

FLUOROPOLYMERS INDUSTRIAL



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Melt Processible Fluoropolymers

A Family of Resins With Unique Properties

Du Pont produces a family of fluoropolymer resins to meet a wide range of demanding end-use requirements. They have gained widespread recognition for their unique properties and design versatility which are helping to solve some of industry's toughest materials problems. Common to all Du Pont fluoropolymers are:

- Outstanding chemical resistance
- High-temperature resistance
- Anti-stick characteristics
- Excellent dielectric properties
- Outstanding resistance to weather and aging

A summary of the physical properties of TEFLON® fluorocarbon resin and TEFZEL® fluoropolymer resin is shown in Table 1 on page 4.

The Melt Processible Resins

The melt processible fluoropolymer resins extend the product line by providing the desirable properties of TEFLON® PTFE fluorocarbon resins in products that can be processed by conventional thermoplastic techniques, such as injection molding and extrusion.

Applications encompass those where designers and end users require a thermoplastic with excellent chemical stability, dielectric properties, anti-stick characteristics and mechanical strength for use in extreme high- and low-temperature environments.

This versatile family of melt processible fluoropolymer resins is available from Du Pont to meet specific end-use requirements and processing needs:

 TEFLON® FEP fluorocarbon resin— TEFLON® FEP is rated for service to 400°F/204°C and retains the chemical resistance and dielectric strength of TEFLON® PTFE fluorocarbon resin.

- TEFLON® PFA fluorocarbon resin— TEFLON® PFA is a premium performance resin with good melt processing characteristics and unique thermal stability. It offers high-temperature strength and stiffness; excellent stress-crack resistance; high flex life and excellent electrical properties. Its high temperature service rating is 500°F/260°C and it resists virtually all chemicals.
- TEFZEL® fluoropolymer—TEFZEL®
 is a strong, tough material with
 chemical resistance, electrical
 properties and aging resistance
 approaching those of TEFLON®
 fluorocarbon resins. Rated for
 use to 302°F/150°C, TEFZEL® has
 excellent processing properties
 using conventional thermoplastic
 techniques.

Fluoropolymer resins are different from most other thermoplastics since they have higher melting points and higher melt viscosities. Accordingly, TEFLON® and TEFZEL® require relatively high processing temperatures and slow injection rates. Special consideration for mold design is needed because of the molding characteristics of these resins, also, process equipment needs to be constructed of corrosion-resistant materials.

Thermal Stability

The flow rate, defined as the number of grams of molten polymer that flows through the orifice of a rheometer in ten minutes, is inversely proportional to the viscosity of the molten polymer. Use of a heated, insulated, and thermostatically controlled laboratory melt indexer (comprising a cylinder and a weighted piston

capable of forcing molten resin through an orifice at the bottom of the cylinder) permits accurate measurement of flow rates for TEFLON® and TEFZEL® resins; (See Table 2 and Figure 1).

Since thermal degradation of polymeric materials is a time-temperature dependent phenomenon, careful measurement of the increase in the flow rate of a polymer during processing provides an excellent technique for monitoring thermal degradation of injection molded parts during the manufacturing process.

The effect of temperature and hold-up time upon the flow rate of TEFLON® and TEFZEL® resins is shown in Figures 2, 3 and 4 on page 6. With TEFLON® FEP, data show little significant change in properties if the change in flow rate, due to thermal degradation. is less than 10%. With TEFLON® PFA, a change of up to 20% may be tolerated without a significant effect on end-use properties. For TEFZEL* fluoropolymers, the flow rate may increase by as much as 50% without significant change in tensile or elongation properties of the resin. Such changes can be measured before any serious losses in important end-product properties occur. This provides sufficient time for corrective action to be taken by a technician so that the proper process variables may be altered to prevent serious damage to production.

Although discoloration or small bubble formation normally indicates resin degradation, dark color can occur in the resin melt from equipment corrosion products or from trace amounts of less thermally stable resins. Continued operation at conditions causing thermal degradation of the resin can result in black specks or streaks of contamina-

Table 1. Properties of Fluoropolymers for Injection Molding

	ASTM		TEFLON* FEP	TEFLON* PFA			LLL	
Property	Standards	Units	100	340	210	200	280	HT-2004
Specific Gravity	D792		2.15	2.15	1.70	1.70	1.70	1.86
Melting Point	DTA-E168	°F °C	500 260	582 305	512 267	512 267	512 267	_
Tensile Strength, 73°F 23°C	D638	psi MPa	3,400 23	3,600 25	5,800 40	6,500 45	6,700 47	12,000 82.7
Elongation, 73°F/23°C	D638	%	325	300	300	300	300	8
Flex Modulus, 73°F 23°C	D790	psi MPa	90,000 586	90,000 586	170,000 1,200	170,000 1,200	170,000 1,200	950,000 6,550
Hardness Durometer	D2240		D56	D60	D67	D63	D72	_
Dielectric Constant, 1MHz	D150		2.02	2.1	2.6	2.6	2.6	3.38
Dielectric Strength, short time, 10 mil 0.25 mm	D149	V/mil kV/mm	2,000 80	>2.000 80	1,800 70	1,800 70	1,800 70	425 16.7
Dissipation Factor, 1MHz	D150		0.0007	0.0001	0.0054	0.0031	0.0072	0.0035
Volume Resistivity	D257	ohm-cm	1018	1018	1017	1017	1017	_
Water Absorption, 24 hr	D570	%	0.004	< 0.03	0.007	0.007	0.007	0.025
Weather Resistance	Florida Exposure	Years Unaffected	20	10	-	5	-	-
Limiting Oxygen Index	D2863	%	93	95	30	30	30	-
Flame Rating*	UL94		vo	VO	VO	vo	vo	VO
mpact Strength, 73°F 23°C	D256	ff-llb/in J/m	no break	no break	no break	no break	no break	no break
-65°F -54°C		ff-llb/in J/m	2.9 154.8	1.2***	2.5 133.5	3.5 186.8	2.0 106.8	_
Chemical Resistance**				- Excellen	t in Chemic	cal Service		

TEFZEL*

tion. Since the injection molding temperatures required for TEFLON® and TEFZEL* will decompose many other thermoplastics, it is absolutely necessary that the molding equipment be thoroughly cleaned of such materials before molding TEFLON® fluorocarbon and TEFZEL® fluoropolymer resins. Should contamination occur, it is imperative that molding cease until the cause for the contamination is discovered. The equipment should then either be thoroughly purged or disassembled and scrupulously cleaned before restarting the molding process.

