® ECTFE is as follows:

- Determine as accurately as possible the chemicals at issue
- Determine the maximum temperature and the normal operating temperature
- Review the maximum recommended temperature from the list provided

The maximum recommended temperatures listed below typically refer to the exposure of non-stressed parts; if relevant stresses are present, a more severe effect on the material should be taken into account.

Moreover, the effect of synergism or reaction or complex formation with mixtures cannot be predicted by the table. In any case, appropriate chemical resistance tests using a representative sample of the media should be performed.

The following table presents an overview of the chemical resistance of Halar® ECTFE to most common chemicals.

Please note that the present document provides the reader a substantial overview. Nevertheless, in case of any doubt, please contact Solvay Specialty Polymers for further information.

Table 13: Overview of the chemical resistance of Halar® ECTFE

Chemical	Formula	Concentration	Max. Temp. [°C]
Acids			
Hydrochloric	HCl	37 %	150
Hydrofluoric	HF	50 %	150
Nitric	HNO_3	65 %	66
Phosphoric	H_3PO_4	85 %	150
Sulphuric	H_2SO_4	98 %	125
		oleum	23
Bases			
Ammonium hydroxide	$NH_4(OH)$	30 %	150
Potassium hydroxide	KOH	30 %	121
Sodium hydroxide	$NaOH$	50 %	121
Sodium hypochlorite	$NaClO$	5% - stabilized at pH 12	150
Hydrocarbons			
n-Hexane	$CH_3(CH_2)_4CH_3$	100 %	150
Toluene	$C_6H_5CH_3$	100 %	66
Alcohols and Ethers			
Methanol	CH_3OH	100 %	65
Ethanol	CH_3CH_2OH	100 %	140
Organic acids, esters and Ketones			
Acetic acid	CH_3COOH	100 %	> 100
		50 %	> 121
Acetone	CH_3COCH_3	100 %	66
Acetophenone	$C_6H_5COCH_3$	100 %	50
Ethyl Acetate		100 %	50
Classic Polymer Solvents			
Dimethyl formamide	$CH_3CON(CH_3)_2$	100 %	50
Dimethyl sulphoxide	CH_3SOCH_3	100 %	> 100
N-Methylpyrrolidone		100 %	25
Halogenated Solvents			
Chlorobenzene	C_6H_5Cl	100 %	66
Chloroform	$CHCl_3$	100 %	not resistant
Amines and nitriles			
Acetonitrile	CH_3CN	100 %	> 100
Aniline		100 %	100
Dimethyl amine		100 %	25
Peroxides			
Hydrogen peroxide	H_2O_2	30 %	> 88
Fluids used in the automotive industry			
Crude oil		100 %	150
Dexron II (gear oil)		100 %	150
Gasoline		100 %	150
Diesel Fuels		100 %	150
Mineral oil		100 %	150

Permeability

In general Halar® ECTFE offers excellent permeation resistance to many chemicals. Barrier properties strongly depend on the nature of the chemicals present in the environment and an overview on the permeation properties of the material can be given according to the features of the penetrating substance.

Gases

Halar® ECTFE has excellent permeation resistance to simple gases.

Figure 28 shows the permeability coefficients of hydrogen, nitrogen, oxygen and ammonia in Halar® ECTFE as a function of temperature. For simple gases – which do not form specific interactions with the polymer chains – permeability increases with decreasing molecular dimensions. Permeability of the polar molecule NH_3, on the other hand, is higher than expected when simply basing it on its size.

Figures 29 and 30 show the permeability coefficients of chlorine and hydrogen sulfide in Halar® ECTFE compared to other fluorinated and hydrogenated materials.

Figure 28: Gas permeability in Halar® ECTFE

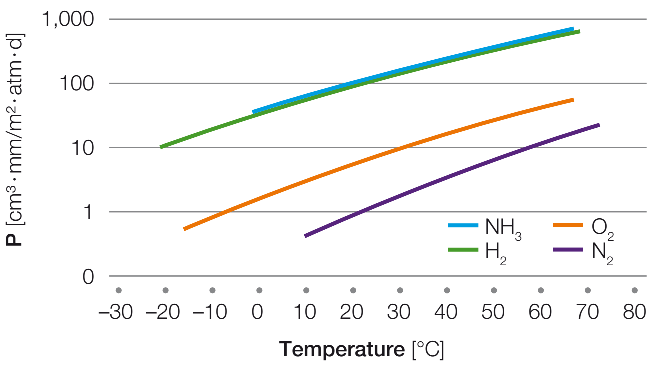


Figure 29: Chlorine permeability of Halar® ECTFE compared with other polymers

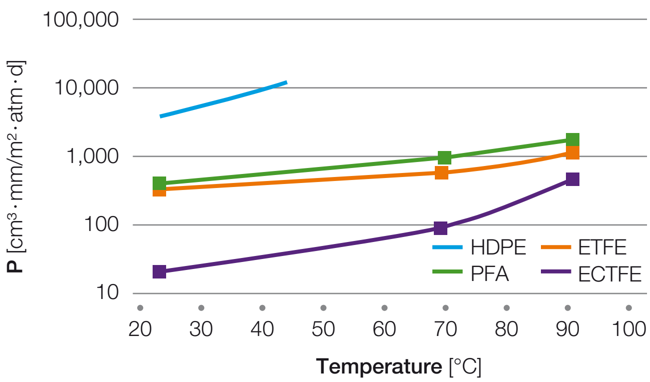
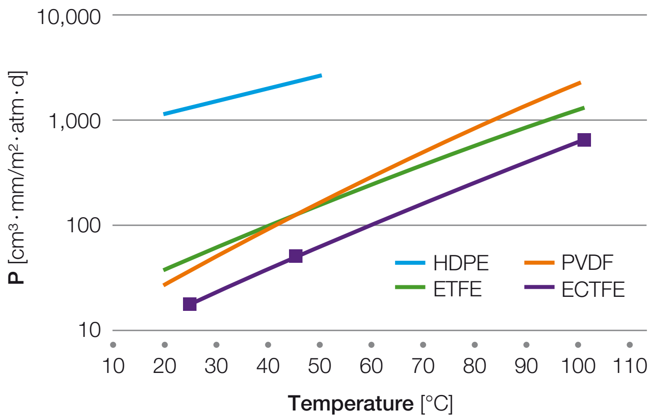


Figure 30: Hydrogen sulfide permeability of Halar® ECTFE compared to other polymers



Water

Water is a small, polar molecule that can interact with polymer chains forming hydrogen bonds. Permeation resistance of Halar® ECTFE to water vapor is better than other fluoropolymers.

