Polymer Composites with Hollow Glass Microspheres: Processing, Properties and Applications

Baris Yalcin, PhD
Product and Application Development Engineer

3M Company
Advanced Materials Division

November 6, 2012

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Outline

Overview of 3M™ Glass Bubbles

- Physical Properties of Hollow Glass Microspheres (HGM)
- Composition, Strength, Density and Particle Size

Formulating with Hollow Glass Microspheres

Polymer Processing with Hollow Glass Microspheres

- Compounding, Extrusion
- Injection Molding

Benefits of Hollow Glass Microspheres

- Material
- Processing

Application Examples

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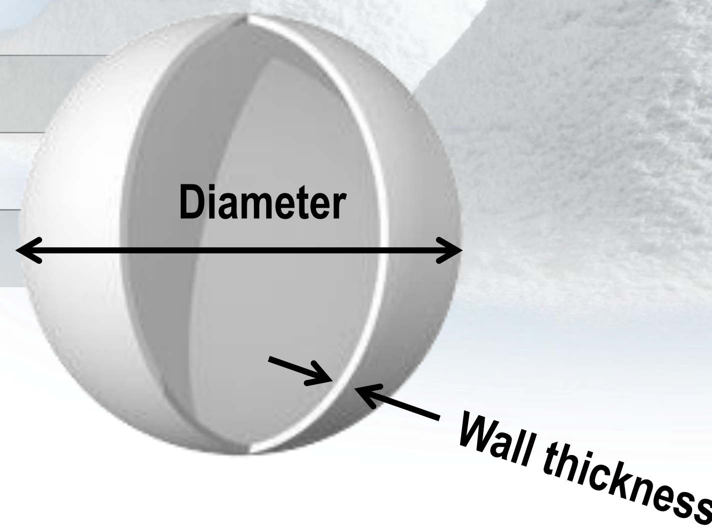
3M™ Glass Bubbles

Overview

3M™ Glass Bubbles

Property	Value
Shape	Hollow, thin walled, unicellular spheres
Composition	Soda-lime borosilicate glass
Color	White
True Density†	0.12 - 0.60 g/cc
Crush Strength*	250 – 28,000 psi
Hardness	Mohs scale 5
Softening Temp	600° C
Size	15 - 65 microns



•ASTM D 3102-78 Standard Practice for the Determination of Isostatic Collapse Strength of Hollow Glass Microspheres (withdrawn in May 1984)