Resin Flow Properties

Melt fracture in molten polymer occurs when the velocity of the flowing resin exceeds the critical velocity (the point where the internal flow stresses in the molten

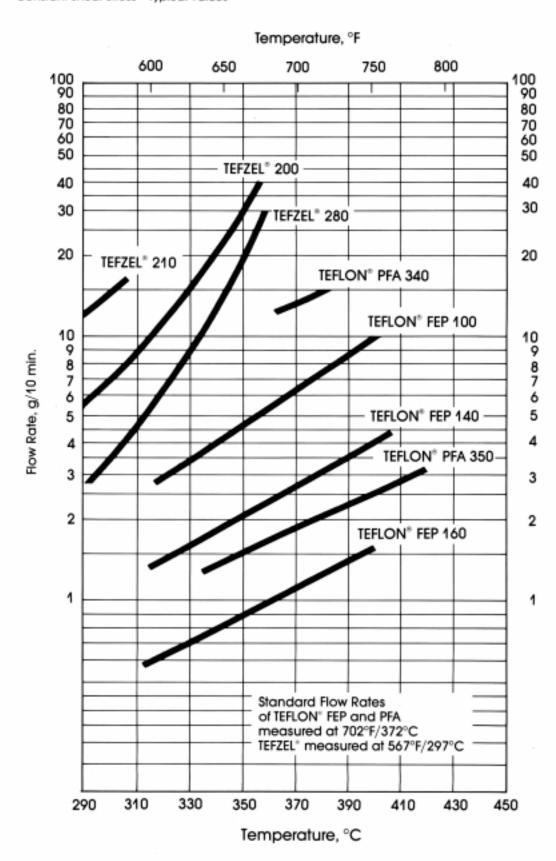
Table 2. Typical Flow Rates (FR) for Fluoropolymers

Resin	Grade	Average FR, grams per 10 minutes
TEFLON® FEP	100	7
Measured at 702°F/372°C	140	3
ASTM Method D2116	160	1.2
TEFLON® PFA Measured at 702°F/372°C ASTM Method D3307	340 350	14 2
TEFZEL®	210	23
Measured at 567°F/297°C	200	7
ASTM Method D3159	280	4

[&]quot;This numerical flame-spread rating is not intended to reflect hazards presented by this or any other material under actual fire conditions.

[&]quot;Refer to "Chemical Use Temperature Guide—TEFZEL" Fluoropolymer" and "TEFLON" Fluorocarbon Resins—in Chemical Service."
"At -320"F (-196"C).

Figure 1
Flow Rates of TEFLON® FEP and PFA and TEFZEL® vs. Temperature at Constant Shear Stress—Typical Values



resin exceed the melt strength of the resin). Although melt fracture can occur with any thermoplastic material, the critical velocity at which it occurs with TEFLON* and TEFZEL* is much lower than most thermoplastic materials. A part molded under melt fracture conditions may have a cloudy or frosty surface, or, in the case of TEFLON* FEP and PFA, it may be internally fractured or delaminated with a normally appearing smooth and shiny surface.

There are three possible techniques for eliminating melt fracture:

- Reduce the resin velocity by enlarging the runners, gates, or cavities, and/or using a slower ram speed.
- Increase the critical flow rate by increasing the melt temperature or the mold temperature.
- Reduce heat losses by shortening the distance through which the resin must flow to fill the cavity (e.g. multiple gates).

The three preventive possibilities are limited as remedies, however, since too slow an injection rate may cause the resin to solidify before the mold cavity is filled. Also, there are limits as to how much the mold area may be enlarged without adversely affecting the design. Maximum mold temperature is limited since it directly affects product parameters like ejectability, surface finish, warpage, shrinkage, and cracking, while maximum melt temperature is limited by resin degradation.

Figure 2 Percent Increase in Flow Rate of TEFLON[®] FEP 100 with Thermal Exposure

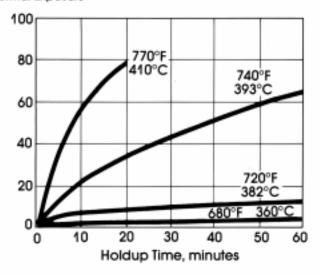


Figure 3 Percent Increase in Flow Rate of TEFLON® PFA 340 with Thermal Exposure

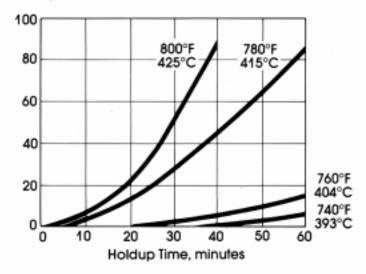
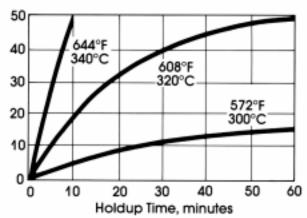


Figure 4
Percent Increase in Flow Rate of TEFZEL® 200 with Thermal Exposure



Equipment for Injection Molding

Although it is possible to injection mold TEFLON® and TEFZEL® in ram type equipment, the reciprocating screw machine is recommended because the screw produces a thoroughly plasticated, uniform melt and provides a much more efficient transmission of pressure to the molten resin flowing into the mold.

Additional inherent advantages for the reciprocating screw machine over the ram type unit are:

- Fewer sites for possible resin stagnation.
- Better dispersion of fillers or pigments in the melt.
- 3. Less resin hold-up time.
- Higher possible melt temperatures, with less thermal degradation.

