

PROCESSING GUIDE

Free flow Granular PTFE

INTRODUCTION

GFL offers a range of free flow granular PTFE under brand name INOFLON[®], each chemically identical but having a different end uses. GFL offers free flow grades INOFLON[®] 210 & INOFLON[®] 220, semi free flow grade INOFLON[®] 230

Table-1 shows typical properties of INOFLON[®] Free flow granular PTFE grades.

BASICS

PTFE is a polymer with high melting point and melt viscosity. The first melting point of PTFE is 342°C (648°F) and the melt viscosity is in the range of 10^{11} – 10^{12} poise at 380°C (716°F). This high melt viscosity inhibits any flow similar to that known for other thermoplastics. Hence non conventional processing methods have been developed to accommodate PTFE's unique properties based on powdered metal processing technology. The main fabrication process is a modified version of compression moulding of metallurgical powders. There are three basic techniques for moulding suspension polymerized or granular PTFE. These techniques are- Billet & Sheet moulding, Automatic moulding and Isostatic moulding.

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 PTFE is called ram extrusion.

This guide describes basic compression moulding of PTFE into shapes and articles for conversion to parts for end-use applications. PTFE powder is compressed into a "preform" at ambient temperature. The preform 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)

Table 1- Typical properties of INOFLON[®] Free flow granular PTFE grades

Properties	Test Method	Unit	210	220	230
			Free flow		Semi free flow
Bulk density	ASTM D 4894	g/l	700	775	750
Average particle size (d_{50})	ASTM D 4894	µm	600	500	300
Mould shrinkage	ASTM D 4894	%	2.50	2.25	2.25
Powder flow	Modified ASTM D 1895	g/min	400	400	-
Std. specific gravity (SSG)	ASTM D 4894	-	2.155	2.155	2.155
Melting point (Initial)	ASTM D 4894	°C (°F)	342 (648)	342 (648)	342 (648)
Melting point (Second)	ASTM D 4894	°C (°F)	327 (621)	327 (621)	327 (621)
Tensile strength	ASTM D 4894	MPa (psi)	30 (4351)	30 (4351)	30 (4351)
Elongation	ASTM D 4894	%	275	275	275

