

3M™ Dyneon™ Translucent Perfluoroelastomer System PFE 301Z

Features and Benefits

- Phosphorous-free and silica-free formulation reduces the potential for contamination
- Optically transparent
- Excellent plasma resistance with low particle count
- Low extractables, low outgassing, and low particle generation for ultra high purity applications
- Excellent temperature resistance optimizes performance in extreme environments (continuous, in-service temperatures to 300°C with peaks up to 350°C)
- Excellent compression set offers enhanced sealing force retention and seal life
- Smaller, easy to store packages for cleanroom handling
- Proportionate polymer and catalyst simplify the mixing process

Note: Data in this document are not for specification purposes.

Typical Properties

Property	PFE 301TZ	PFE 301C
Specific Gravity	2.1	-
Color	White	Off-White
Form	Crumb	Powder
Fluorine Content	73.0%	-
Mooney Viscosity – ML (1+10) @ 121°C	110	-

Product Form

3M™ Dyneon™ PFE 301Z contains two 1 kg bags of 3M™ Dyneon™ PFE 301TZ polymer and one 25 g bottle of 3M™ Dyneon™ PFE 301C catalyst.

only in well ventilated areas; (2) Do not smoke in areas contaminated with dust from 3M perfluoroelastomers; (3) Avoid eye contact; (4) After handling 3M perfluoroelastomers wash any contacted skin with soap and water. Potential hazards, including evolution of toxic vapors, can occur during compounding or processing under high temperature conditions. When compounding, be sure to read and follow all precautions and directions for use from other compound ingredient suppliers.

Safety/Toxicology

Before processing 3M perfluoroelastomers, read and follow all precautions and directions for use contained in the product label and Material Safety Data Sheet (MSDS). General handling precautions and directions for use include: (1) Store and use all 3M perfluoroelastomers

Typical Properties of Vulcanizate

Compound	phr
PFE 301TZ (Polymer)	100
PFE 301C (Catalyst)	1.1

Typical Rheological Properties [ASTM D5289] Moving Die Rheometer (MDR) 100 cpm, 0.5° Arc, 10 Minutes @ 165°C (330°F)

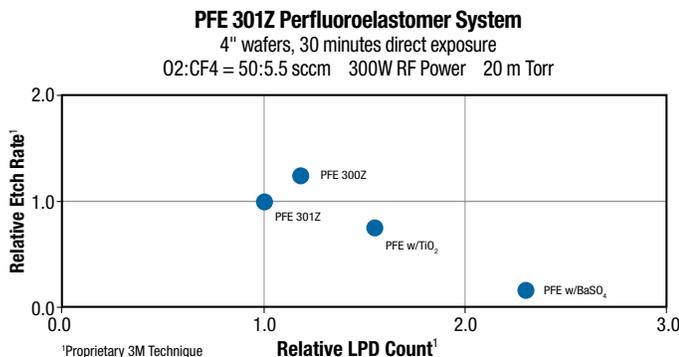
Property	
ML, Minimum Torque, Inch-lb (dN m)	1.9 (2.1)
t _{s2} , Time to 2 Inch-lb Rise from Minimum – Minutes	2.2
t'50, Time to 50% Cure – Minutes	2.2
t'90, Time to 90% Cure – Minutes	5.8
MH, Maximum Torque, Inch-lb (dN m)	5.9 (6.7)

Typical Physical Properties
 Press Cure 10 Minutes @ 165°C (330°F)
 Ramp Post Cure in Air* ASTM D412 Die D Dumbbells

- * Post cure conditions
- 1) Room Temperature to 150°C (302°F) over 1 hour
 - 2) Hold at 150°C (302°F) for 3 hours
 - 3) 150°C (302°F) to 300°C (572°F) over 6 hours
 - 4) Hold at 300°C (572°F) for 4 hours
 - 5) 300°C (572°F) to Room Temperature over 2 hours

Property	
Tensile, psi (MPa)	2750 (18.9)
100% Modulus, psi (MPa)	520 (3.6)
Elongation at Break, %	260
Hardness, Shore A [ASTM D2240]	72
Compression Set Resistance, % [ASTM D395 Method B, -214 O-rings]	
70 Hours @ 250°C (482°F)	26

PFE Plasma Resistance
 Plasma resistance was assessed at 3M using a RIE (Reactive Ion Etch) plasma system coupled with wafer surface scan capability. Materials are placed on 4" wafers and exposed to direct plasma for a period of time. The data generated from this test versus a standard 3M material under the same conditions allows for an estimate of the Relative Etch Rate vs. Relative LPD Count (Light Particle Defect) as shown to the right. The equipment used for this test has a variety of controllable variables such as gas mixtures and flow rates, RF or ICP power levels, and chamber pressure; please contact your 3M Technical Service Representative for additional information on testing capability for your processes.