Materials of Construction

Since molten TEFLON® fluorocarbon and TEFZEL® fluoropolymer

resins are corrosive to most metals. it is most important that corrosionresistant metals be used for all parts in continuous contact with the molten resin. The only exception to this rule is for short, prototype runs. Traces of corrosion products that accumulate on the metal surfaces can break away, contaminating the finished product, and possibly adversely affecting physical properties. It is suggested that "Hastelloy" C1, "Hastelloy" C-2761, "Duranickel"2, or "Monel" be used for the screw, adapter, and nozzle. For the cylinder lining the use of "Xaloy" 3093, "Brux"4, "Relloy"5 or "Bernex"6 is suggested.

Since high operating temperatures are the rule, it is recommended that a high temperature resistant thread lubricant such as "Never Seez" be used to facilitate ease of machine part disassembly.

Because the mold is maintained at temperatures below the melting point of the resin, the corrosion rate of the mold surfaces will be less than for other parts of the machine. Except for long production runs, unplated molds of hardened tool steel, hardened stainless steel, or high quality chrome or nickel plated material may be satisfactory. For long runs, more corrosion-resistant materials of construction might be desirable.

Screw Design

Figure 5 is a schematic diagram depicting a recommended screw design for molding TEFLON® and TEFZEL® resins. It is a metering type screw with a metering section which occupies 25% of the total length. The screw should have a constant pitch and a flight depth ratio from the feed section to the metering section of 3:1. For TEFZEL®

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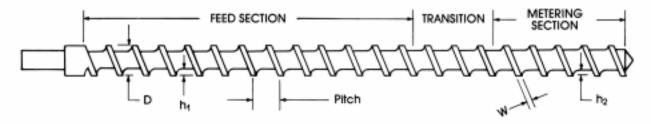
*International Nickel Company, P.O. Box 1958, Huntington, WV 25720.

³Xaloy, Inc., 3 Terminal Road, New Brunswick, N.I. 08903

*Brookes (Oldbury) Ltd. 1, Oldbury, Warley, Worcestershire, 869 2DL, England.

Reitoy Metal GmbH (Reitenhäuser) Germany, 521 Traisdorf Bezirk Käln, Past Bax 1345. Berna AG, Otten, CH-4600 Otten.

Figure 5 Schematic of Screw Design for Injection Molding TEFLON * and TEFZEL*



Suggested dimensions of screw

Diam	eter (D)	Pito	h (P)		offeed in (h ₁)		metering on (h ₂)		tth of d (W)
in.	(mm)	in.	(mm)	in.	(mm)	in.	(mm)	in.	(mm)
1½ 1¾ 2 2½	(38.1) (44.5) (50.8) (63.5)	11/2 13/4 2 21/2	(38.1) (44.5) (50.8) (63.5)	0.255 0.291 0.330 0.420	(6.5) (7.4) (8.4) (10.7)	0.085 0.097 0.110 0.140	(2.159) (2.464) (2.794) (3.556)	0.150 0.175 0.200 0.250	(3.810) (4.445) (5.080) (6.350)

it is recommended that a 3-turn transition zone be used, while for TEFLON® a ½-turn transition section is recommended. Although other screw designs have been used successfully, the two designs described are recommended.

Nozzle

Figure 6 shows a conventional type, reverse tapered nozzle recommended for TEFLON® fluorocarbon and TEFZEL® fluoropolymer resins. The bore should be as large as possible and tapered to prevent dead spots or rapid changes in resin velocity. The sprue should extend into the nozzle ½ to 1 in./ 13 to 25 mm to prevent formation of a cold slug. An included angle of 4° is suggested to permit the material in the tapered portion of the nozzle to be withdrawn with the shot. To decrease the possibility of peening the nozzle bore, which in turn could prevent removal of chilled material from the nozzle, it is recommended that the radius of the nozzle orifice exit be 10 mils/0.25 mm as indicated in Figure 6. To provide a smooth, uninterrupted flow path, the nozzle bore must match the adapter and be equipped with its own separate heater and temperature control.

Non-Return Valve

The non-return or check ring valve, shown in Figure 7, prevents the molten resin from flowing backward along the screw flights during the injection process. The flow path must be streamlined and the joint between the valve and the screw must be smooth and tight in order to avoid areas of stagnant resin flow or holdup. The tip of the screw should be pointed to provide a streamlined flow path for the resin and to reduce the free volume in front of the screw after injection. A leaking valve will cause poor control of part packing and tolerances.

Figure 6 Conventional Reverse Tapered Nozzle

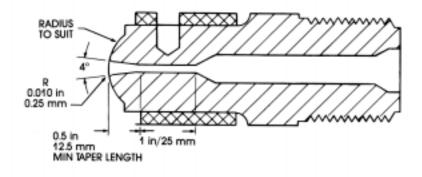
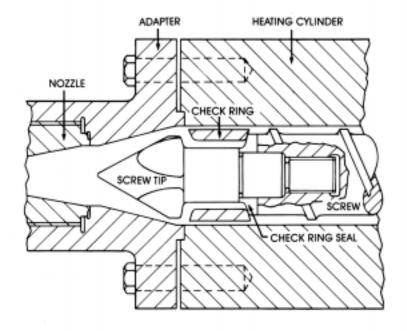


Figure 7
Design of Adapter and Non-return Valve



Smear Head

A smear head, Figure 8, which can be used in place of a nonreturn valve, is a device that uses a small diametral clearance with the cylinder over an extended land length, thus restricting backward melt flow during the injection stroke of the screw. When the screw is rotating during retraction, the melt is forced forward through a narrow annulus; this shearing or smearing action increases melt temperature, improves mixing, and reduces effective packing pressure. The smear head may be preferred over the non-return valve for the following reasons:

- Less tendency for resin stagnation
- Lower possibility of overpacking the mold (with attendant delamination for TEFLON*)
- Less tendency to form streaks in the molded part
- Less abrasion on relatively soft corrosion-resistant alloys

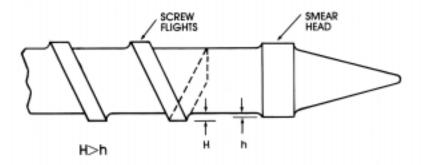
It is suggested that a check ring non-return valve be used when injection molding TEFZEL* 210, due to the resin's lower viscosity. When molding TEFLON* PFA and FEP or TEFZEL* 200 and 280, a smear head would normally be used in place of the non-return valve.

