Free flow Granular Modified PTFE

PROCESSING GUIDE

INTRODUCTION

GFL offers a range of free flow granular modified PTFE under brand name INOFLON®, each chemically identical but having a different end uses. GFL offers free flow grades INOFLON® M290 & INOFLON® M295. Table-1 shows typical properties of INOFLON® Free flow granular modified PTFE grades.

BASICS

Modified PTFE is a polymer with high melting point similar to standard PTFE but has melt viscosity lower than standard PTFE. Non conventional processing methods similar to standard PTFE are used for modified PTFE such as powdered metal processing technology. There are four basic techniques for moulding granular modified PTFE. These techniques are- Billet and Sheet moulding, Automatic moulding, Isostatic moulding and Ram extrusion.

All of them are variations of compression moulding. These techniques are applied to convert granular resins into parts ranging in weight from a few grams to several hundred kilograms. The only continuous process for manufacturing parts from granular modified PTFE is called ram extrusion. This guide describes basic compression moulding of modified PTFE into shapes and articles for conversion to parts for end-use applications. Modified PTFE powder is compressed into a "preform" at ambient temperature. The reform has sufficient strength to be handled, roughly equivalent to blackboard chalk. After removal from the mould, the preform is heated in an oven above its melting point and is sintered. The consolidation of particles during sintering is referred to as coalescence, which produces a homogenous and strong structure. Varying the cooling rate, the crystallinity of the part can be controlled. (see figure 1)

PROPERTIES

Properties	Test Method	Unit	M690	M695
Bulk density	ASTM D 4894	g/l	700	700
Average particle size (d50)	ASTM D 4894	µm	475	475
Mould shrinkage	ASTM D 4894	%	4.0	4.0
Powder flow	Modified ASTM D 1895	g/min	400	350
Std. specific gravity (SSG)	ASTM D 4894	-	2.155	2.160
Melting point (Initial)	ASTM D 4894	°C (°F)	342 (648)	342 (648)
Melting point (Second)	ASTM D 4894	°C (°F)	327 (621)	327 (621)
Tensile strength	ASTM D 4894	MPa (psi)	30 (4351)	30 (4351)
Elongation	ASTM D 4894	%	400	400
Dielectric strength	ASTM D 149	kV/mm	77	80

Note- These are typical properties and not to be used for specification purpose

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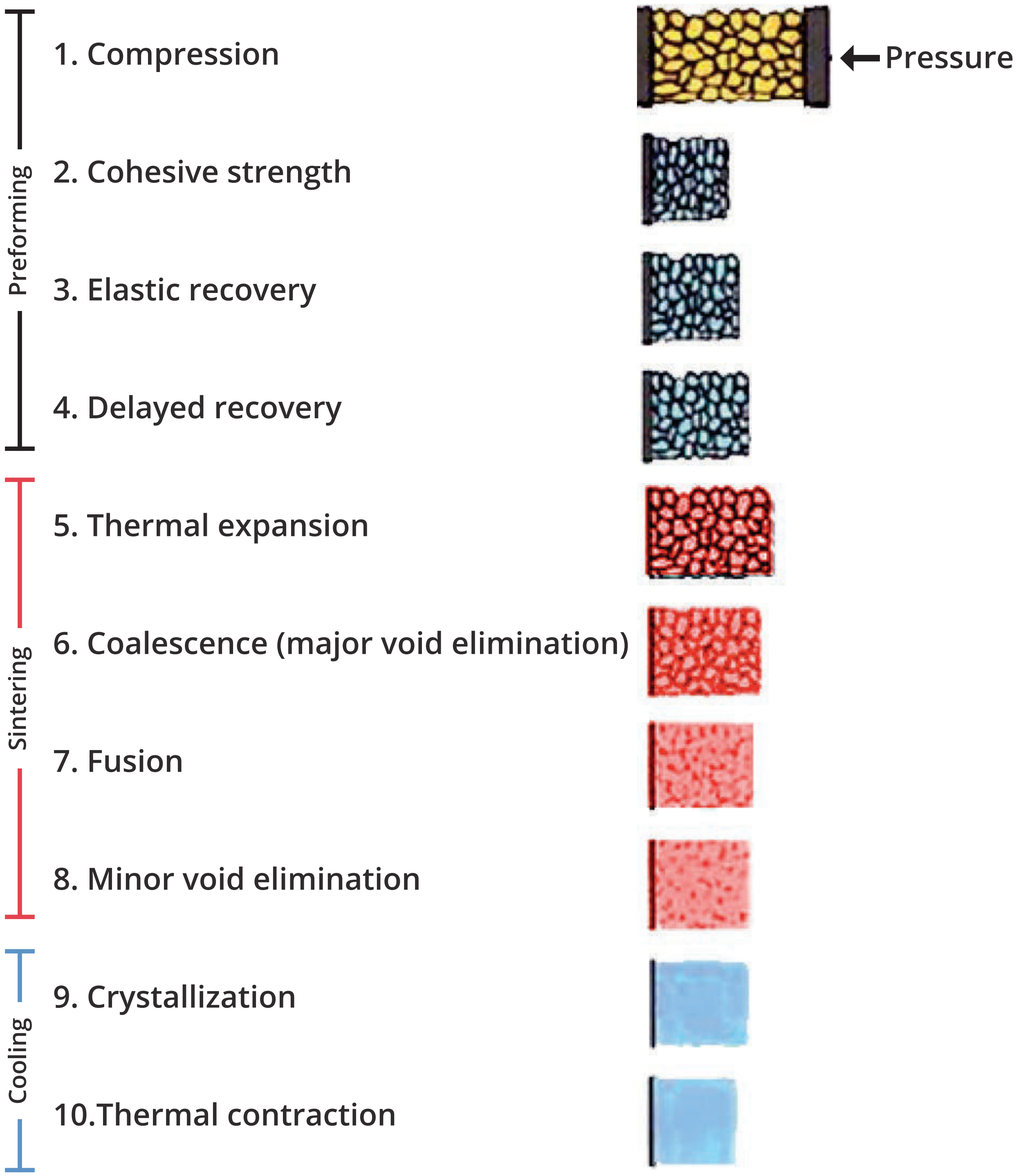