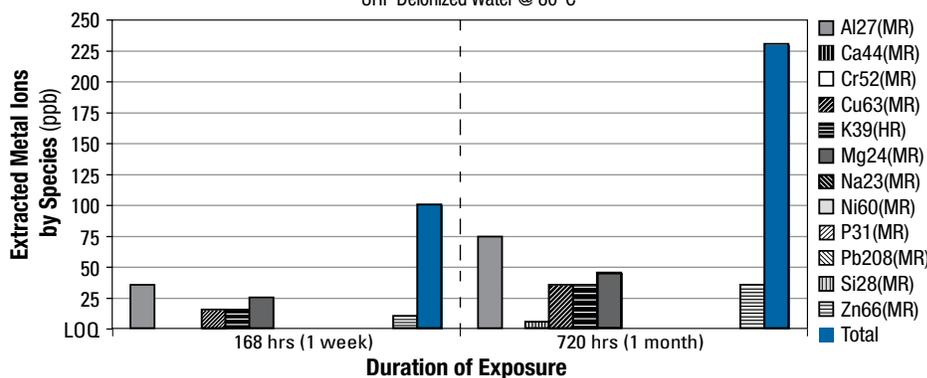
Metal Ion Extraction & Outgassing Study
 Press Cure 6 Minutes @ 165°C (330°F), -214 O-rings
 Ramp Post Cure in Air*, O-rings Cleaned Prior to Testing
 Exposed @ 80°C (176°F) in UHP Deionized Water

Element	Duration: 168 Hours			Duration: 720 Hours		
	Concentration (ppb) ¹	Variance (±ppb)	LOQ (ppb)	Concentration (ppb) ¹	Variance (±ppb)	LOQ (ppb)
Al27(MR)	35	15		75	20	
Ca44(MR)	<LOQ		15	<LOQ		20
Cr52(MR)	<LOQ		10	5	5	
Cu63(MR)	15	5		35	10	
K39(HR)	15	5		35	5	
Mg24(MR)	25	10		45	15	
Na23(MR)	<LOQ		15	<LOQ		25
Ni60(MR)	<LOQ		10	<LOQ		10
P31(MR)	<LOQ		15	<LOQ		15
Pb208(MR)	<LOQ		5	<LOQ		5
Si28(MR)	<LOQ		25	<LOQ		20
Zn66(MR)	10	5		35	15	

¹ Proprietary 3M Technique LOQ = Limit of Quantification

Note: Data in this document are not for specification purposes.

PFE 301Z Clear Perfluoroelastomer System
 Extracted Metal Ion Species vs. Exposure Time
 UHP Deionized Water @ 80°C



Outgassing Rates for PFE 301Z

Temperature	100°C	200°C	300°C	400°C
Outgassing Rate (torr-liters/second/gram)	7.4×10^{-5}	4.5×10^{-5}	1.8×10^{-4}	2.1×10^{-3}

Dry Heat Resistance of Vulcanizate [ASTM D573]

Temperature	250°C (482°F)	250°C (482°F)	250°C (482°F)
Time (Hours)	70	168	336
Tensile Strength at Break, psi (MPa)	3045 (21.0)	3206 (22.1)	3335 (23.0)
Elongation at Break, %	270	265	290
100% Modulus, psi (MPa)	515 (3.6)	535 (3.7)	480(3.3)
Hardness, Shore A [ASTM D2240]	74	74	72
Change in Tensile Strength, %	+11	+17	+21
Change in Elongation at Break, %	+4	+2	+12
Change in 100% Modulus, %	-1	+3	-8
Change in Hardness, pts	+2	+2	0
Change in Weight, %	-0.1	-0.2	-0.5

Temperature	275°C (527°F)	275°C (527°F)	275°C (527°F)
Time (Hours)	70	168	336
Tensile Strength at Break, psi (MPa)	3245 (22.4)	3430 (23.6)	3020(20.8)
Elongation at Break, %	280	280	310
100% Modulus, psi (MPa)	495 (3.4)	460 (3.2)	405 (2.8)
Hardness, Shore A [ASTM D2240]	73	73	71
Change in Tensile Strength, %	+18	+25	+10
Change in Elongation at Break, %	+8	+8	+19
Change in 100% Modulus, %	-5	-12	-22
Change in Hardness, pts	+1	+1	-1
Change in Weight, %	-0.4	-0.9	-1.9