Check rings may be constructed of Hastelloy C or Monel 400. Since no indestructible material of construction for check rings is known, wearing of the check ring should be monitored.

Temperature Control

It is recommended that three independently controlled heater zones be used for the cylinder and one for the adapter. A separate controller should be used on the nozzle. The heater controllers must be capable of accurate temperature control up to 700°F/371°C for TEFZEL® and up to 800°F/427°C for TEFLON® FEP and PFA. This level of control requires a heater watt density of 30 to 40W/in²/4.6 to 6.2 W/cm².

Figure 8 Smear Head



Hydraulic System

When injection molding TEFLON® fluorocarbon and TEFZEL® fluoropolymer resins, it is often necessary to use an extremely slow injection rate in order to avoid either surface or internal melt fracture. The hydraulic system, therefore, should be capable of producing a very uniform and controlled ram speed as slow as 60 seconds per shot. A higher injection rate is permissible with TEFZEL® 210 since it does not melt fracture as readily as other fluoropolymers. Thus, the hydraulic system must be capable of producing high rates of ram speed as well as very low rates.

Streamlining

It is most important for the entire flow path of the resin through the machine to be streamlined and that there be no areas of stagnation. Localized holdup, such as may exist in the non-return valve of a reciprocating screw machine, can lead to thermal degradation of the resin and unacceptable production.

Sizing Injection Machines

In conjunction with the weight of the part and the runner, these melt densities should be considered for adequate injection machine size at normal processing conditions:

- —for TEFLON® FEP and PFA ~0.054 lbs/in³ (~1492 Kg/m³)
- -for TEFZEL* ~0.047 lbs./in³ (~1298 Kg/m³)

Clamp tonnage should be appropriate to cavity mold pressure and the area of the mold cavity which will oppose the clamp tonnage. One expects that a clamp pressure of 5 tons/in² of projected area should be adequate for molding parts from Du Pont fluoropolymers.

Mold Design

Materials of Construction

Mold cavities can be constructed from corrosion-resistant materials such as Hastelloy C, Monel, or Duranickel, but these materials provide a degree of corrosion resistance far greater than is usually necessary. Should unprotected tool steel or hardened stainless steel be used, the mold should be thoroughly cleaned before storage with a moderately alkaline material (e.g., ammonia water), dried, and coated with a rust preventative to avoid rusting and pitting. This procedure is particularly important where high humidity conditions prevail. Rusting and pitting may be avoided by plating the mold with either nickel or chrome (chrome should not be used with TEFLON® PFA) to a thickness of 1/2 to 1 mil/0.013 to 0.025 mm; to avoid stripping the plating from the mold, use a high quality plate devoid of pinholes.

Sprue Bushing

The diameter of the sprue bushing should be at least 1/15 in./1.6 mm greater in diameter than the main runner and just slightly greater than the nozzle orifice. Generally, a standard taper of ½ or ¾ in./ft/4 or 6 mm/m is used.

Runners

In order to minimize both heat and pressure losses, large diameter, full round runners of the shortest possible length should be used. A second preference would be trapezoidal runners which are usually easier to machine than round runners. Runner walls should be free of any restrictions and should blend smoothly into the gates.

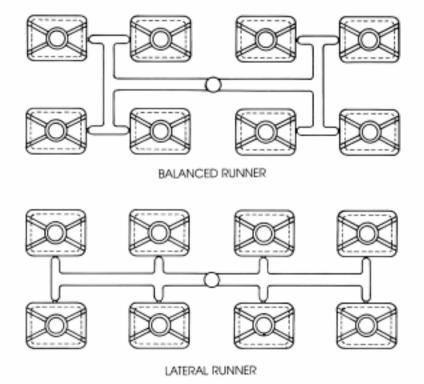
Generally, the thicker the molded part, the larger and shorter the runner should be. Parts of average thickness, up to approximately 0.5 in./12.7 mm, require a runner diameter of 0.25 in./6.4 mm or larger. Thicker parts require the runner diameter to be ½x to 1x the thickness of the part. The runner length or layout dictates the

amount of scrap produced and the pressure drop. Both "balanced" and "lateral" runner systems are shown in Figure 9. A runner system is "balanced" when the resin flow distances between cavities and sprue are equal. When the number of cavities results in a complex or lengthy resin flow the "balanced" runner system is not recommended. A "lateral" runner system can be used with both short and long resin flow distances in most instances.

Gates

Gates should either be as large as possible or eliminated altogether. The land, or length, of the gate should be kept very short. Rectangular tab or fan type gates, generously flared into the mold cavity, are preferred over round gates, since they provide a more

Figure 9 Runner Systems Used in Injection Molding



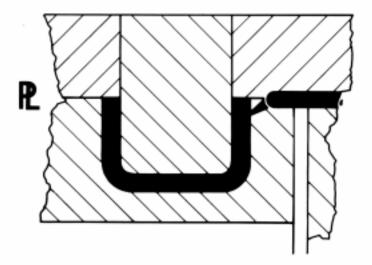
effective means of reducing stress in the resin. Round gates generally are easier to remove from a part but do not permit the same degree of independent control of cavity fill and gate freeze-time as rectangular gates do. The thickness (diameter) of the gate should be ½ to 1x the thickness of the part. Transitions from the runner to the gate to the part should be smooth, with no abrupt changes in the direction of resin flow.