Water vapor permeability in Halar® ECTFE is about 750 $cm^3 \cdot mm/m^2 \cdot atm \cdot d$ at 23 °C (73 °F) and 7,600 $cm^3 \cdot mm/m^2 \cdot atm \cdot d$ at 90 °C (195 °F).

The following graphs, compare the water vapor permeability of Halar® ECTFE with other polymers and fluoropolymers at room temperature and at 90 °C (195 °F)

Figure 31: Water vapor permeability comparison of different polymers at 23 °C (73 °F)

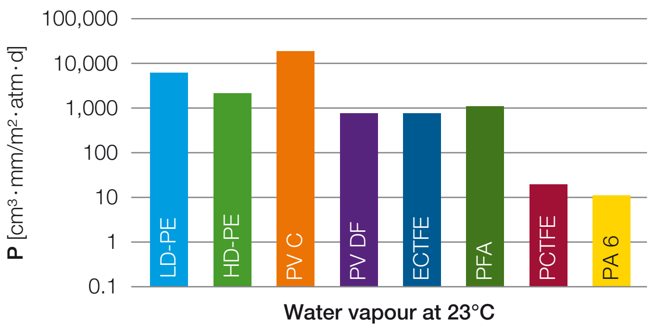
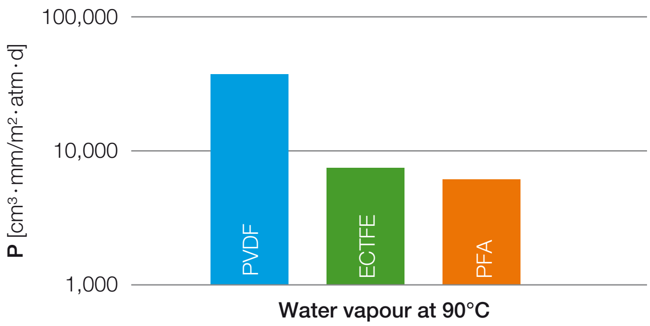


Figure 32: Water vapor permeability comparison of different polymers at 90 °C (195 °F)



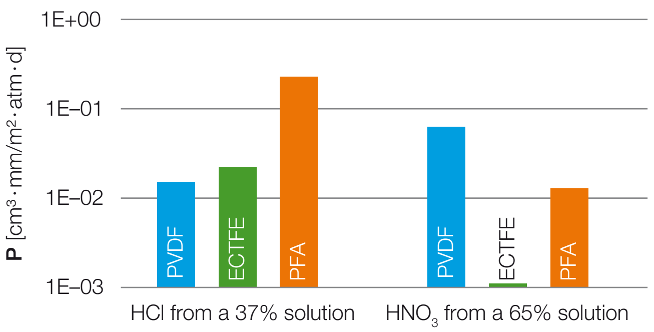
Aqueous Electrolytes

The permeation of electrolytes $A^{n+}{}_xB^{m-}{}_y$ in Halar® ECTFE – as in hydrophobic fluoropolymers – involves the passage of the neutral substance A_xB_y and not the ions A^{n+} and B^{m-}.

In general the permeability coefficients of electrolytes are low even from concentrated solutions and are related to the volatility of the electrolyte: only volatile substances have a non negligible permeation rate, while the permeation of non volatile electrolytes cannot be detected even after years.

However, when considering the permeation of aqueous solutions, the permeation of water discussed above should be considered. As shown in Figure 33, Halar® ECTFE posses outstanding resistance to electrolyte permeation even compared to other partially fluorinated and perfluorinated polymers.

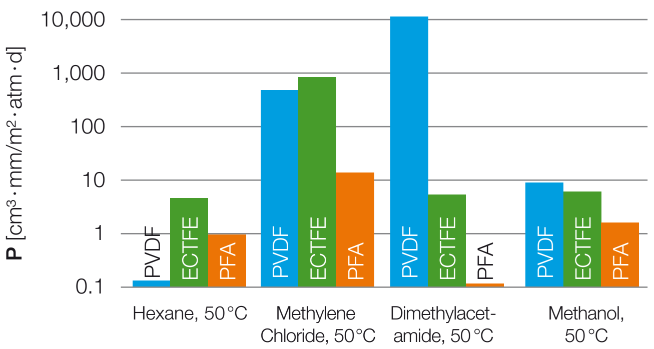
Figure 33: Permeabilities of HCl and HNO_3 molecules in fluoropolymers from aqueous solutions



Organic Chemicals

As the permeation process can be described as the sorption of the penetrating substances on the material surface followed by its diffusion through the polymer chains, the link between permeability and swelling must be clear: chemicals that are known as swelling agents for Halar® ECTFE (see the section above) are also expected to have a significant permeation rate in the polymer.

Figure 34: Liquid permeabilities of a few common chemicals in Halar® ECTFE, compared with other fluoropolymers



Weathering Resistance

Halar® ECTFE undergoes very little change in properties or appearance upon outdoor exposure to sunlight. Both accelerated and outdoor weathering studies demonstrate the remarkable stability of the polymer when exposed to UV light and weather. Mechanical and optical properties of Halar® ECTFE are barely affected after 9,000 hours exposure to the UVB-313 source of light in the Q-UV Weatherometer*, after 10,000 hours exposure to Xenon Arc lamp Weatherometer** or after 9 years of the Florida outdoor weathering. The Figures 35 and 36 illustrate the exceptional weathering resistance of Halar® ECTFE films.