† Helium Gas Pycnometer

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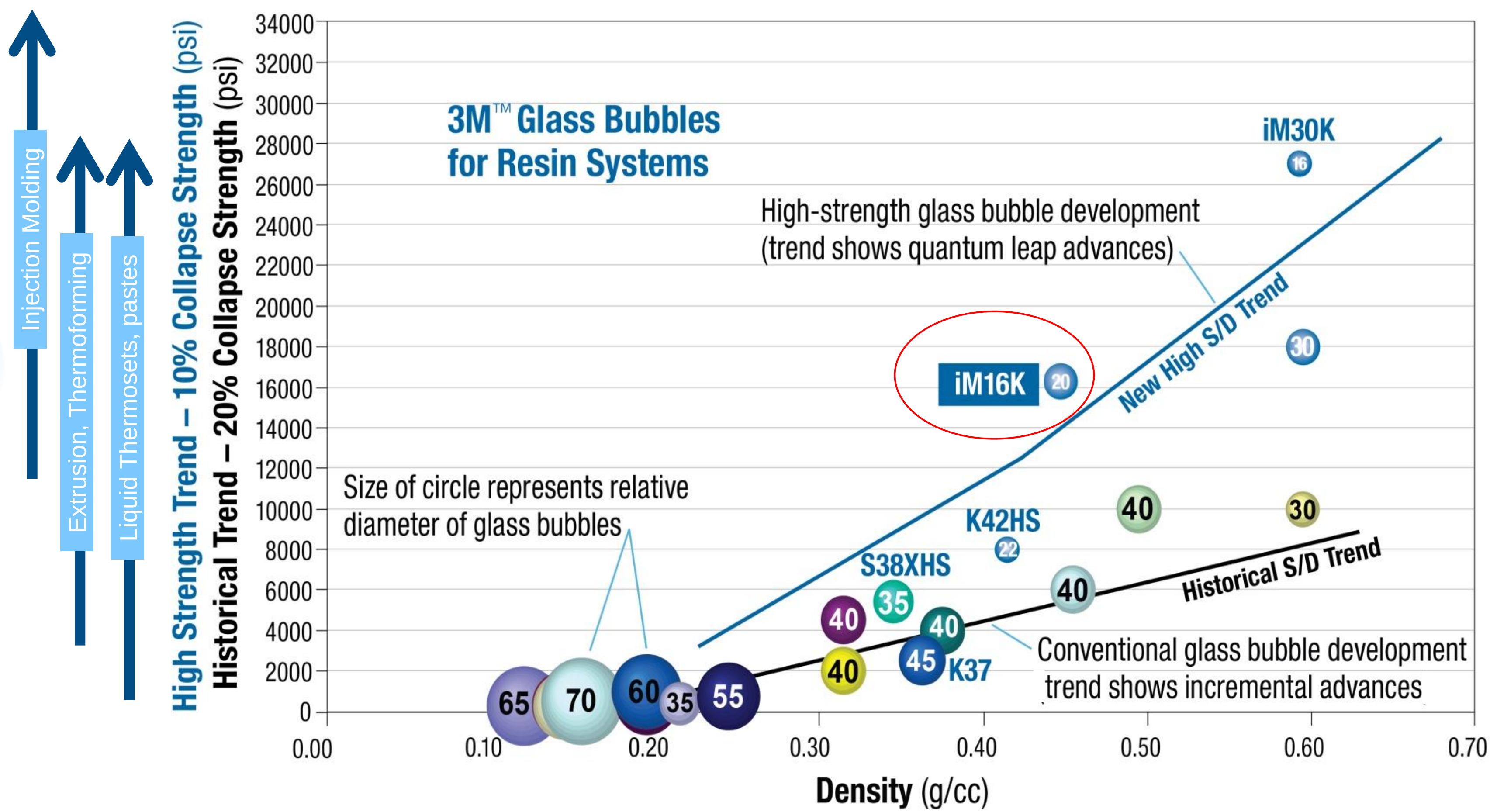
3M™ Glass Bubbles Isostatic Collapse Strength



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Formulating with Hollow Glass Microspheres

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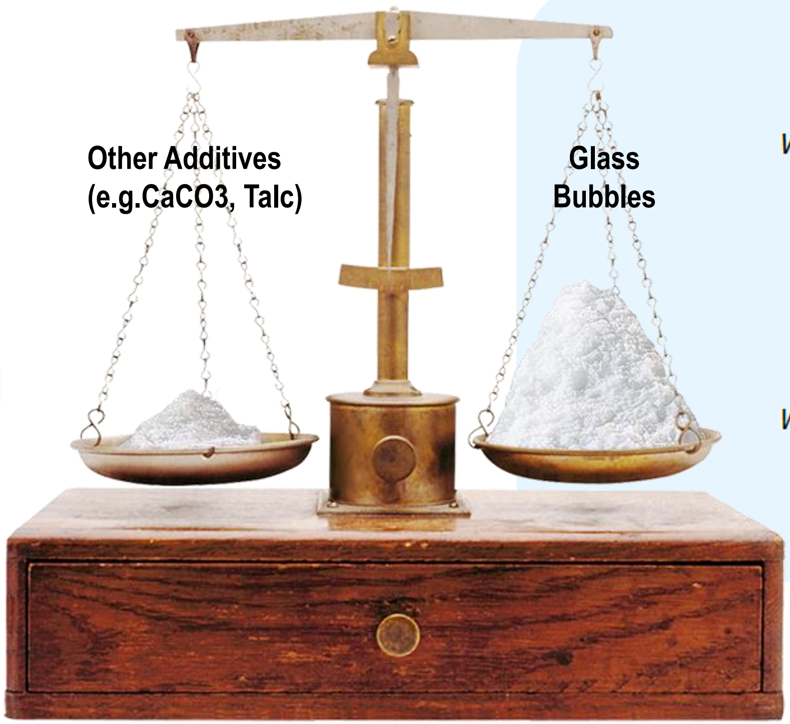
Formulating with GBs on Volume Basis



$$vol\%\ GB = \frac{\dfrac{\text{wt\% GB}}{\text{density of GB}}}{\dfrac{\text{wt\% GB}}{\text{density of GB}} + \dfrac{\text{wt\% polymer}}{\text{density of polymer}}}$$

or

$$wt\%\ GB = \frac{\text{vol\% GB} \times \text{density of GB}}{(\text{vol\% GB} \times \text{density of GB}) + (\text{vol\% polymer} \times \text{density of polymer})}$$