Diaphragm or ring gates can be used for molding cylindrical parts where concentricity is critical or where weld lines cannot be tolerated. Pinpoint gates should be avoided except when molding small parts which are injected very rapidly as with TEFZEL® fluoropolymer resin. Tunnel gating, as shown in Figure 10, can also be used for TEFZEL®.

Gate locations should be at the following points:

- Where the part will not be highly stressed by bending motion or by impact while in use.
- So that weld lines occur in noncritical areas.
- Wherever finishing the gate site would be unnecessary or inexpensive.
- At or near the thickest section in order to minimize sink marks and to avoid pushing resin through a thin section to fill a thicker one.
- At locations consistent with venting requirements (vents are normally required at weld lines or at the bottom of blind cavities).
- In the center of a circular part.

Figure 10 Tunnel Gate



Other Considerations

When the necessary functional and appearance requirements of the part have been established, the final part design should be made with the following considerations in mind:

- Generous filleting.
- Streamlined angles and intersections.
- Uniform wall thickness (if different wall thicknesses are required, blend as gradually as possible).
- Simplicity (the overall design should be kept as simple as possible).

In addition, the following are good practices for consideration:

- Post-molding operations such as drilling holes in the part are usually preferred to the incorporation of pins.
- The number of cavities should decrease as the complexity of the part increases.

 Jetting, the rapid flow of a thin resin stream across a mold cavity, should be avoided.

Mold Heating

Although a mold can normally be heated by use of a high temperature circulating oil heater, when an injection molding process requires a mold temperature in excess of 375°F/191°C, electrical heating should be used. Both halves of the mold should be insulated from the platens to reduce heat losses. Sheets of "Transite" board 0.25 in./6.4 mm thick are satisfactory for this purpose.

Dimensional Considerations

Tolerances

The achievement of close tolerance molding is contingent upon precise control of operating parameters, such as resin feed rate to the cylinder, cylinder and melt temperature, ram or screw speed, pressure, and the overall cycle, all of which must be kept constant. Mold design is also a critical factor in meeting specified tolerances.

In any manufacturing process, as tolerance requirements tighten, the process becomes more complex and expensive. Generally, plastic parts are capable of functioning with wider tolerances than metal counterparts because of the higher inherent resiliency of plastic. Figure 11 shows some suggested tolerances for plastic parts.

A few general comments and cautions relating to tolerances are:

- Tolerances should never be specified closer than necessary.
- Cost increases when close tolerances are specified on several dimensions of a part.
- Do not specify close tolerances for parts with major variations in wall thickness.
- It is not good practice to specify fine tolerances across a parting line or for dimensions controlled by movable cores or sliding cams.

Table 3. Estimated Mold Shrinkage

Thickness of

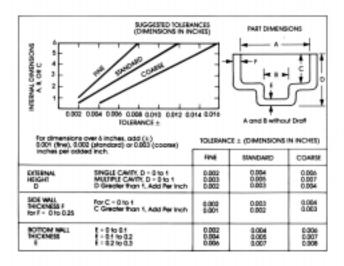
Molde	d Article	Shrinkag	е
in.	(mm)	mils/in. (mm/m)	%
1/8	(3.2)	35-40	3.5-4.0
1/4	(6.4) (12.7)	40-45 45-50	4.0-4.5 4.5-5.0
3/4	(19.1)	50-60	5.0-6.0

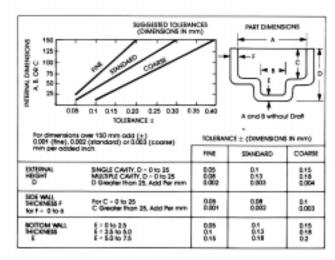
Shrinkage

Listed below are the basic factors affecting the shrinkage of parts injection molded from fluoropolymers:

- Increasing either part thickness or mold temperature increases the part shrinkage, since changes of this nature result in a slower cooling rate for the part, which in turn produces a higher level of crystallinity (order) along with some relaxation in internal stresses.
- Most plastic parts exhibit directional shrinkage differences: part shrinkage is lowest in the direction of resin flow due to the relatively high degree of molecular orientation in that direction. Generally, the straighter the path, the lower the shrinkage, which leads to the conclusion that it is advisable to design the part and locate the gates so as to create the straightest flow path in that direction having the greatest restriction upon dimensional tolerance.
- An increase in injection pressure causes a decrease in shrinkage.
- Generally, parts molded at higher stock temperatures will exhibit higher mold shrinkage.
- The addition of filler material reduces part shrinkage.
- Table 3 shows estimated mold shrinkage versus thickness for TEFLON® FEP and PFA.
- Figure 12 shows estimated mold shrinkage versus thickness for TEFZEL®.
- Figure 13 shows estimated mold shrinkage versus mold temperature for TEFZEL*.

Figure 11 Suggested Tolerances



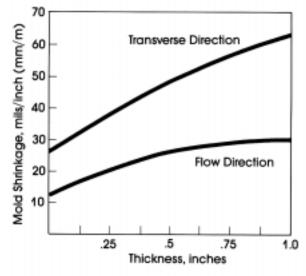


Shrinkage Allowance

If accurate part dimensions are required, shrinkage allowance should be determined by test molding the part in question. Listed below are the steps recommended for construction of a mold:

 Machine a single cavity in the production mold base using the specified dimensions for the

Figure 12 Mold Shrinkage vs. Thickness for TEFZEL® 200



Mold Temperature = 200°F/93°C Melt Temperature = 625°F/330°C Injection Pressure = 10,000 psi/70 MPa

- cavity and oversized dimensions for mold parts which form the inside dimensions.
- Machine the sprue bushing and all the runners up to the points where future production cavities will be inserted.
- Perform several trial moldings with the single cavity mold and determine the best molding conditions for production of satisfactory parts.
- Check the dimensions of the molded article after it has been maintained at its expected operational temperature for 24 hours.
- Use the shrinkage data obtained from step 4 to design the proper dimensions for the production cavities.
- Machine the production cavities to the correct dimensions and insert them into the production mold base used for the single cavity test mold.
- Without altering the runners or gates, mold a part under the previously established conditions. Any compensation required for slight differences in dimensions can be accomplished by slight variations in the molding conditions.