All of this makes Halar® ECTFE a suitable material for long-term outdoor exposure applications, including: photovoltaic flexible frontsheets, photovoltaic backsheet laminate components and architectural tension membranes.

Notes:

* *Q-UV Panel conditions: 8 hours UVB-313 lamps at 70 °C and 4 hours dark condensation at 50 °C*

** *WOM. ci35 conditions: Xenon Arc lamps, irradiance 0.35 W/m², black panel: 60 °C inner and outer filter in borosilicate, no dark cycle, no rain cycle*

Figure 35: Optical properties of Halar® ECTFE films under 9,000 hours of QUV weatheromoter

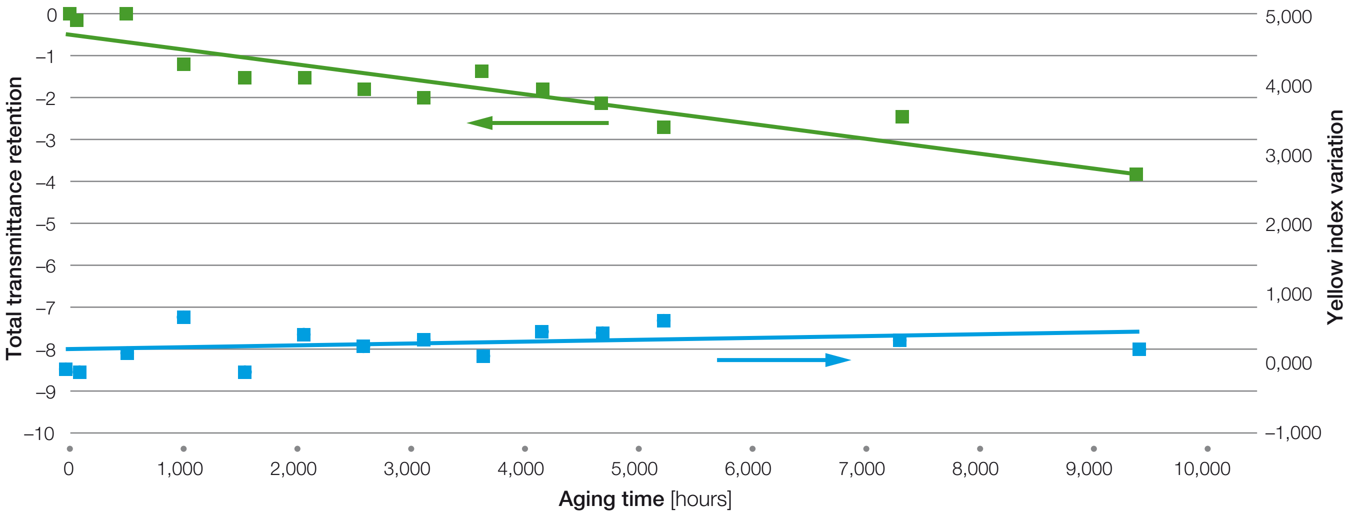
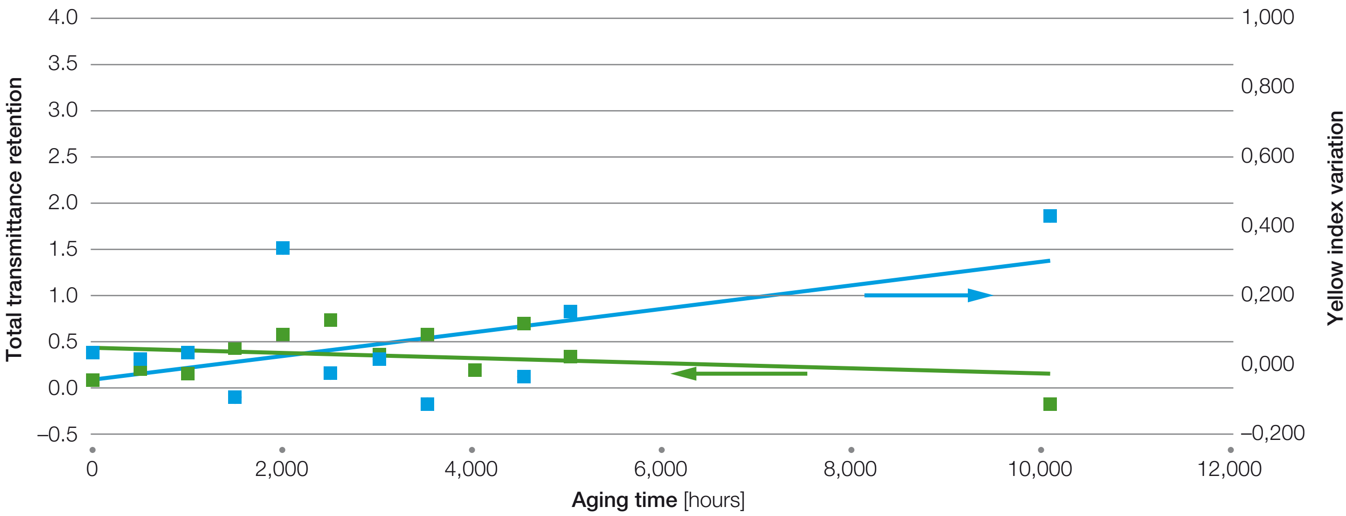


Figure 36: Optical properties of Halar® ECTFE films under 10,000 hours of Xenon Arc weatheromoter