ALWAYS THINK IN TERMS OF VOLUME LOADING

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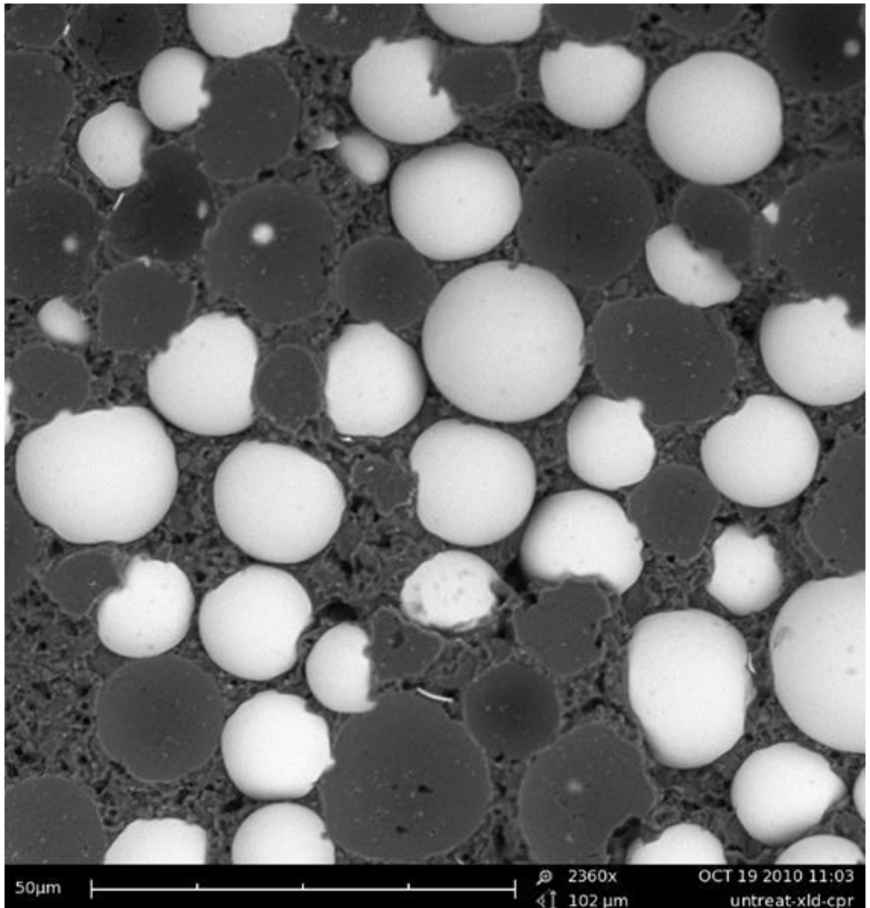
Maximum practical GB loading



Scanning electron microscopy

45 vol%

Component	Density g/cc	Weight %	Volume %
PP	0.9000	64.000	54.237
GB	0.6000	36.00	45.76
Totals	**0.7627**	**100.000**	**100.000**

Component	Density g/cc	Weight %	Volume %
PP	0.9000	70.000	54.392
GB	0.4600	30.00	45.61
Density	**0.6993**	**100.000**	**100.000**

Component	Density g/cc	Weight %	Volume %
PVC	1.4000	74.000	54.950
GB	0.6000	26.00	45.05
Totals	**1.0396**	**100.000**	**100.000**

Component	Density g/cc	Weight %	Volume %
PVC	1.4000	78.500	54.539
GB	0.4600	21.50	45.46
Totals	**0.9727**	**100.000**	**100.000**

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Formulating with GBs- TPOs

		Function
Polymer Phase	PP (Polypropylene) homo, co, high crystallinity) PE (polyethylene)	Main Matrix
Elastomer Phase	EPR (Ethylene propylene rubber) EPDM (EP-diene rubber) EO (ethylene-octene), EB (ethylene-butadiene) SEBS (Styrene-ethylene-butadiene-styrene)	Improve cold temperature impact properties
Reinforcing Filler Phase	Talc, Nano Clay, Mica Glass fiber (Short, Long), wollastonite, whiskers, ceramic fibers	Increase stiffness (strength, modulus), HDT
Additives	Pigments, Stabilizers	UV, Heat, etc