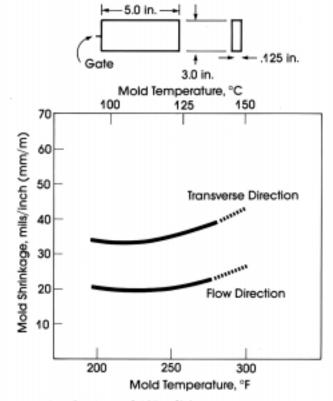
Figure 13 Mold Shrinkage vs. Mold Temperature for TEFZEL * 200

Heat Treating

Heat treatment or annealing, is a process which minimizes the shrinkage experienced by a part during its operational life. Annealing consists of exposing a molded part to a temperature slightly higher (approximately 10-15°F/ 5-8°C) than its intended operating temperature for approximately 15 minutes per 0.125 in./3.175 mm of part thickness and then cooling very slowly; the time is measured from the point at which the part reaches the desired temperature. The slow cooling allows relaxation of internal stress, thus minimizing any dimensional change during the service life of the part.

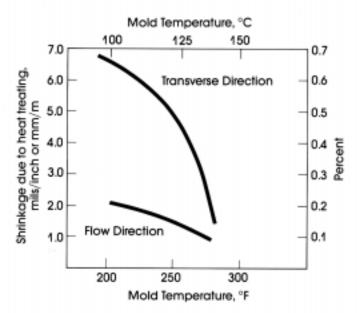
The actual amount of additional shrinkage induced by annealing is a function of mold temperature, i.e. the hotter the mold, the less shrinkage is seen upon annealing the part. This suggests that if the mold temperature were to be set at a point higher than the service temperature of the part, no additional annealing would be required.

EXAMPLE: Figure 14 shows the relationship between shrinkage induced by heat treatment and mold temperature; in this example, the plaque shown in Figure 13 had been heat aged for 72 hours at 300°F/149°C. The data show that additional shrinkage induced by heat treatment approaches the order of 0.10% as the mold temperature approaches 275°F/135°C.



Part Thickness 0.125 in/3.2 mm Melt Temperature 625-650°F/330-345°C Injection Pressure 7,000-10,000 psi/50-70 MPa

Figure 14
Shrinkage after Heat Treatment vs. Mold Temperature for Plaque of Figure 13 (TEFZEL * 200)



Molding Operation

Shutdown and Startup Procedure

If molding equipment is turned off without following proper shutdown procedures, resin degradation may occur and severe corrosion of the equipment could also result if the equipment is constructed of non-corrosion resistant materials. When overnight shutdown without cleanup is desired, the following shutdown procedure is recommended:

- Reduce all temperature controllers to the following levels:
 - a) 600°F/316°C for TEFLON° PFA or FEP fluorocarbon resins
 - b) 550°F/284°C for TEFZEL° fluoropolymer resin
- When all temperatures have been dropped to the levels indicated in Step 1, purge the machine to a dry condition, leave the injection screw in the forward position, and finally shut off the power supply.

The restart procedure is as follows:

- Starting with the temperature controllers for the nozzle, then for the adapter, then the rearbarrel, followed by the front barrel and finally the middle barrel, sequentially raise all the temperature controllers at each zone to the following levels:
 A 2005 (314%) for TEL ONE DEA
 - a) 600°F/316°C for TEFLON® PFA or FEP
- b) 550°F/284°C for TEFZEL° A heat soak of 1 hour may be necessary to melt all resin and heat all metal components to these set temperatures.
- Start the machine slowly after all temperatures have stabilized, setting the temperature controllers to operating levels.
- Commence production when operating temperatures have been reached.

Cleanout Procedure

The following steps outline a suggested cleanout procedure:

- While maintaining operating temperatures, start rotating the screw and continue to rotate it until the resin ceases to flow from the nozzle.
- Reduce cylinder temperatures to the following levels:
 - a) 600°F/316°C for TEFLON® PFA or FEP
 - b) 550°F/284°C for TEFZEL®
- Shut off the screw and remove both the nozzle and the adapter.
 Be sure to clean the nozzle, while it is hot, with a soft metal scraper and copper mesh. Oven burnout is not required and should be avoided.

NOTE: At this point a purge compound of ground cast acrylic or polyethylene may be used when molding either TEFLON® FEP or TEFZEL® resins.

- Slowly remove the hot screw from the cylinder, cleaning it with a wire brush.
- Clean the inside of the cylinder with copper mesh wrapped around a boiler tube brush for a tight fit; then wipe the cylinder clean with a lint-free cloth.

When operating in equipment constructed of corrosion-resistant metal, it is permissible to leave a purge (either ground, cast acrylic or polyethylene sheeting) in the equipment overnight without danger of damage to the metal.

Basic Operating Conditions

Typical basic operating conditions are listed in Table 4. Some additional basic guidelines are listed below:

Melt Temperature (resin leaving the nozzle)

- Decrease the melt temperature as holdup time is increased.
- Runner, gate, and orifice size are additional factors to be considered.

Temperature Profiles

- When operating with high melt temperature and a long holdup time (10 to 15 mins.), the rear zone should be set at a lower temperature than the front zone in order to minimize resin degradation.
- When operating with short holdup times, the temperature of the front and rear zones should be set at the same point.
- The location of the heater thermocouples, the machine size, the speed and the type of the injection screw, shot size, and cycle time are additional factors for consideration.
- Occasionally, high melt temperatures result from mechanical working of the resin melt.
- If the temperature of the rear zone is too high, bridging may occur, resulting in erratic feed.
- If the temperature of the rear zone is too low, high torque loads created by the partially melted resin could cause the screw to stall, thereby reducing the plasticating capacity of the equipment.

Injection Speed

- Allowable ram speed is dictated by the smallest channel through which the molten resin must pass.
- A rough or rippled surface indicates an inappropriate injection speed was used. If surface appearance is rough or frosty, injection speed was too fast; and conversely, if a rippled surface results, the injection speed was too slow.
- Shot size, melt temperature, and mold temperature are additional factors for consideration.