Figure 1- Schematic diagram of preforming and sintering sequence with modified PTFE

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PREFORMING

Before using the powder, it must be conditioned above 19°C (66°F). Preforming at temperatures in the range of 23–28°C (73–82°F) is most preferable. Resin temperature must be above 19°C (66°F) during moulding because of a special molecular transition of modified PTFE at 19°C (66°F) (see Figure 2). Modified PTFE molecule, which has a helical shape, tightens up by transition from a helix where 15 carbons are required for 180° turn to 13 carbons. Below 19°C (66°F) modified PTFE becomes stiff and less conformable, thus there is a chance that moulded parts could end up cracked. Modified PTFE powder becomes sticky, forms lumps and flow is reduced at temperatures above 28°C (82°F).

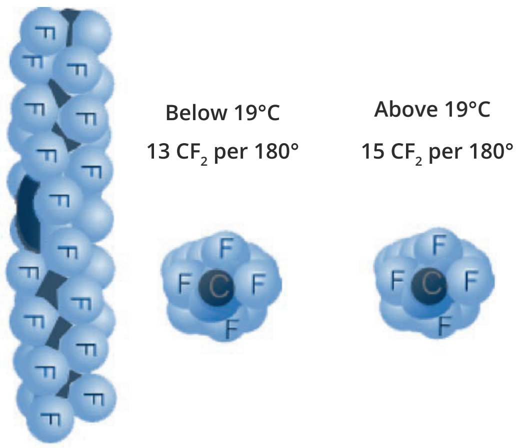

Figure 2- Transition of Modified PTFE Molecule

First the mould is filled manually with the resin. Next, it is compacted into a preform that has a shape similar to the final shape of the desired moulding. The preform is then placed in an oven where it undergoes heating and cooling cycles in which heating and cooling rates and dwell times are defined and programmed. The two cycles together are commonly called sintering cycle. The preform is heated to a temperature above the crystalline melting point of the resin during the sintering cycle. The cooling cycle is used to control the crystallinity of the part. The properties of a part are function of preforming pressure, dwell time, sintering time and temperature, and the cooling rate.

The general effect of preform pressure on the properties of INOFLON® Free flow granular modified PTFE grades can be seen in the Figure 3 to 4, when simple billets are moulded in the laboratory. A note of caution, processors must determine the optimal pressure in their own equipment. The exact required preform pressure depends on the type of process, part size and shape and application requirements.