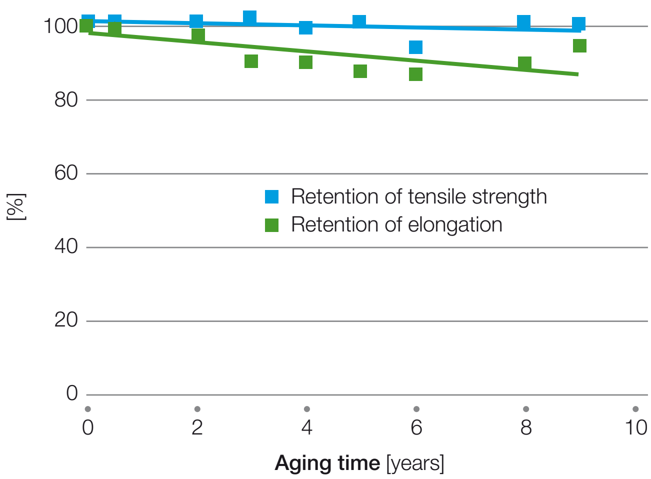
Resistance to High Energy Radiation

The first point to consider, when evaluating the resistance to energetic radiation of a particular material, is the amount of radiation with which it comes into contact

In general, Halar® ECTFE has demonstrated excellent resistance to many sources of radiation up to 200 Mrad. However, care must be taken for the continuous exposure of the polymers to gamma radiation as it is very energetic and thus it can affect the long-term performance of the materials.

Please note that, as in the case of chemical attack, the effect of such modification is cumulative and, if the irradiation is repeated in time, may lead to severe damage.

Figure 37: Mechanical property variations of Halar® ECTFE films after 9,000 hrs. in Q-UVb panel



Fire Resistance

Halar® ECTFE offers a superior combination of properties when compared to other partially fluorinated plastics, according to the following independent tests:

- UL 94
- Limiting oxygen index (LOI)
- Auto ignition temperature
- Factory Mutual (FM)

When placed in a flame, unlike most thermoplastics, Halar® ECTFE does not melt or drip. Char is formed, which serves as an oxygen and heat transfer barrier. On removal of the flame, it immediately extinguishes. It will not ignite or propagate flame in atmospheres which contain up to 52 % of oxygen. Halar® ECTFE has excellent low smoke properties.

UL 94	V–0 Rating at 0.18 mm
Limiting Oxygen Index (ASTM D 2863)	> 52 %
Auto-Ignition Temperature (ASTM D1929)	655 °C
Factory Mutual (FM 4910)	Compliant for Halar® 901 grade

There are two types of pre-selection test programs conducted on plastic materials to measure flammability characteristics.

The first determines the material's tendency either to extinguish or to spread the flame once the specimen has been ignited; this program is described in the UL-94 standard. Specimens molded from the plastic material are oriented in either a horizontal or vertical position, depending on the specifications of the relevant test method, and are subjected to a defined flame ignition source for a specified period of time. The vertical rating V–0 indicates that the material was tested in a vertical position and self-extinguished within the shortest burn time after the ignition source was removed, and didn't drip flaming particles, showing highest safety.

The second test program measures the ignition resistance of the plastic to electrical ignition sources. The material's resistance to ignition and surface tracking characteristics is described in the UL 746A standard.

The basic tests used to evaluate a material's ability to resist ignition are:

- Hot-Wire Ignition (HWI): this test determines the resistance of plastic materials to ignition from an electrically heated wire
- High-Current Arc Ignition (HAI):this test measures the relative resistance of insulating materials to ignition from arcing electrical sources
- High-Voltage Arc Tracking Rate (HVTR): this test determines the susceptibility of an insulating material to track or form a visible carbonized conducting path over the surface when subjected to high-voltage, low current arcing.
- High-Voltage, Low-Current Dry Arc Resistance (D495): this test measures the time that an insulating material resists the formation of a conductive path due to localized thermal and chemical decomposition and erosion.
- Comparative Tracking Index (CTI): this test determines the voltage that causes a permanent electrically conductive carbon path after 50 drops of electrolyte have fallen on the material.

Halar® ECTFE grades tested according to the UL standard 746A are 300 and 500, and the values are reported in Table 14.

Table 14: Ignition resistance according to UL standard 746A

Thickness [mm]	Flame Class	HWI	HAI	HVTR	D495	CTI
0.18	V-0	–	–	2	7	0
1.5	V-0	2	0	2	7	0
3.0	V-0	2	0	2	7	0

Limiting Oxygen Index – LOI

The oxygen index is defined by ASTM D 2863 as the minimum concentration of oxygen, expressed as volume percent, in a mixture of oxygen and nitrogen that will support flaming combustion of a material initially at room temperature under the conditions of this method.

Since ordinary air contains roughly 21 percent oxygen, a material whose oxygen index is appreciably higher than 21 is considered flame resistant because it will only burn in an oxygen-enriched atmosphere.

Accordingly, Halar® ECTFE polymers are considered good flame resistant materials, as shown in Table 15.

Table 15: Limiting Oxygen Index (LOI) for Halar® ECTFE in comparison with ETFE polymer

	Halar® ECTFE	ETFE
LOI	> 52 %	32 %

UL Thermal Index (RTI)

Halar® ECTFE has been investigated with respect to retention of certain critical properties, according to UL Standard 746B. The end-of-life of a material is assumed to be the time when the value of the critical property has decreased to 50 percent of its original value. The Relative Temperature Index (RTI) is defined as the maximum service temperature for a material at which it will not be unacceptably compromised through chemical degradation.

More than one RTI may be appropriate for a given material depending on the property requirements for a given application:

- RTI Elec: Electrical RTI, associated with critical electrical insulating properties
- RTI Mech Imp: Mechanical Impact RTI, associated with critical impact resistance, resilience and flexibility properties
- RTI Mech Str: mechanical Strength RTI, associated with critical mechanical strength where impact resistance, resilience and flexibility are not essential

Halar® ECTFE grades tested according to the UL standard 746A are 300 and 500, and the values are reported in Table 16.