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Comparison of 3M™ Glass Bubbles to typical fillers used in TPOS

•Mica,
•Talc
•Clay (e.g. Montmorillonite)

•Glass Fiber
•Wollastonite
•Metal oxide whisker

Hollow Glass Microspheres	Mica, Talc, Clay	Glass Fiber, Wollastonite, Metal oxide whisker
0.46 -0.6 g/cc	2.8 g/cc	2.5 g/cc
1:1	20:1	30–50:1

Low —— **Aspect Ratio** ——→ High

Due to the differences in density, size and aspect ratio, glass bubble containing recipes need to be carefully formulated to maintain a good balance of mechanical properties while reducing density

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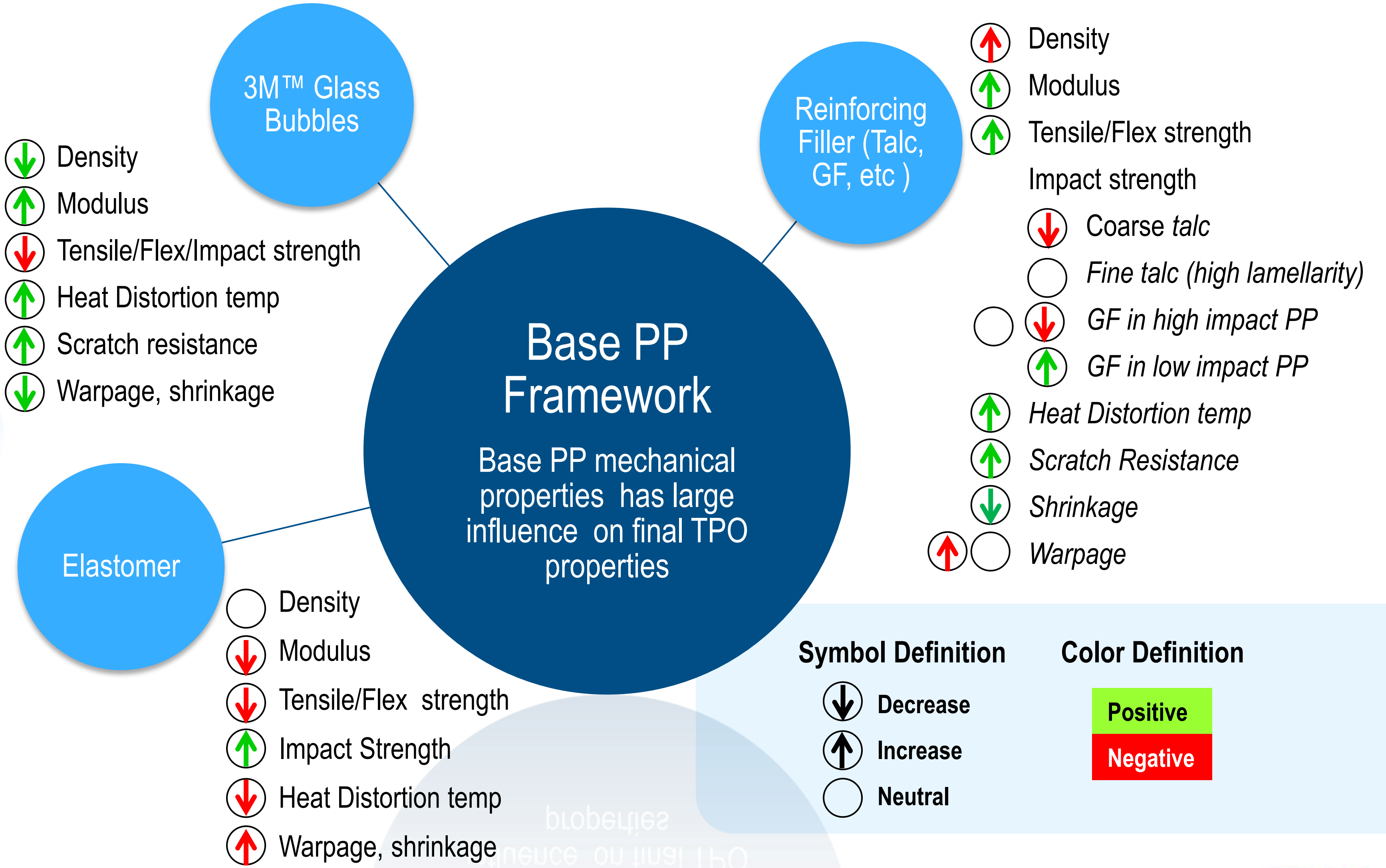
Effect of Major Components on TPO Properties