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Temperature	300°C (572°F)	300°C (572°F)	300°C (572°F)
Time (Hours)	70	168	336
Tensile Strength at Break, psi (MPa)	2735 (18.9)	2690 (18.5)	2575 (17.8)
Elongation at Break, %	335	355	380
100% Modulus, psi (MPa)	350 (2.4)	330 (2.3)	325 (2.2)
Hardness, Shore A [ASTM D2240]	70	69	70
Change in Tensile Strength, %	-1	-2	-6
Change in Elongation at Break, %	+29	+37	+46
Change in 100% Modulus, %	-33	-37	-38
Change in Hardness, pts	-2	-3	-2
Change in Weight, %	-2.4	-4.8	-9.0

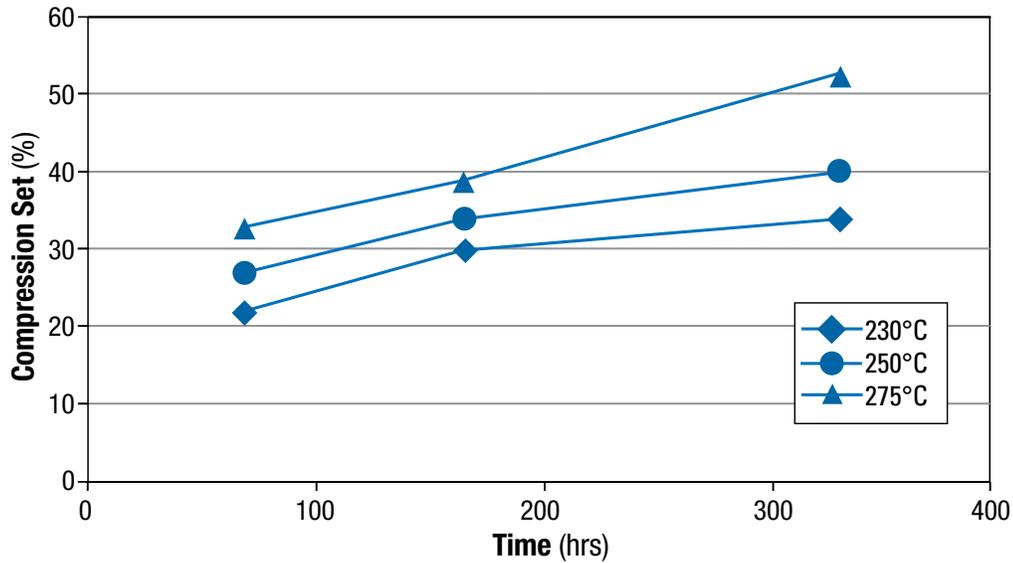
Typical Compression Set Resistance
Press Cure 10 Minutes @ 165°C (330°F) Ramp Post Cure in Air*
Compression Set, % [ASTM D395 Method B, -214 O-rings], 25% Deflection

- * Post cure conditions
 1) Room Temperature to 150°C (302°F) over 1 hour
 2) Hold at 150°C (302°F) for 3 hours
 3) 150°C (302°F) to 300°C (572°F) over 6 hours
 4) Hold at 300°C (572°F) for 4 hours
 5) 300°C (572°F) to Room Temperature over 2 hours

70 hours @ 230°C (450°F)	21
168 hours @ 230°C (450°F)	29
336 hours @ 230°C (450°F)	33
70 hours @ 250°C (482°F)	26
168 hours @ 250°C (482°F)	33
336 hours @ 250°C (482°F)	40
70 hours @ 275°C (527°F)	32
168 hours @ 275°C (527°F)	38
336 hours @ 275°C (527°F)	52

Note: Data in this document are not for specification purposes.

3M™ Dyneon™ PFE 301Z Translucent Perfluoroelastomer System
 Compression Set Resistance vs. Temperature and Time
 ASTM D395 Method B at 25% deflection



Processing Recommendations

3M™ Dyneon™ PFE 301Z is an advanced perfluoroelastomer system that incorporates polymer and catalyst technologies. For best results and to reduce chances for contamination, dedicated mixing, extrusion and molding equipment in a cleanroom environment is recommended for processing this material.

Due to the nature of the perfluoroelastomer materials, Dyneon PFE 301Z is best suited for open-mill mixing techniques. PFE 301Z, like most perfluoroelastomers, builds heat rapidly during mixing. For best results, it is recommended that roll temperature be controlled to between 20-30°C (70-90°F).

Keeping the polymer and the resulting compound relatively warm during the mixing process is critical to the success of mixing PFE 301Z. Avoid using cooling water on the mill unless necessary for handling. The best compound and mill-roll temperature is dependent on batch size and the equipment, and should be determined through experimental optimization.