Table 16: UL thermal index (RTI) for Halar® ECTFE

Thickness [mm]	RTI Elec	RTI Mech Imp	RTI Mech Str
0.18	150	150	150
1.5	160	150	160
3.0	160	150	160

Safety, Hygiene, Health Effects

Fluoropolymers like Halar® ECTFE are known for their high chemical stability and low reactivity.

Where toxicological studies have been conducted on fluoropolymers, no significant findings for human health hazard assessment have been reported. None of the fluoropolymers is known to be a skin irritant or sensitizer in humans.

Following massive exposure to fluoropolymer dust by inhalation, increases in urinary fluoride were produced; however, no toxic effects were observed.

Some Halar® ECTFE polymers are formulated with additives such as fillers, pigments, stabilizers, etc, to provide favourable processing, or other characteristics. These additives may present other hazards when using the polymers.

The Safety Data Sheet, available for each of the commercial grades, should be consulted for specific health information and all the necessary safety instructions should be followed.

For further details, please consult the brochure "Guide for the Safe Handling of Fluoropolymers".

Toxicity of Decomposition Products

The main Halar® ECTFE grades must be processed at temperatures between 260 °C and 280 °C. Under these conditions, there is no risk of decomposition of the ECTFE polymer (except in the presence of contaminants).

In general, it is important to ensure good ventilation in the workplace. In order to avoid decomposition, it is imperative that the material should not be heated to a temperature above 350 °C. The main fluorinated product emitted during combustion is hydrofluoric acid (HF) which is dangerous if inhaled or if it comes into contact with the skin or mucous membranes.

As an indication with respect to HF, the ACGIHTLV-Ceiling value (the concentration that should not be exceeded during any part of the working exposure) is 2 ppm (1.7 mg/m^3), the indicative occupational exposure limit values established by Directive 2000/39/EC is 3 ppm (2.5 mg/m^3) for short-term (15 minutes) exposure period and the IDLH (Immediately Dangerous to Life or Health Concentrations) value set by NIOSH is 30 ppm.

In the event of fire, it is preferable to extinguish it with sand or extinguishing powder; the use of water may lead to the formation of acidic solutions.

Introduction

Halar® ECTFE is a melt-processable fluoropolymer that can be processed in the same way as conventional thermoplastics. However, some particulars have to be taken into account. Basic processing recommendations are described below.

As a general reference about safety when processing Halar® ECTFE polymers, please consult the "Guide to the Safe Handling of Fluoropolymers" published by PlasticsEurope (Brussels, Belgium) or by the Society of the Plastics Industry (Washington, DC).

All Halar® ECTFE melt processable grades are in pellet form; in addition, a powder grade (Halar® 5001C) is available for compounding purposes.

Construction Materials

All parts coming into contact with hot Halar® ECTFE polymer should be made using corrosion resistant materials such as "Xaloy" 306, B.C.I. No.2, Duranickel, or Hastelloy C. The hoppers, slides and throats should be sufficiently corrosion resistant so that rust is not introduced into the polymer. It is especially important to prevent contact of the melt with copper alloys and unprotected tool steel which can reduce the melt stability of the polymer. However, corrosion testing on metal plaques of carbon steel show that the current Halar® ECTFE technology reduces the corrosivity of the polymer.

Table 17: Extruder type

Machine size	No limitation
Length/Diameter ratio	20:1 – 30:1
Barrel heating	Standard heating methods, three or more zones
Flange heating	Required
Screw type	Single flight Compression ratio 2.5:1 – 3:1 Metering length: 25% Smooth transition (at least 3–4 flights)
Breaker plate	Recommended
Screen pack	60, 80, 100 mesh (optional)
Drive	Adjustable from 5 to 100 rpm
Melt thermocouple	Recommended
Pressure gauge	Recommended

General Considerations

Temperatures should be set to produce a melt temperature in the range of 260 to 280 °C (500 to 540 °F). At startup, the melt is kept at the low end of the temperature range. When all equipment is running satisfactorily, the melt temperature is adjusted to produce the best extrudate. At the end of all runs, the Halar® ECTFE polymer should be purged from the machine and the temperature lowered below 200 °C (400 °F).

Handling

No special treatment is required. Drying is unnecessary since the polymer will not absorb water. The low water absorption inhibits the dissipation of frictional static charges. Consequently, the polymer container should be covered at all times to prevent the deposition of contaminants on the pellets or powder. When bringing the polymer from a colder room, the closed drum liner should not be opened until the polymer has reached the temperature of the processing room. This avoids condensing atmospheric moisture on the pellets or the powder.

Regrind

Regrind can be used with no significant loss in properties below 15 % of the total composition. Regrind which has excessively darkened should be discarded.

Safety

Please refer to the Halar® ECTFE Safety Data Sheet for detailed recommended procedures for safe handling and use. As with all polymeric materials exposed to high temperatures, good safety practice requires the use of adequate ventilation when processing Halar® ECTFE. Ventilation should be provided to prevent exposure to fumes and gases that may be generated. Excessive heating may produce fumes and gases that are irritating or toxic.

Thermal Stability

Although Halar® ECTFE polymer is a stable material, degradation can occur if the maximum recommended processing temperature is exceeded. Degradation is a function of time, temperature and nature of the metal surface in contact with the molten polymer. Development of a grey-tan color in the extrudant serves as a warning sign that degradation is occurring. Black specks in the extrudant indicate severe localized degradation at hot spots or spots in the system. If black specks appear in the extrudant, it is recommended to shut down and thoroughly clean the equipment.