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

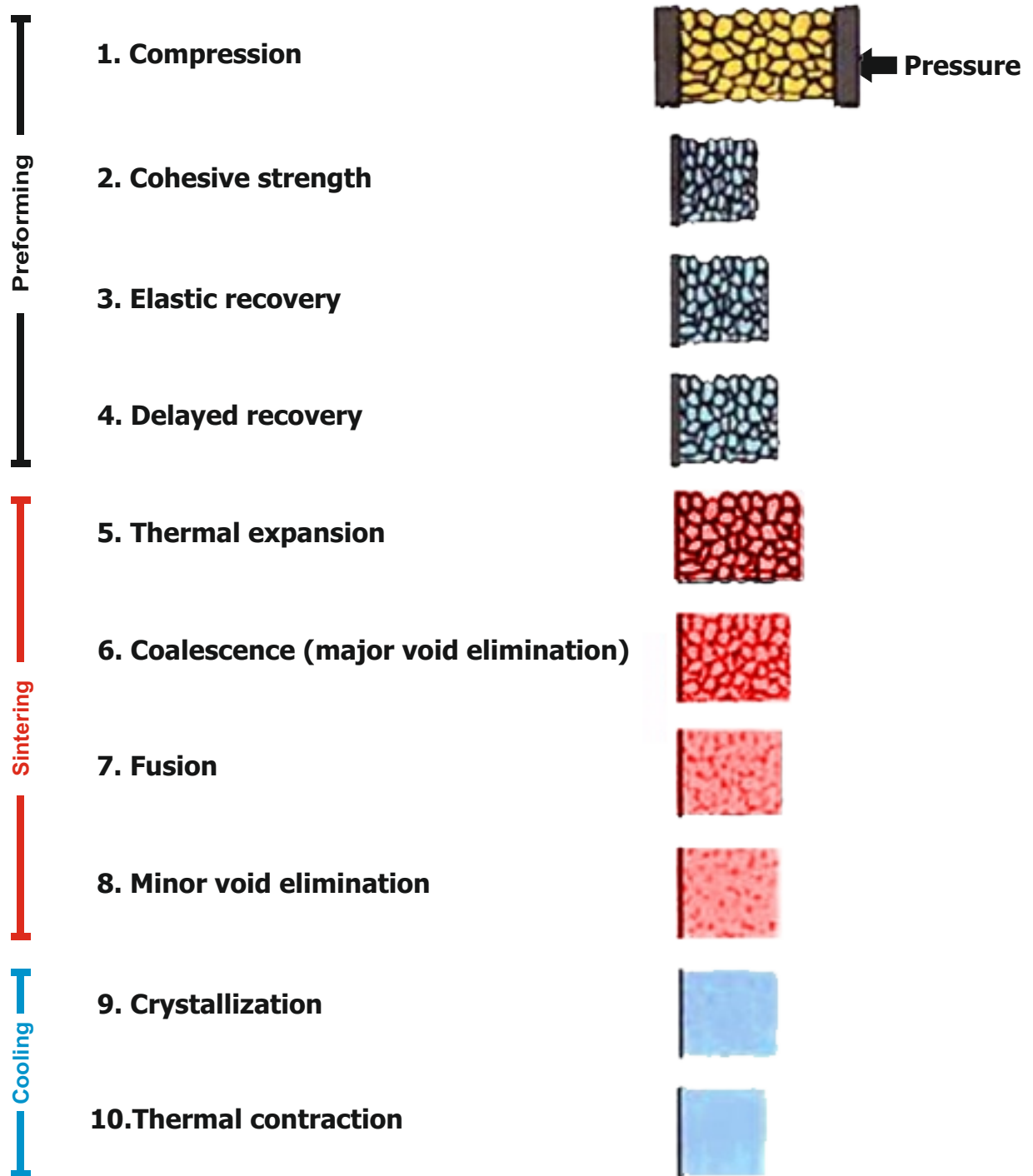


Figure 1- Schematic diagram of preforming and sintering sequence with PTFE

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 PTFE at 19°C (66°F) (see figure 2). 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) PTFE becomes stiff and less conformable, thus there is a chance that moulded parts could end up cracked. PTFE powder becomes sticky, forms lumps and flow is reduced at temperatures above 28°C (82°F).

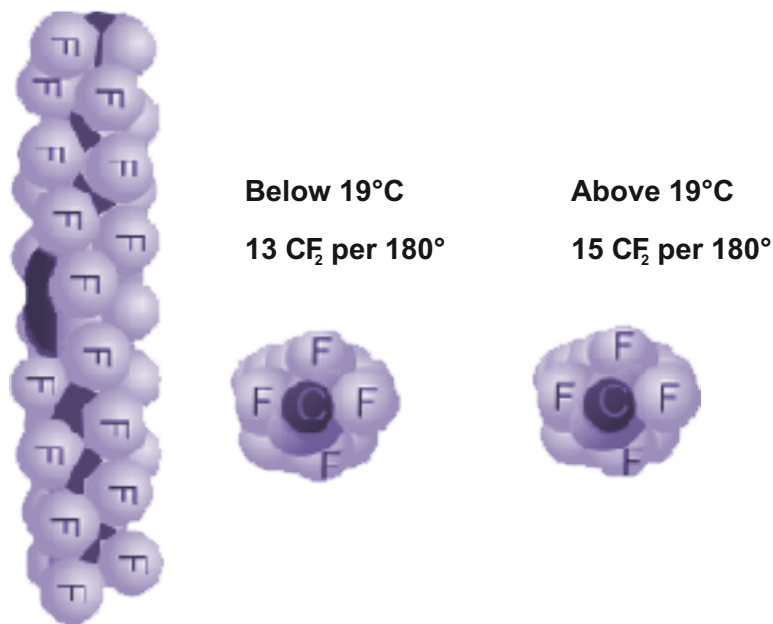


Figure 2- Transition of PTFE Molecule

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 PTFE grades can be seen in the Figure 3 to 4, when simple billets are moulded in the laboratory. One important observation is that INOFLON® grades are highly mouldable resins and yield excellent properties at relatively low preform pressures. 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, shape and application requirements.