Outgassing of the billets after moulding can be an issue that is more critical than for non-modified grades. Entrapped air from moulding process requires time to be released from the 'green' billet. A simple test for demonstrating this effect is to place a freshly moulded green billet in water: air bubbles are coming out at least for hours, big billets up to > 500 kg with wall thickness > 200 mm may need a few days for complete air release. In the production flow therefore it is recommended, to let big billets sit a few days after moulding before sintering process starts.

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Figure 3 - Effect of preforming pressure on tensile strength of INOFLON® Free flow granular modified PTFE grades

INOFLON® Modified PTFE Free flow Grades

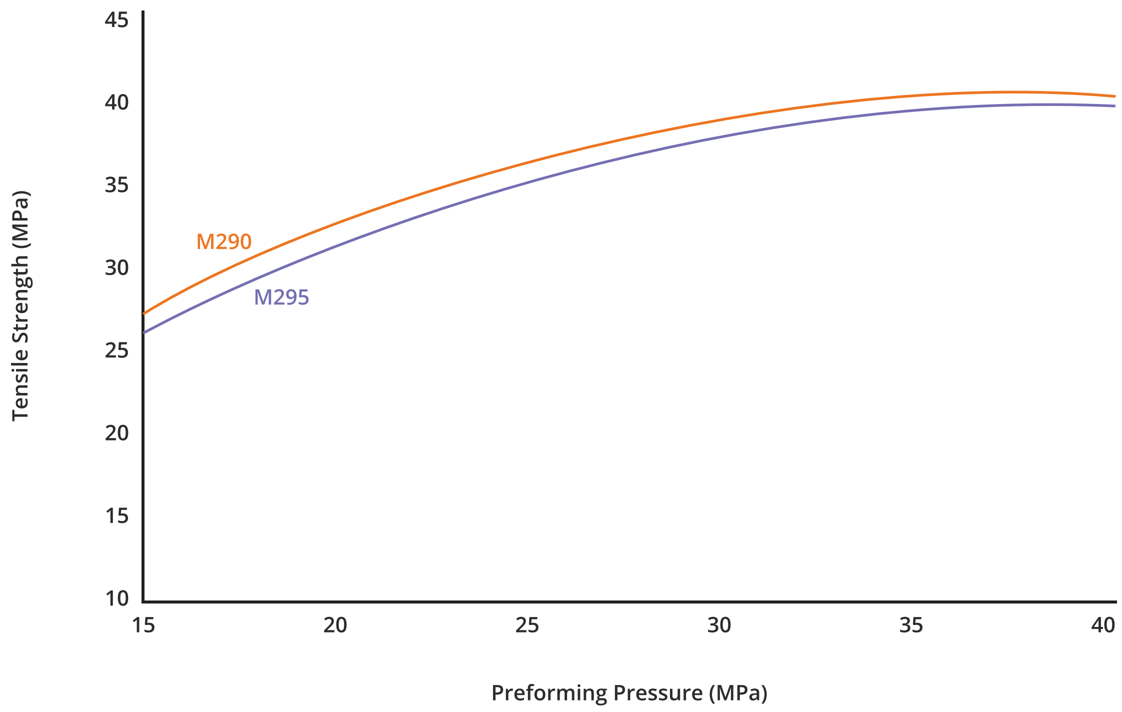

Figure 4 - Effect of preforming pressure on elongation of INOFLON® Free flow granular modified PTFE grades