Temperature Limitations

Thermogravimetric analysis (TGA) of Halar® ECTFE polymer indicates that the polymer decomposes thermally at 350 °C (662 °F). Thermal decomposition can also be expected at lower temperatures if the exposure time is long enough (e.g., excessive residence time that may be encountered in extruders and injection molding machines). In practice, discoloration, black specks, etc. may be encountered when the melt temperature exceeds 300 °C (575 °F) for an extended period of time. If interruptions in processing occur, the polymer should be purged immediately from the barrel. Polypropylene or high density polyethylene may be employed for this purpose. If purging is not possible, the temperature should be lowered to 200 °C (400 °F) while repairs are being made.

Recommendations for Extrusion

Corrosion-resistant materials are recommended for all surfaces in contact with hot polymer. Halar® ECTFE polymer can thermally degrade to HCl which is corrosive to metal surfaces. Studies have indicated that the polymer begins to degrade after 45 minutes at 270 °C (520 °F); thus, residence times in extruders should be held to a minimum and care should be taken not to overheat Halar® ECTFE polymer during processing.

Corrosion-resistant materials for construction are recommended not only to ensure reasonable equipment life but also to protect Halar® ECTFE polymer from degradation. Molten Halar® ECTFE polymer will decompose on extended contact with iron, copper or brass. The products of decomposition are a black degraded polymer with HCl gas The recommended practice when extrusion is interrupted is to purge the equipment.

To minimize degradation, the temperature profile should be developed upward from the minimum temperatures recommended. This will ensure optimum results with no danger of degradation.

Recommendations for Injection Molding

For the injection molding processing of Halar® ECTFE, conventional reciprocating single screw extruders are employed.

Corrosion-resistant materials are recommended for all surfaces in contact with hot Halar® ECTFE polymer. This requirement pertains to internal cylinder walls and the screw. Some surface-hardened tool steels have been used successfully in limited duration runs. The standard practice of never allowing the hot polymer to remain stagnant in the injection molding equipment should be carefully followed. If molding is interrupted, the polymer should be purged out of the equipment immediately with polypropylene or high-density polyethylene. If purging is not possible, temperatures should be lowered to 200 °C (400 °F) while changes are being made.

Shot Size

When injection molding with Halar® ECTFE polymer, the recommended shot size (including sprue and runners) is between 40 and 70 % of machine capacity. If undersized shot weights are used, the polymer tends to degrade because of long residence times in the cylinder. Oversized shots result in uneven heating and/or cold materials.

Injection Molding Conditions

Part design, mold design, cycle time and plasticizing capacity of the press cause the molding condition to vary from part to part. A certain amount of trial and error is therefore necessary to determine optimum molding conditions. It is recommended to start at the lower temperature and pressure levels and gradually increase alternately until optimum is achieved.

Temperature of the Injection Cylinder

Temperatures higher than 287 °C (550 °F) should be avoided. As a general rule, temperatures should not be set higher than necessary to obtain rapid fill at reasonable injection pressures.

Injection Pressure

Pressure exerted on the material can range from 50 bar (700 psi) to 1,380 bar (20,000 psi); thinner sections require higher pressures.

Mold Temperature

Moldings with good surfaces and optimum physical properties normally require mold temperatures between 90 and 150 °C (200 and 300 °F). If only a water heater is available, it should be run as hot as possible. With this type of heater, the surface of the parts will be somewhat less glossy and small cavities may be difficult to fill. Oil or electrical heating is preferred.

Mold Cycles

The time cycle required for a particular mold depends to a very large extent upon the design of the mold and the thickness of the part.

Usually total cycle time is 20–40 seconds for a part less than 3 mm (1/8 inches) thick. The ram forward time is approximately 10 seconds. A thicker part requires longer time with 60–150 seconds being typical for a part of over 6 mm (¼ inches) thickness. In this case, the ram forward time would be increased to 25 seconds.

Mold Release

Halar® ECTFE seldom requires a mold release agent. If it is found necessary to use a release agent, one that has been found to work well is FreKote 44-NC manufactured by Dexter Corporation (Seabrook, New Hampshire).

Table 18: Typical molding conditions

Temperatures	
Rear cylinder	230–245 °C (450–470 °F)
Mid cylinder	245–260 °C (470–500 °F)
Forward cylinder	260–275 °C (500–525 °F)
Nozzle	255–265 °C (490–510 °F)
Mold	100–110 °C (220–230 °F)
Pressure exerted on material	55–140 bar (800–2,000 psi)
Timing	
Total cycle (seconds)	20–150
Ram forward time (seconds)	10–25
Screw speed (rpm)	30–100

Recommendations for Compression Molding

The following procedure can be followed as a guideline for a typical compression molding cycle.

Use a positive pressure mold; it consists of a top plate, a bottom plate and a frame.

- Heat the mold to 260 °C (500 °F).
- Feed the room temperature pellets into the mold.
- Apply a pressure of 15 bar (200 psi) for 5–10 seconds.
- Reduce pressure to 5 bar (40 psi) and maintain pressure; the press will close gradually as the material melts; always keep the melt and plates in contact; complete melting will take approximately 1–10 hours for a 15 mm (5/8 inch) thick plaque. Increase the pressure in steps throughout the melting cycle until 15 bar (200 psi) is reached.
- After 1–10 hours, turn on the cold water.
- Maintain 15 bar (200 psi) until the plaque is at room temperature (about 20 minutes for a 15 mm, 5/8 inch thickness)

Recommendations for Membrane Preparation Processes

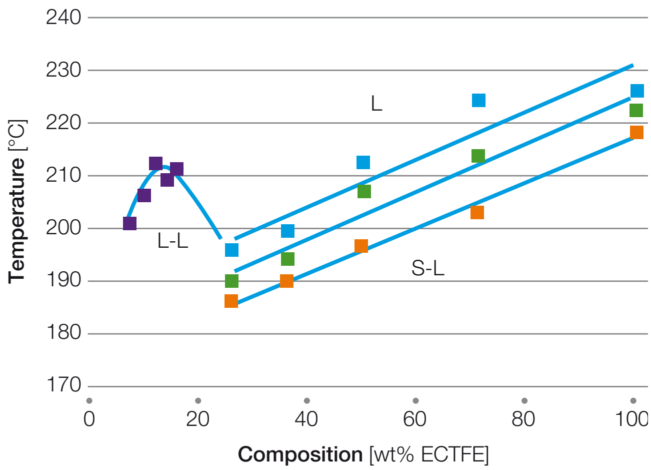
Halar® ECTFE is a suitable material for manufacturing specialty membranes, which can be utilized wherever high resistance against aggressive chemical agents is required.