Generally, the lower range of the preform pressure is likely to result in lower end use properties of the finished part, especially in large parts. Preform pressure has a direct bearing on void closure and part properties in applications such as electrical insulation uses. Preform pressure has to be optimized for an application as mould shrinkage and tensile properties vary with preform pressure, sintering conditions and geometry of the part.

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

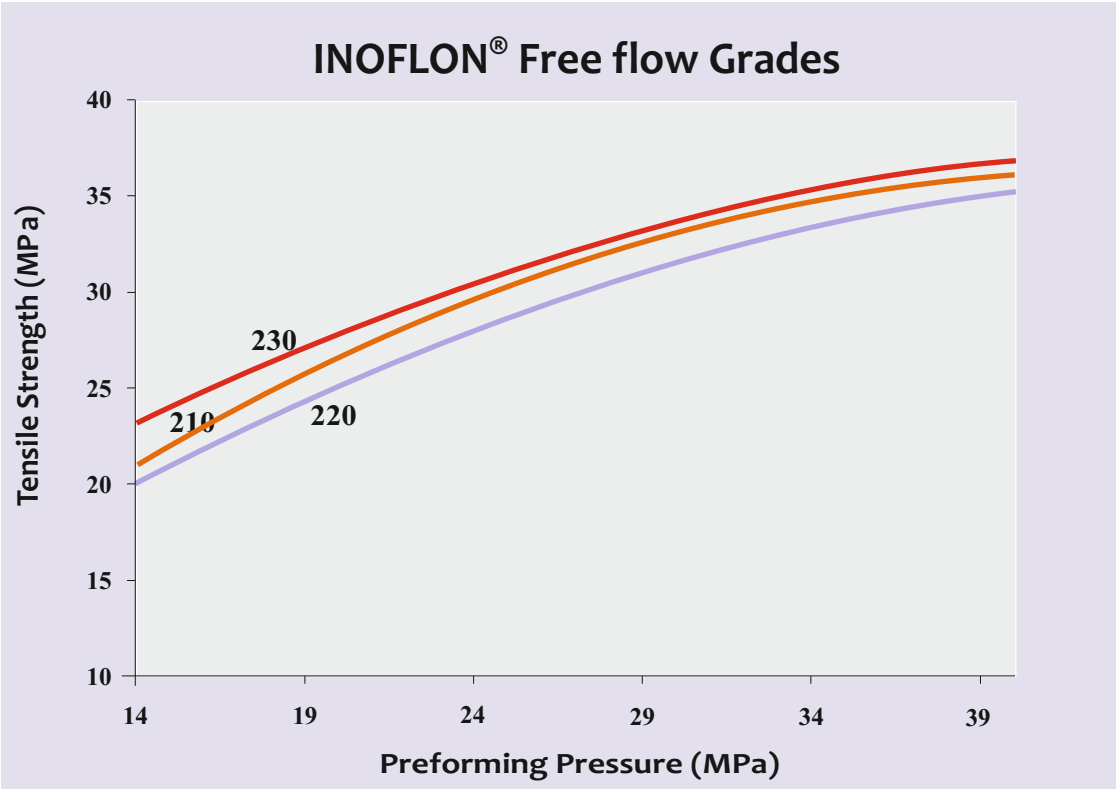


Figure 4 - Effect of preforming pressure on elongation of INOFLON® Free flow granular PTFE grades