Injection Pressure

- Injection pressure should normally be as low as possible.
- Low injection pressure reduces frozen-in stresses and improves dimensional stability.
- To reduce sink marks or improve weld lines, injection pressure should be increased.
- Equipment and part design must also be considered.

Screw Rotation

- Generally, screw rotation should be as slow as possible.
- High screw speeds, combined with the appropriate back pressure, are used occasionally to produce high melt temperatures necessary for the molding of long, thin parts.

Mold Temperature

- Extremely hot molds should not normally be used for thick-walled sections.
- When the resin flow path is long relative to the part thickness, higher than normal mold temperatures are required.
- Increasing mold temperature reduces the probability of delamination (of TEFLON*).
- When adjusting mold temperature consideration should be given to interrelated parameters, such as part geometry, surface finish, pressure drop, effect upon cycle time, stresses, ejectability of the part, and shrinkage.

Back Pressure

- Back pressure should normally be kept as low as possible.
- Increasing back pressure, however, can sometimes be an effective technique to increase the stock temperature.

Overall Cycle

Overall cycle time is influenced by a number of interrelated manufacturing variables, such as process temperatures and pressures, part geometry, tolerances, warping, and ejectability. Cycle time is usually estimated on the basis of 30 to 40 seconds per 0.125 in./3.2 mm of thickness. Except for thin sections, the longest portion of the cycle is often devoted to the ram-in-motion.

"Packing" the resin, which involves leaving the ram in the forward position while under pressure, should be kept to a minimum. Normally, packing is used only when molding thick sections to reduce sink marks or eliminate voids. Excessive packing usually results in delamination of the part for TEFLON® FEP and PFA, but generally not for TEFZEL®. Use of a smear head reduces the possibility of overpacking.

Record Keeping

Typical formats suggested for recording molding data and for mold inspection and repair information are located in the "Miscellany" section of this guidebook.

TEEZEL®

Table 4. Suggested Molding Conditions for Du Pont Fluoropolymers

Example Temperature	TEFLON® FEP	TEFLON * PFA	fluoropolymer
& Profile	fluorocarbon resin	fluorocarbon resin	resin
Rear Cylinder, °F	600-625	600-630	525-575
°C	315-329	315-332	273-302
Center Cylinder, °F	625-650	625-650	575-625
°C	329-343	329-343	302-330
Front Cylinder, °F	700	700	575-625
°C	371	371	302-330
Nozzie, °F	700	700	650
°C	371	371	343
Mold Temperature, °F	>200	300-500	RT-375
°C	>93	149-260	RT-190
Stock Temperature, °F	650-720	650-750	575-625
°C	343-382	343-399	302-329
Injection Speed	Slow	Slow	Moderately Fast
Injection Pressure, psi	3,000-8,000	3,000-8,000	3,000-15,000
MPa	21-55	21-55	21-103

Auxiliary Operations

Reuse of Resin

TEFLON® FEP fluorocarbon and TEFZEL® fluoropolymer resins can be reworked without significantly sacrificing the properties inherent to the virgin material. TEFLON® PFA has been reworked up to six times without loss of tensile properties or significant increase in melt flow number. However, when reworking resin a number of important precautions should be observed.

Resin for rework must be kept clean since contamination may change its processing characteristics or impair its properties. It is necessary to use corrosion-resistant equipment and to follow the proper equipment cleanup procedures, previously outlined in this guide, to aid in avoiding resin contamination.

Reworked resin should be cut to a size and shape approximately equal to that of virgin resin. A conventional, unchilled, rotary knife cutter equipped with a screen is suitable for obtaining a cut with a minimum of shredding. If the cut obtained upon reworking is too fluffy, it will not feed well enough when blended with virgin resin to permit uniform delivery. Fluffy-cut resin should be extruded as beading and cut into molding pellets.

Pigmentation

Both TEFLON® and TEFZEL® resins may be pigmented with commercially available pigments that are thermally stable at the molding temperatures of the resins; inorganic pigments are the best choice. The simplest method for coloring resin is to blend the unpigmented resin with color concentrates (available from Du Pont) although pigments may also be dry blended by the following procedure:

- Dry the desired pigment overnight at 300°F/149°C in a vacuum oven or a non-circulating air oven to remove absorbed gases and moisture.
- Weigh the pigment and if greater opacity is desired, add and blend the appropriate amounts of titanium dioxide pigment to the color pigment.
- Place the resin pellets in a clean container, such as the original shipping carton, and then screen the pigment through a 100-mesh screen directly onto the pellets.
- Dry blend the color and the pellets by rolling or tumbling the mixture for at least 15 minutes.
- Use the pigmented resin pellets within 30 minutes or store them in an airtight container to prevent the absorption of moisture.

Thin-Section Molding

Generally it is difficult to injection mold very thin sections with most thermoplastic resins, particularly where a relatively large surface area is involved.

With TEFLON®, anything below 0.1 in./2.5 mm may be considered a thin section. When processing thin sections, a faster ram speed must be used since a full shot is of primary importance. There is, however, a problem in obtaining both a full shot and a part free of delamination. The latter product property can generally be obtained only with a slow ram speed, an operating condition that usually produces a resin freeze in the gate or cavity for a thin-walled section before a full shot can be obtained. Therefore, a high mold temperature in the range of 400°F/204°C is necessary to minimize the tendency toward delamination. Packing should not be used, i.e., the ram should be retracted as soon as the mold is full.

Laminations in parts of TEFLON® FEP and PFA may become apparent in a section which appears and feels smooth when it is subjected to either heat aging or repeated flexing. If a part is to maintain a good, smooth surface after being flexed, it must be thick enough to permit a slow ram speed.