Halar® ECTFE is a hydrophobic material due to its chemical composition; it is however possible to modify surface properties with post-treatments that are typically performed at industrial level for obtaining desired hydrophilic performances on finished membranes.

Because of its exceptional chemical resistance, this material cannot be processed using solution phase inversion. It must be processed at temperatures close to its melting point (200–240 °C) using a TIPS process for manufacturing either hollow fibers and flat sheet membranes. Typical solvents that can be utilized for processing Halar® ECTFE are acetyl tributyl citrate, glycerol triacetate (GTA), dibuthyl phthalate (DBP), dioctyl phthalate (DOP).

Phase diagrams of Halar® ECTFE are available in the literature. An example of solubility of ECTFE in dibuthyl phthalate (DBP) is shown in Figure 38. When using this solvent it is possible to obtain a liquid-liquid demixing for concentrations below 25 % and solid-liquid demixing with polymer crystallization above 25 % polymer content. It is possible to extend the liquid-liquid demixing region of the phase diagram by changing the polarity of the solvent, for instance by adding a non-solvent in the polymer mixture.

Figure 38: Phase diagram of Halar® ECTFE in dibuthyl phthalate (DBP)



All the information given in these pages can only be considered as examples for processing of Halar® ECTFE. It cannot be considered as specifications or as a guarantee for successful extrusion or molding of Halar® ECTFE.

Foamable Grade
Halar® 558 Fluoropolymer

Halar® 558 is a new polymer grade for foam extrusion applications, including primary insulations, coaxial cable cores and foam jackets for cables. Like the other Halar® ECTFE grades, this polymer offers a broad service temperature range, from cryogenic temperatures to 150 °C; excellent performance in flammability tests (including ASTM 84 Steiner tunnel tests), and excellent chemical resistance to a wide variety of acids, bases and organic solvents. In addition, Halar® 558 polymer has excellent mechanical properties even in thin walls and is resistant to high doses of radiation.

Halar® 558 is a compounded fluoropolymer which contains a nucleating agent, a chemical blowing agent that decomposes when the polymer is extruded, and a processing aid. The void content of foam products extruded from this polymer can be controlled by adjusting processing conditions such as melt temperature, head pressure and quench rates. If it is required to reduce void content through reduction of blowing-agent concentration, Halar® 500 may be blended with Halar® 558. Moreover, for gas-injection extrusion, Halar® ECTFE polymers which contain only nucleating agents and processing aids can be supplied.

In common with all other grades of Halar® ECTFE polymers, this foamable grade can be easily colored with commercially available color concentrates and can be readily printed.

Please contact Solvay Specialty Polymers for further information concerning properties and processing of this grade.

Welding

Halar® ECTFE can be welded using the standard techniques known for common plastics, such as PE or PVC. In particular, hot gas welding is routinely used to thermo-weld Halar® ECTFE liners. Tensile tests performed on the welded seams have proven that fusions are 100 % as reliable as the original material.

The following general recommendations will be applied with hot gas welding Halar® ECTFE liners.

Equipment

Use welding guns with heating power of 800 W or higher.

Proper temperature measurement is crucial to ensure consistent welds. It is good practice to measure the temperature of the gas stream inside the nozzle, at 5–7 mm (¼ inch) from the outlet.

Good quality Halar® ECTFE welds can be obtained when nitrogen or clean and dry air is used. Welding in nitrogen is recommended when the welding facility lacks a clean and dry source of air.

Different welding tips are available. High speed welding tips are used for the primary weld, while tacking tips can be used to hold in place the various sections of the liner.

Health, Safety and Environment

As with all polymers exposed to high temperatures, good safety practice requires the use of adequate ventilation when processing Halar® ECTFE. Excessive heating may produce fumes and gases that are irritating or toxic. Ventilation or proper breathing equipment should be provided to prevent exposure to fumes and gases that may be generated.
Refer to the Halar® ECTFE Safety Data Sheets for detailed recommended procedures for safe handling and use. Contact your regional Solvay Specialty Polymers office for a copy.

Recommendations for Welding

Use round welding rods made of the same Halar® ECTFE grade as the profiles to be welded.

Warning: Welding together profiles made from different grades is not recommended. If it is unavoidable contact your regional Solvay Specialty Polymers Technical Service representative.

Scrape carefully the surfaces to be welded. When using fabric backed sheets, remove the fabric along the welding line (2 or 3 mm on each sheet) to prevent fiber inclusions. Align and hold the two sheets to be welded at a distance no greater than 0.5–1 mm.

It is recommended to cut a V-shaped groove between the two sheets using the appropriate scraper. Avoid the use of makeshift tools as it could result in an irregular weld bead. Thoroughly clean the welding area and the welding rod.

Warning: Use of a cleaning solvent may cause fire hazard due to the heat generated by the gun.

Clean the nozzle of the welding gun with a brass brush, adjust the air flow to 50–60 standard liters/minute (1.8–2.1 cfm) and set the temperature of the welding gun on 380–425 °C (380–400 °C for thin liners).

Note: The temperatures recommended in this document must be intended as measured inside the nozzle. If the welding gun is equipped with a thermometer, check the readings using a thermocouple before beginning the welding operations.