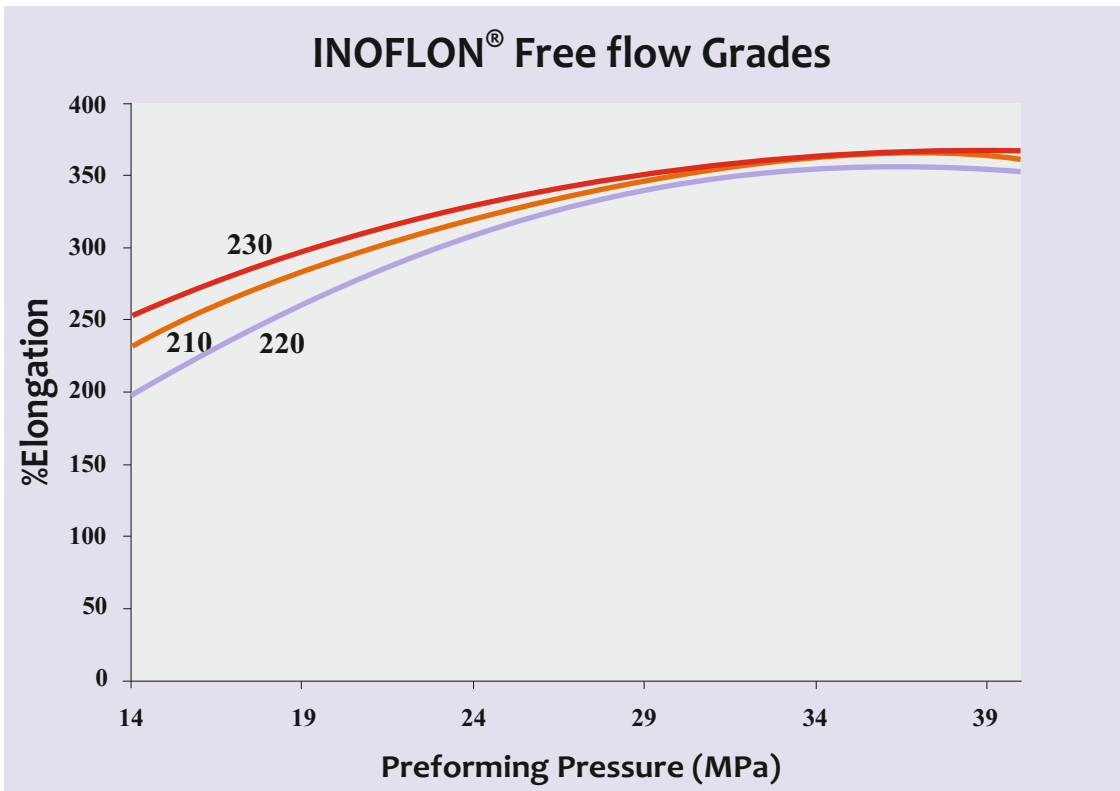


Table 2- Recommended preforming pressure

INOFLON® Grades	Recommended preforming pressure (MPa)
210	30-35
220	30-35
230	25-30

Coalescence and void elimination require time because of the limited mobility of PTFE molecules. The sintering temperature is held for a period of time to allow fusion, coalescence and void elimination to proceed and maximize properties in the part. A time is reached beyond which the part properties no longer improve and degradation begins. Property development should be balanced against cost in selecting a sintering cycle.

SINTERING

A 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 360°C to 380°C (680–716°F).

Various steps of the sintering process are described here. First the preform completes its elastic recovery and begins to thermally expand past the PTFE melting point, 342°C (648°F). The expansion can reach up to 25–30% by volume depending on the type of resin, powder, preforming pressure and temperature.

Above 342°C (648°F), PTFE is a transparent gel due to the absence of a crystalline phase. At the sintering temperature, adjacent melted PTFE particles fuse together and coalesce. After two particles have completely coalesced, they would be indistinguishable from a larger particle and voids are eliminated under the driving force of surface tension. Smaller particle resins and higher preform pressures improve coalescence.

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.