1. Open the mill-gap slightly less than that of the PFE 301TZ crumb thickness and pass the crumb through the mill. Reduce the mill-gap with each pass until the tightest setting is achieved.
2. As heat builds and the gap tightens, the crumb will coalesce into a transparent gum. Manually “stretch” the gum (pulling it as the gum passes through the mill) 2-5 times before banding on the mill.
3. Band the gum on the mill once the polymer sticks to the roll (temperature related). Sweep any crumb that fell off the mill into the band. Avoid contaminating the band if the crumb is not clean.
4. Cut the gum and pull the bank through the mill-rolls 3-4 times to obtain homogeneous temperature in the polymer. Widen the mill-gap to reduce the bank to a minimum while maintaining a band around the mill-roll.
5. Dispersion of the 3M™ Dyneon™ PFE 301C catalyst can sometimes present problems with the end part appearance (discolored defects in larger cross section parts). To minimize this, it is recommended to carefully add the accurately weighed Dyneon PFE 301C in small amounts to the gum over a longer period of time, adding more cut-downs. Depending on the equipment and milling technique, this may not completely disperse the catalyst. Alternatively, solvents can be used to pre-disperse the catalyst prior to mixing. Please contact your 3M technical representative for additional information.

6. Continue adding small catalyst additions to the gum by cutting and passing the bank through the mill-rolls as in step #4 until all the catalyst has been incorporated and the gum is consistently translucent. Small concentrated bands of white in the gum mean the catalyst is not dispersed in those areas.
7. Once there are no large visible white streaks, perform a minimum of 6 gum cuts (3 each direction) and allow the bank to pass through before re-banding. Remove the gum from the mill.
8. Set the mill to the tightest setting, fold the slab together and cigar-roll through the mill a minimum of three times.
9. Open the gap on the mill to about 0.25 inch (7 mm) and pass the gum through the mill. Slowly increase the gap in the mill while passing the gum through until desired thickness is achieved. This will minimize potential air entrapment in the slab form.

Refreshing the Compound

Due to the high-viscosity nature of perfluoroelastomer compounds, care must be taken when refreshing the compound, especially if the compound is cold.

1. Open the mill-gap slightly more than the compound thickness and pass the compound through the mill. Reduce the mill-gap with each pass until the tightest setting is achieved. Note that failure to do so can result in equipment damage.
2. As heat builds and the gap tightens, the compound will begin to become smooth in texture. Manually “stretch” the gum (pulling it as the gum passes through the mill) 2-5 times before banding on the mill.
3. Band the gum on the mill once the compound sticks to the roll (temperature related).
4. Cut the gum and pull the bank through the mill-rolls 5-6 times to obtain homogeneous re-mix. Once the compound is deemed refreshed, open the gap on the mill to about 0.25 inch (7 mm) and pass the gum through the mill. Slowly increase the gap in the mill while passing the gum through until desired thickness is achieved. This will minimize potential air entrapment in the slab form.

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Note: When using solvents or other chemicals, be sure to follow the manufacturer's precautions and directions for use.

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- * Post cure conditions
- 1) Room Temperature to 150°C (302°F) over 1 hour
 - 2) Hold at 150°C (302°F) for 3 hours
 - 3) 150°C (302°F) to 300°C (572°F) over 6 hours
 - 4) Hold at 300°C (572°F) for 4 hours
 - 5) 300°C (572°F) to Room Temperature over 2 hours

Compound Shelf Life

Although 3M™ Dyneon™ PFE 301TZ and 3M™ Dyneon™ PFE 301C individually have shelf lives of 3 years from date of manufacture when stored under normal conditions in the original unopened 3M containers, once the two components are mixed, the shelf life of the compound is considerably less. If care is taken to store the mixed compound in a sealed container in a cool dry place, the compound shelf life has been shown to be approximately 5-6 weeks.

Refreshing the compound multiple times or extruding multiple times can shorten the shelf life. Care must be taken to keep track of the processing history of the product. Regardless of shelf life limitations, refreshing the compound multiple times also has the potential to increase contamination in the compound.

Extruding

Dyneon PFE 301Z perfluoroelastomer compounds are very easy to extrude. A thoroughly cleaned extruder should be utilized. An L/D ratio of 12:1 or higher is recommended. As with any elastomeric material, temperature and resonance time are key factors for this process. Initial processing setting recommendations are as follows at 25 RPM:

Feed Zone	50-70°C	Head	70-90°C
Screw	60-80°C	Die	70-90°C
Barrel	70-90°C		

Since differences in equipment and throughput dramatically change the shear, process settings should be developed for your specific application. Temperatures provided are for initial settings in this process development.