Weld holding the gun at a 45–60° angle and adjust the welding pressure and speed ensuring that the welding rod and the sheets melt simultaneously. Welding speeds in the 0.1–0.5 cm/s (or 1/16"–1/4" per second) range are usually suitable.

If the speed is too low, the welding rod will overheat and start flowing; on the other hand, if the speed is too high, the welding rod will not melt properly and the groove between the two sheets will not be duly filled by the molten material.

Similarly, if the welding pressure is too low, the groove between the two sheets will not be completely filled, while an excessive force may cause dimples along the welding bead which will eventually act as stress concentrators.

Machining

The machining of Halar® ECTFE is very similar to that of nylon. The following procedures provide guidelines for successful machining operations with this versatile fluoropolymer.

Internal stresses may often be created during the machining of Halar® ECTFE. These stresses may lead to the warping of a component. To avoid creating stresses during machining, attention should be given to the following points:

1. Use sharp tools
2. Avoid excessive clamping or cutting forces
3. Prevent overheating by use of coolants

Generally, when the above principles are followed, stress-free parts will be obtained. In those cases where optimal dimensional control is required, annealing is recommended.

Annealing consists of a heat treatment in oils or other liquids at temperatures about 30 °C (50 °F) above the maximum exposure temperature to be encountered. At 150 °C (300 °F) in sections of 12.5 mm (1/2 inches), 15 minutes is adequate. For sections 25 mm (1 inch) in thickness, 4 hours is needed, and an additional 2 hours is added for each additional inch of thickness. Due to the low thermal conductivity of Halar® ECTFE, slow heating and cooling is required for this step.

Halar® ECTFE can easily be machined on most standard metal working machines. For best results, particularly on long production runs, the following should be considered:

1. Due to the previously mentioned low thermal conductivity, the surface temperature of the work will rise rapidly during machining. To prevent this, coolants are recommended.

2. The relatively low melting point of the material, 242 °C (468 °F), combined with the low thermal conductivity may cause softening of the work surface unless the proper machining procedures are followed.

For turning, the general type of tool used for machining soft metals such as aluminum are also suitable for Halar® ECTFE. For optimum results, the angles should be somewhat different. Rake angles of 30 to 40° with a side clearance angle of 5° as well as a 5° end clearance and end cutting edge angles of 8 to 10° are used. The cutting edge of the tool should be the same height as the turning center – a low tool position causes "running" of the work on the tool and a high tool position impairs the cutting action. In order to obtain a smooth surface finish on the work, it is advisable to use a rounded tool for the final cut rather than the one described above which is intended for general purpose turning. In addition to the use of a coolant, the lapping of the tool face will contribute to a smoother finish.

For cut-off, a tool with 5° side reliefs, 10 to 15° end clearance, and 5° side clearances with the top side of the tool level to keep from "biting" into the work is recommended.

When turning Halar® ECTFE, there is a tendency to form a continuous ribbon which may wind around the work. This can be overcome by using the proper rake angle and adjusting the cutting speed. Burring can be avoided or minimized by using sharp, well designed tools, proper cutting speeds, and a good coolant. In order to prevent deformation of thin-walled parts, it may be desirable to clamp the work in a collet rather than at three or four points.

For milling, standard cutters (gear, wheel, face and side, cylindrical, key-way, and finger) can be used with Halar® ECTFE as with steel, provided that they are sharp. The angles of these cutters do not need to be changed although the angles used on cutters designed for aluminum are the best since their shape is adapted to machining soft, tough materials.

Basically, the same RPM, feed, and cutting depth would be used in milling as in turning. A good coolant is also essential. In order to avoid distortion of the work and "biting" of the milling cutter, careful and uniform clamping is necessary.

To avoid the formation of burrs during milling, it may be advisable to back up the work with another plate. A less expensive material such as nylon could be used.

Halar® ECTFE can be readily sawed. When using a power hacksaw, there are no special procedures different from steel. There are no limits for the thickness of the material. It is desirable to use a coarse saw blade with about 4 to 6 teeth per inch, and there should be some set to these.

A vertical band saw may also be used but with a little more care. The speed of the band should not be too high (for example, 450 m/min (1,500 ft/min) for a thickness of 75 mm (3 inches)). Again, a coarse tooth (4 to 6 per inch) such a skip tooth or buttress type should be used. No coolant is used normally in this method, and the material should not be pressed too hard against the blade.

When using circular saws, regular, hollow ground metal working blades are acceptable for thin sections up to about 8.5 mm (1/3 inches). For heavier sections, special skip tooth or buttress type blades are required.

To drill Halar® ECTFE, standard drills are generally suitable. Sharp bits and a cooling fluid are advisable. Regular up and down movement of the drill helps in cooling and in clearing the hole. The feed should be reduced as the depth of the hole increases.

Due to the elasticity of Halar® ECTFE and because of the temperature rising during drilling, it may be necessary to use a drill diameter 0.1 to 0.5 mm (0.004 to 0.020 inches) greater than the size of the derived hole. When several holes have to be drilled close to one another, it may be necessary to plug holes already drilled to prevent deformation. These procedures are best established by experience.

Reaming is difficult because of the elasticity of the material. The best results are obtained by using a sharp, spiral fluted reamer. Some machinists fill the hole to be reamed with a wax or tallow prior to reaming.

Screw threading and tapping is easy with Halar® ECTFE. It is advisable to use a cutting oil to avoid excessive heat and ensure the best finish. The use of the first tap can be omitted and for very small holes only the third tap needs to be used.

Halar® ECTFE sheet can be punched easily. The tools must be carefully ground and lapped if possible. The piece to be punched should be tightly clamped.

Halar® ECTFE rods and tubes can be centerless ground on conventional equipment. It is recommended that the work center be approximately 0.100 inches below the center line of the wheels and that water-soluble oil be used as a coolant.

Note: All the information given in these pages can only be considered as examples for processing of Halar® ECTFE. Please contact Solvay Specialty Polymers for detailed information.

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