INOFLON® Modified PTFE Free flow Grades

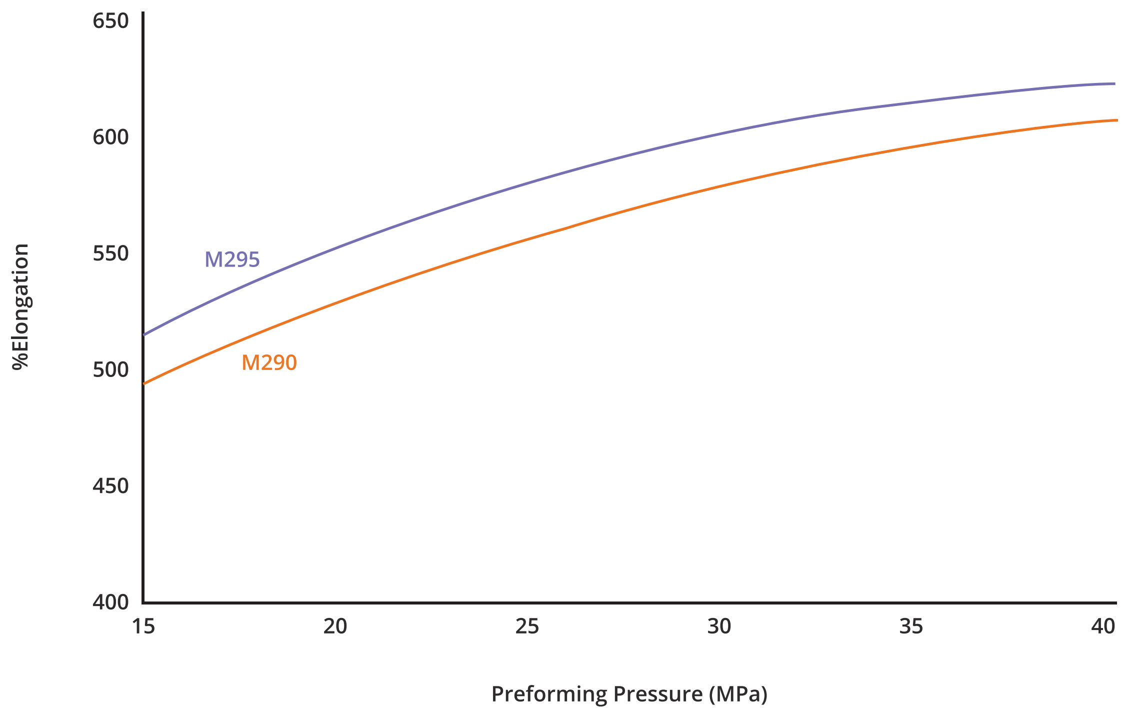

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Table 2- Recommended preforming pressure

INOFLON® Grades	Recommended preforming pressure (MPa)
M290	30-35
M295	30-35

SINTERING

A modified PTFE preform has limited cohesive strength and is essentially useless; sintering allows coalescence of the resin particles, which provides strength and void reduction. Sintering cycle profiles of time and temperature affect the final properties of the billet. Sintering temperatures exceed the melting point of PTFE 342°C (648°F) and range from 365°C to 375°C (689–707°F).

Similar sinter cycle is recommended for INOFLON® modified PTFE as for standard INOFLON® PTFE. Especially or big billets, Elephants foot formation may occur when sintered high billets statically in a vertical position.

Sintering in a horizontal position by simultaneously rotation helps to avoid elephant's foot formation totally. Elephant's foot formation is always connected with internal stress causing wavy films when skived.

When heating up billets in the first phase of the sinter cycle, modified PTFE is being molten from the outside to the inside of the wall. As soon as the outside gets molten, the billet surface is 'closing' and a high barrier for air release is being formed. Due to the excellent fusing performance of the modified PTFE particles, the closing phenomenon is more critical for modified PTFE than for standard PTFE. That's why air release prior to sintering is so important for modified PTFE. In case, there is no time in production flow for sufficient air release, a temperature dwell plateau below the crystalline melting temperature, i.e. at around 310-320 °C (590-608 °F) , will also support efficient air release before gel-closing at the outside.

Figure 7 and Figure 8 provide examples of sintering cycles for a variety of cylindrical shapes and dimensions. These examples should be used as a conservative starting cycles, which allow a margin for shortcomings in the moulding and degassing operations. They can be further refined, optimized and possibly shortened by processors.

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Figure 7- Typical sintering cycle for INOFLON® Free flow granular modified PTFE, Solid billets (Ref. - Table 3)