Delamination is not a characteristic of TEFZEL® and the precautions needed to avoid it with TEFLON® are not needed when molding TEFZEL®

Glass-Reinforced TEFZEL® Fluoropolymer

Glass-reinforced TEFZEL® has the potential to be produced on a faster cycle than unreinforced resin, since short glass fibers remain well dispersed without straining-out in pinpoint gated molds used in the production of very small parts. Injection molding with reinforced resin differs from the molding process without reinforcement in three ways:

Injection Rate

The injection rate should be as high as possible to produce the smoothest possible surface, since a part molded at too low an injection rate will have a surface with rough, glass-rich areas. Larger gates may be required to achieve the necessary rapid injection rates. and a higher level of mold venting may be needed since a fast fill rate has a tendency to trap air. If the mold does not have adequate venting, trapped air is rapidly compressed to extremely high pressure, producing localized temperatures high enough to scorch portions of the surface area.

Injection Pressure and Packout

Glass fiber reinforced TEFZEL® resin should be injection molded at relatively high pressure in the range of 15,000-20,000 psi/105 to 140 MPa and packed out at high pressure until the mold gate is completely frozen. (These conditions are unlike unreinforced TEFZEL* which is injection molded at the lowest practical injection pressure with little or no packout). In the fabrication of heavy-walled parts, too low a pressure or inadequate packout may result in a molded part with either a void or a pulpy core.

Shrinkage

For glass-reinforced TEFZEL* in the thickness range of 0.125 to 0.25 in./3 to 6 mm, mold shrinkage is 1 to 3 mils/in, or mm/m in the flow direction and approximately 25-35 mils/in. or mm/m in the direction transverse to the flow. Transverse shrinkage, which is strongly influenced by mold cavity pressure, can in some instances, be reduced 40-60% to 15 mils/in, or mm/m by use of large gates, nozzles, sprue bushings, and short runners to reduce the injection pressure drop from the cylinder to the mold cavity. Since the highly directional nature of shrinkage in reinforced materials can produce warpage. close attention should be paid to proper part design and gate location to minimize the shrinkage effect.

Safety

Since their discovery, millions of pounds of TEFLON® fluorocarbon resins have been processed at temperatures in excess of 662°F/350°C, and subsequently placed in end-use applications. many of which have been at or above rated use temperatures. In this period, spanning more than forty years, there have been no reported cases of serious injury, prolonged illness, or death resulting from the handling of these resins. This record includes the experience of Du Pont personnel. hundreds of processors, and thousands of end-users who handle these resins every day.

However, when heated to processing temperatures, TERLON*, as well as other plastics and organic materials, gives off fumes that are objectionable from the standpoint of health and safety.

Also, when grossly overheated, plastics and organic materials, including TEFLON®, undergo some decomposition and actual breakdown in chemical structure.

Fumes from the pyrolysis of many resins and elastomers, as well as those from naturally occurring polymers like rubber, coal, silk, and wood, may be toxic.

Over the years, much effort has been spent at the Du Pont Haskell Laboratory for Toxicology and Industrial Medicine in careful investigation of fluoropolymer resin. In addition, Du Pont research laboratories have studied intensively the thermal behavior of fluoropolymer resins. A number of other laboratories, including those in the United States Department of Health and Human Services, have conducted similar studies related to the safety of these resins.

The knowledge gained through these studies is summarized in these publications, which should be carefully read prior to the handling or processing of any Du Pont fluoropolymer: "TEFLON" Fluorocarbon Resins—Safety in Handling and Use" (E-35824-1) and "TEFZEL" Fluoropolymer Resins—Safety in Handling and Use" (E-64073).

The major safety consideration for injection molding fluoropolymers and other organic polymers is the installation of exhaust hoods to remove off-gases released from hot polymers into work areas. Exhaust hoods over the die and at the hopper heater are recommended. Extruding into water—either a quench tank or a partially filled container—for purging is also recommended.

The safety in handling and use bulletins mentioned above contain data for the design of hoods to capture the gases generated by injection molding of fluoropolymer resins.

Proper procedures and controls must also be maintained to assure that the molding operation will never exceed specified operating temperatures or equipment design pressures. "Blow backs," gas releases from autocatalytic polymer degradation initiated by high temperatures, are possible, although the TEFLON® resins are among the most stable of organic polymers and therefore more resistant to this hazard. TEFZEL® is much more susceptible to autocatalytic degradation than the TEFLON® polymers, suggesting extra care and attention to good operating practices.

Miscellany

Troubleshooting Guide

There are many variables in the injection molding process which affect product quality. The following table is a troubleshooting guide which can be used to help define causes of specific problems.

It should be noted, however, that many injection molding problems are compounded by the interaction of more than one variable. The symptoms of more serious problems tend to mask minor ones. Thus, successful troubleshooting may require careful observation, and the tackling of several problems in sequence.

Technical assistance on extrusion problems is available through your Du Pont representative.

Troubleshooting Guide

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Enlarge nozzle orifice												Х	Х	Х				Х	П
Increase clamp pressure							Х												П
Check screw refraction													Х						П
Increase taper															Х				Н
Check for burs/radius of nazzle															Х				П
Increase nozzie temperature			Х									Х		х	Х				П
Increase size of gate								х				Х		х				Х	П
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Change gate location		Х						х				Х							П
Reduce screw speed					Х	х													П
Check for contamination	х				Х														П
Check for resin stagnation	Х				Х														П
Check materials of construction	Х				Х														П
Check for resin cross-contamination	X				Х			Х											П
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DU PONT POLYMER PRODUCTS DEPARTMENT

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DU PONT POLYMER PRODUCTS DEPARTMENT MOLD INSPECTION AND REPAIR RECORD

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MOLD SIZE: MOLD SIZE: length	DATE RECEIVED.	DATE				
MOLD NAME PART NUMBER(S) CUSTOMER'S PART NUMBER(S)	HIS FOLLOWING MACHINES. DATE R SPECIAL SET-UP EQUIPMENT REQUIRED. Mark (✔) if item is O.K. for next run. Mark (R) if item must be repaired before next run. Give brief explanation.	MOLD CONDITION				

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