Molding

PFE 301Z perfluoroelastomer compounds show excellent moldability. Best practice is to prepare blanks of well-controlled dimension and weight. The recommended molding temperature range is 155-165°C (311-330°F). At this temperature, the material exhibits good flow behavior with excellent mold release. The surface-finish and the cleanliness of the mold all affect the appearance of the final molded part. A clean mold with a mold-finish of less than 15 µin gives the best results. The mold design including the flash grooves, land length, parting lines and plating material may be critical for complex shapes and or large diameter cross-sections and should be determined through experimental optimization.

Recommended Post Cure Conditions

This section describes the recommended post cure conditions for production parts using PFE 301Z. To eliminate yellowing while maximizing thermal and compression set resistance, a full post cure is recommended.

Effects of Post Curing on PFE 301Z Under Air* in Forced Air Oven

Compound	phr
PFE 301TZ (Polymer)	100
PFE 301C (Catalyst)	1.1

Press Cure 10 Minutes @ 165°C (330°F) Compression Set, %, [ASTM D395 Method B,-214 O-rings]

Property	
70 Hours @ 250°C (482°F)	
Press Cure Only	98
Full Post Cure	26

Full Post Cure Conditions

The full post cure conditions for PFE 301Z will vary depending on the oven load, temperature control of the oven and the thickness of the part being post cured. A programmable post cure oven controller is a must for post curing the PFE 301Z system since this product requires ramp up and ramp down to ensure part dimensional stability. Rapidly heating parts to above 150°C (302°F) or rapidly cooling parts that have a core temperature above 150°C (302°F) will often exhibit warping or deformation.

A full post cure condition can be described as:

1. Ramp up from Room Temperature to 150°C (302°F) over 45-60 minutes.
2. Hold at 150°C (302°F) for minimum of 3 hours. Typically 3-5 hours.
3. Ramp up from 150°C (302°F) to 300°C (572°F) over 5-6 hours.
4. Hold at 300°C (572°F) for between 4 hours and 24 hours depending on thickness of the part being post cured.
5. Ramp back to room temperature over 1-2 hours.

Notes: Use of gradual cooling is critical to maintaining the dimensional stability. The time needed to cool down will be dependent on oven loads and thickness of the part and should be determined through experimental optimization.

Oven Considerations

Exposure in a forced air oven can have an effect on part appearance. Care should be taken to protect the parts from direct air flow, yet provide adequate ventilation during the post cure cycle. Please see your 3M technical representative for more information.

Part Thickness and Post Curing

Part Thickness	Post Curing
Up to 0.103 inches (2.61 mm)	Hold the parts at 300°C (572°F) for a minimum of 4 hours, but longer times may be needed dependent on oven loads and should be confirmed through experimental optimization.
Over 0.103 to 0.139 inches (2.61 to 3.53 mm)	Hold the parts at 300°C (572°F) for a minimum of 8 hours, but longer times may be needed dependent on oven loads and should be confirmed through experimental optimization.
Over 0.139 inches (3.53 mm)	Data generated on 0.250 inch (6.35 mm) and 0.500 inch (12.7 mm) ASTM buttons have shown that a hold time of approximately 24 hours at 300°C (572°F) is adequate to generate a translucent part. For parts over 0.500 inch (12.7 mm) longer times may be needed and should be confirmed through experimental optimization.

Note: Data in this document are not for specification purposes.

300°C (572°F) Exposure Time

Data generated on samples post cured for up to 24 hours at 300°C (572°F) versus the standard 4 hours suggests no negative effects on the physical properties of the final part.

For more information please contact your 3M Representative.

Product Stewardship—Replacement Emulsifier: Dyneon™ and Dynamar™ products identified with a “Z” at the end of the product name indicate products that are made using a replacement emulsifier. This emulsifier, which Dyneon, a 3M subsidiary, began using in the manufacturing processes for these products in 2008, is a polymerization aid used to manufacture certain fluoropolymers and is not an intended ingredient in the polymers. The new emulsifier eliminates the use of the former polymerization aid, APFO (ammonium perfluorooctanoate, the ammonium salt of perfluorooctanoic acid (PFOA)), in the manufacture of these fluoropolymers. The use of the replacement emulsifier in the manufacture of these products is consistent with our product stewardship principles and our commitment to US EPA’s Voluntary PFOA Stewardship Program under which fluoropolymer manufacturers agreed to work towards eliminating PFOA in emissions and product content by the year 2015. **We are pleased to report that Dyneon completely eliminated the use of APFO in its manufacturing processes in December 2008.**

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