Typical Sintering Cycle

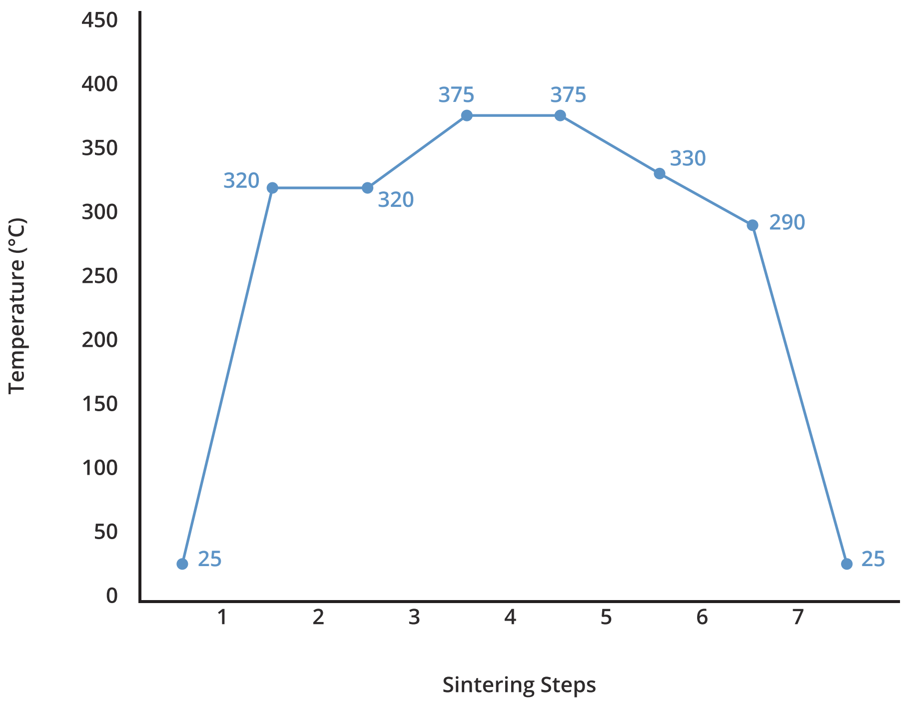

Table 3-Typical sintering cycle for INOFLON® Free flow granular modified PTFE, Solid billets:

Cycle	Diameter (mm)	Time Duration (Hours)							Total
Sintering Steps	→	1	2	3	4	5	6	7	Time (hrs)
A	25	3	1	1	3	1	1	3	13
B	50	3.5	2	1.5	4	2	1.5	3.5	18
C	75	4	2.5	2	5	2.5	2	4	22
D	100	4	3.5	2.5	6	3.5	4.5	4	28
E	125	4.5	4	3	7	5	5	4.5	33
F	150	5.5	4.5	3.5	9	6	6	5.5	40

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Figure 8- Typical sintering cycle for INOFLON® Free flow granular modified PTFE, Annular billets (Ref. - Table 4)

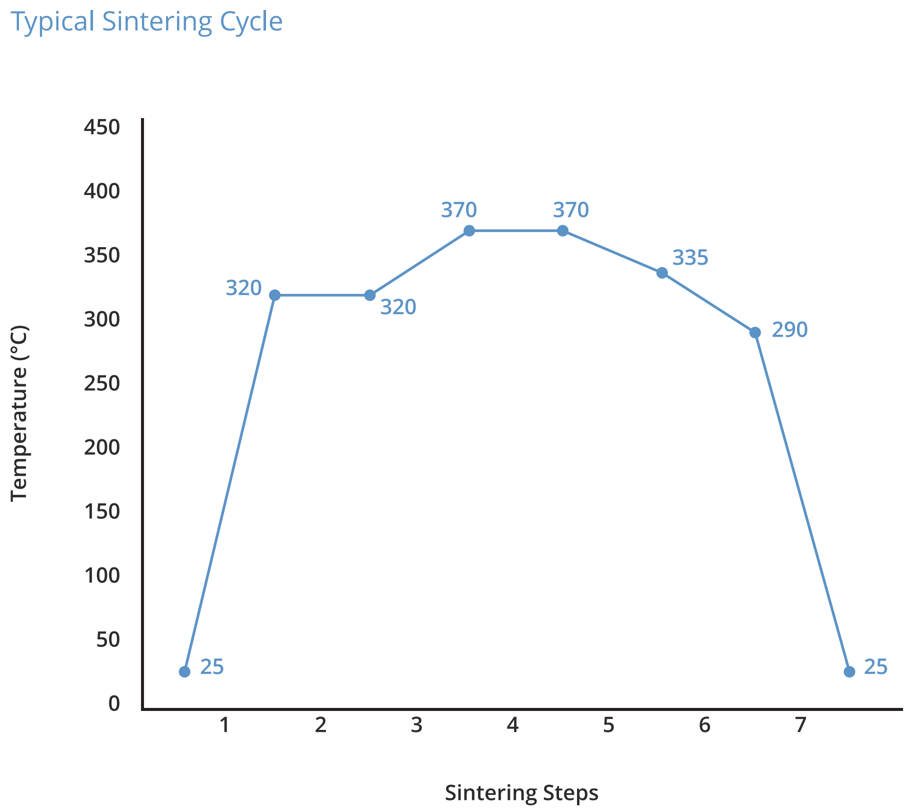

Table 4 -Typical sintering cycle for INOFLON® Free flow granular modified PTFE, Annular billets:

Cycle	Wall thickness (mm)	Time Duration (Hours)							Total
Sintering Steps	⟶	1	2	3	4	5	6	7	Time (hrs)
A	25	3	1.5	1	3	2	1.5	3	15
B	50	4	3.5	1.5	5.5	4	2.5	3	24
C	75	6	5	2.5	8.5	5.5	4.5	5	37
D	100	8	7	3	12	8	5	7	50
E	125	9.5	9	4	14	9	6.5	8	60
F	150	11.5	10.5	5	16.5	11	7.5	10	72

ANNEALING STEPS

A typical stress annealing cycle looks as follows:
After placing the pre-machined parts in a sinter oven, enhance the temperature with full heating power of the oven up to 260–280 °C (500-536 °F). The higher the temperature in this temperature range, the more efficient the stress annealing process.

REMARKS

All stress annealing cycles have to stay with maximum temperature below the melting temperature of 327°C (621°F). As soon as the melting temperature is exceeded, the whole story' begins from zero again. Stay at temperature 260 – 280°C (500-536°F) at least for 2 – 6 hours. Dwell time depends on part dimensions and stress release requirement.

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Cooling down speed can be at – 1°C per minute or even faster. Opening the oven at 100 – 80°C (212-176°F) for even faster cooling is possible too. Start machining not before the parts have reached ambient temperatures.

MACHINING

Same machining tools are recommended to use as for standard PTFE.
If complex shapes are being machined to accurate final dimensions, one or even more than one stress annealing steps are recommended to place between the different machining steps.

SAFTEY PRECAUTIONS

Handling and processing of PTFE must be done in ventilated area to prevent personnel exposure to the fumes liberated during sintering and heating of the resin. Fumes should not be inhaled and eye and skin contact must be avoided. In case of skin contacts wash with soap and water. In case of eye contact flush with water diately and seek medical help. Smoking tobacco or cigarettes contaminated with PTFE may result in a flu-like condition including chills, fever and sore throat that may not occur for a few hours after exposure has taken place. This symptom usually passes within about 24 hours.

Vapors and gases generated by PTFE during sintering must be completely removed from the factory areas. Mixtures of some metal powders such as magnesium or aluminum are flammable and explosive under some conditions. Please read the Material Safety Data Sheet and the detailed information in the “Guide to the Safe Handling of Fluoropolymer Resins” published by the Fluoropolymer Division of The Society of the Plastics Industry available at www.fluoropolymers.org

HANDLING AND STORAGE

For best results the powder processing areas should be kept clean and free of all contamination. Organic contamination and foreign matter usually appear as dark spots often easily visible against the white PTFE background. Organic contamination material degrades at the sintering temperatures and forms discolored spots. They oxidize away as carbon dioxide wherever sufficient oxygen exposure takes place. It is, therefore, not unusual to encounter discoloration inside a part away from the surface where hardly any oxygen is present. Storage of PTFE at 20°C (68°F) or lower prevents lump formation as a result of movement and transportation.

SALES AND TECHNICAL SUPPORT

Corporate & Marketing office:
Gujarat Fluorochemicals Limited
Inox Towers, Plot no. 17, Sector 16/A
Noida-202301, U.P., INDIA
Tel: +91-120-6149600
Fax: +91-120-6149610

Europe
Gujarat Fluorochemicals GmbH
Esplanade 40, 9th Floor
20354 Hamburg, Germany
+49 040 5582 395- 80

Works
Gujarat Fluorochemicals Limited
12/A, GIDC Dahej Industrial Estate.
Tehsil- Vagra, Distt. Bharuch-392230, Gujarat, INDIA
Website: www.fluonox.co.in; www.gfl.co.in
Email: contact@gfl.co.in

Americas
GFL Americas, LLC
1212 Corporate Dr., Suite-540,
Irving, TX 75038, USA
+1 512 446 7700

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