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

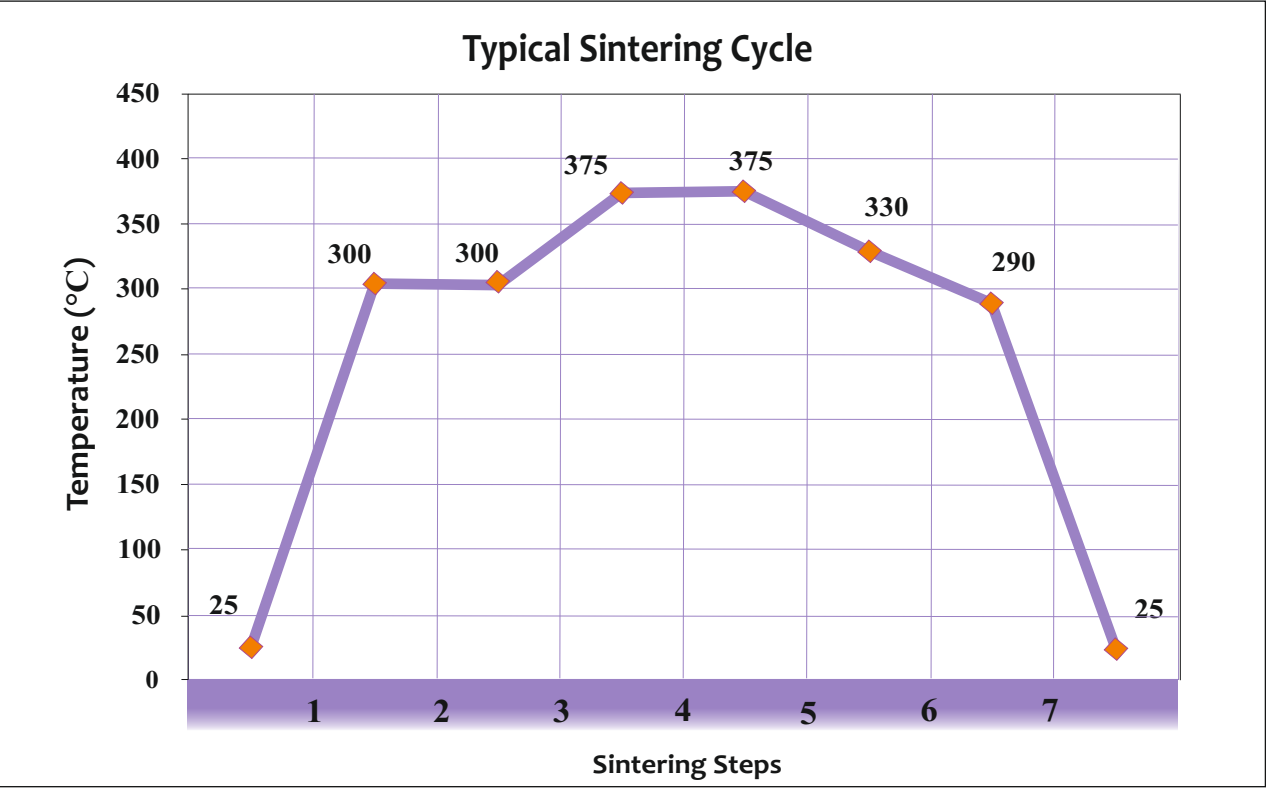


Table 3-Typical sintering cycle for INOFLON® Free flow granular PTFE, Solid billets:

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

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

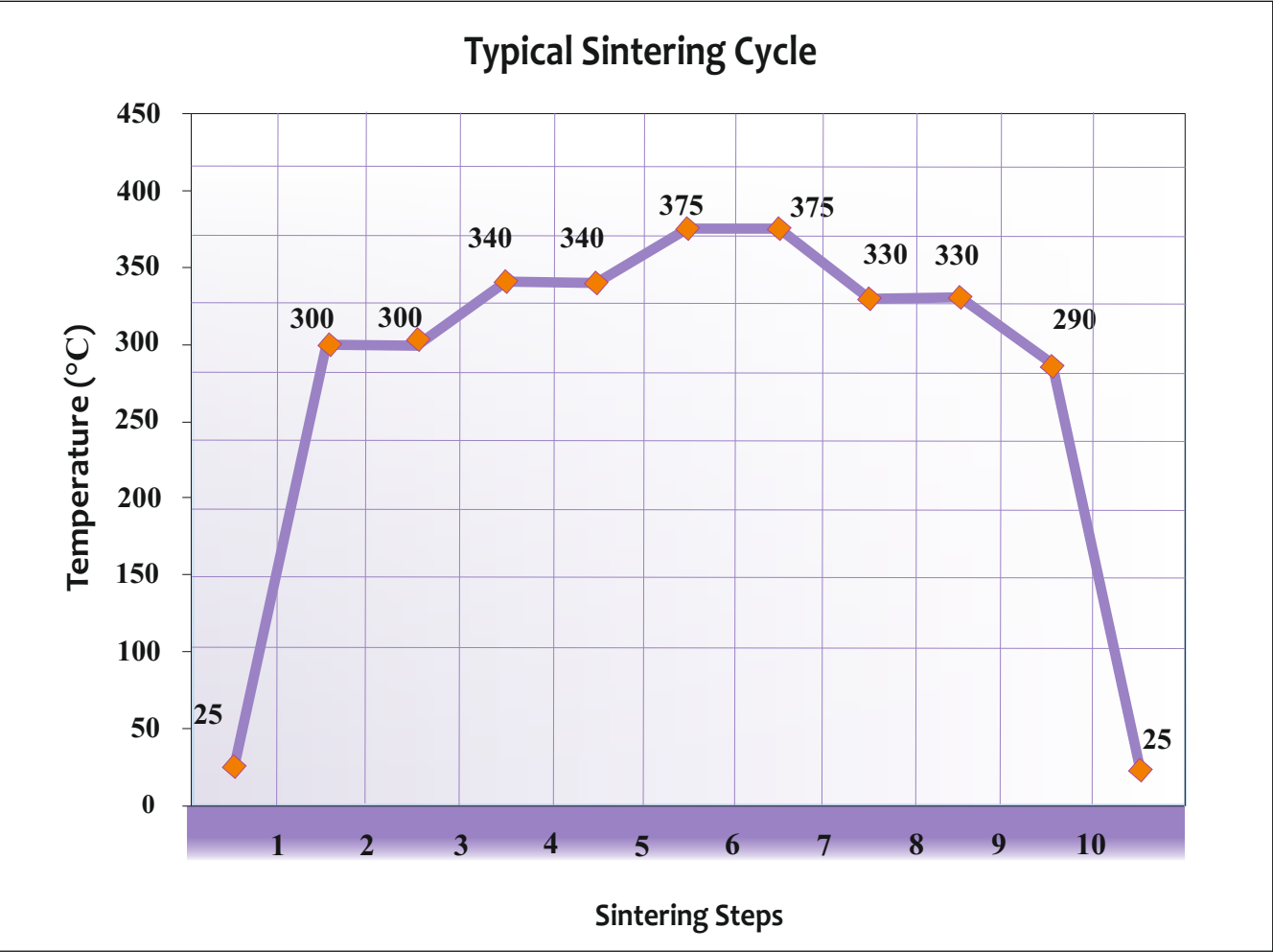


Table 4 -Typical sintering cycle for INOFLON® Free flow granular PTFE, Annular billets:

Cycle	Wall Thickness (mm)	Time Duration (Hours)										Total
Sintering Steps	→	1	2	3	4	5	6	7	8	9	10	Time (hrs)
A	25	3	1	1	0.5	1	3	2	0.5	2	3	17
B	50	3.5	3	1.5	1	2	6	3.5	1	4	3.5	29
C	75	5.5	4	2	1	2	9	5.5	1.5	6	5.5	42
D	100	6.5	6	3	1.5	2	11	7.5	2	8	6.5	54
E	125	8.5	8	4	2	2.5	14	9	2.5	10	8.5	69
F	150	10.5	10	4.5	2.5	3	17	11	3	12	10.5	84

SAFETY 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 immediately 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.

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NOTE warning: Do not use any of INOFLON® PTFE resins in medical devices that are designed for permanent implantation in the human body. For other medical uses, prior permission of GFL may be sought.

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