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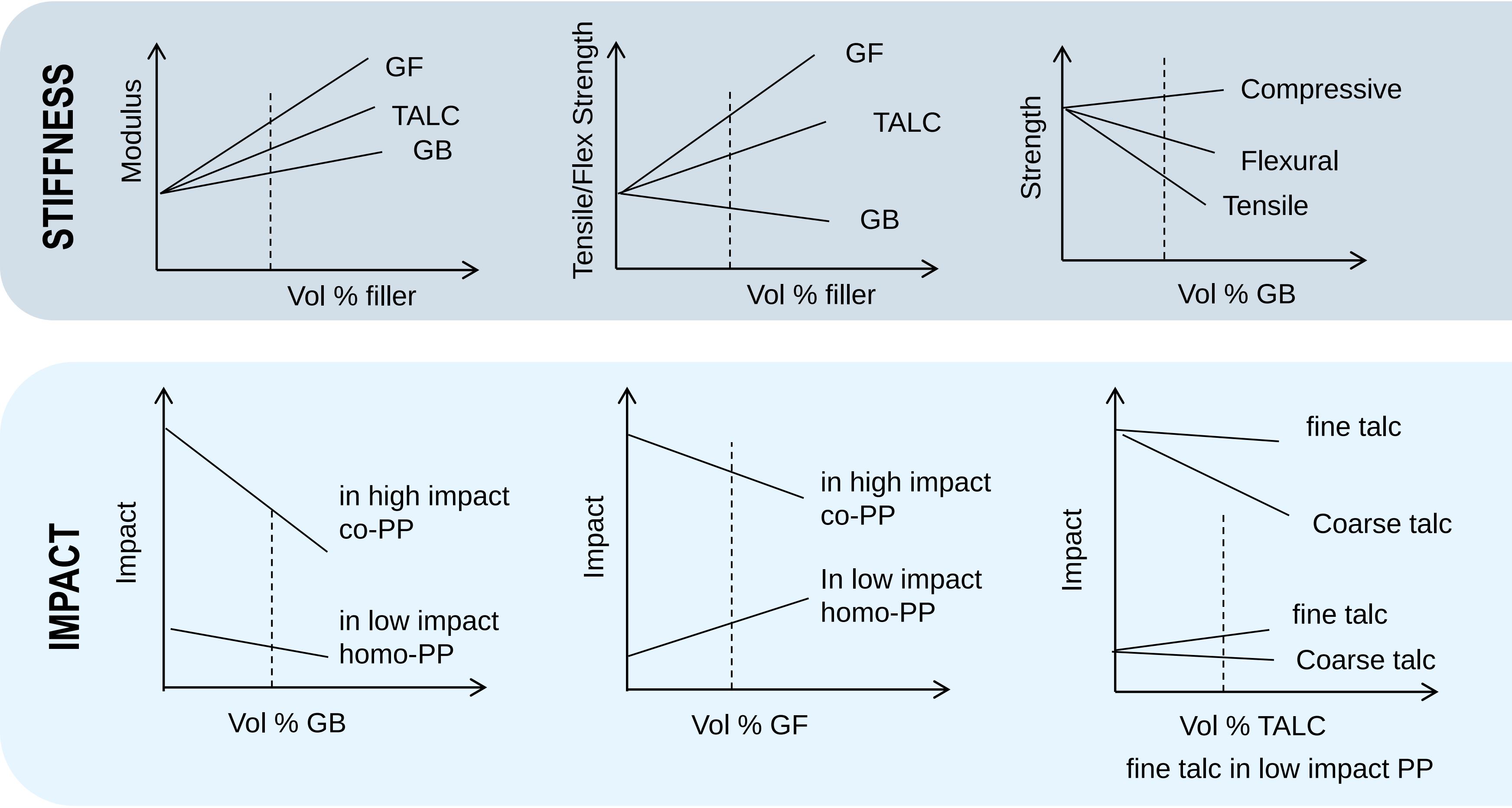
Extent of Individual Contribution of Fillers to Stiffness and Impact



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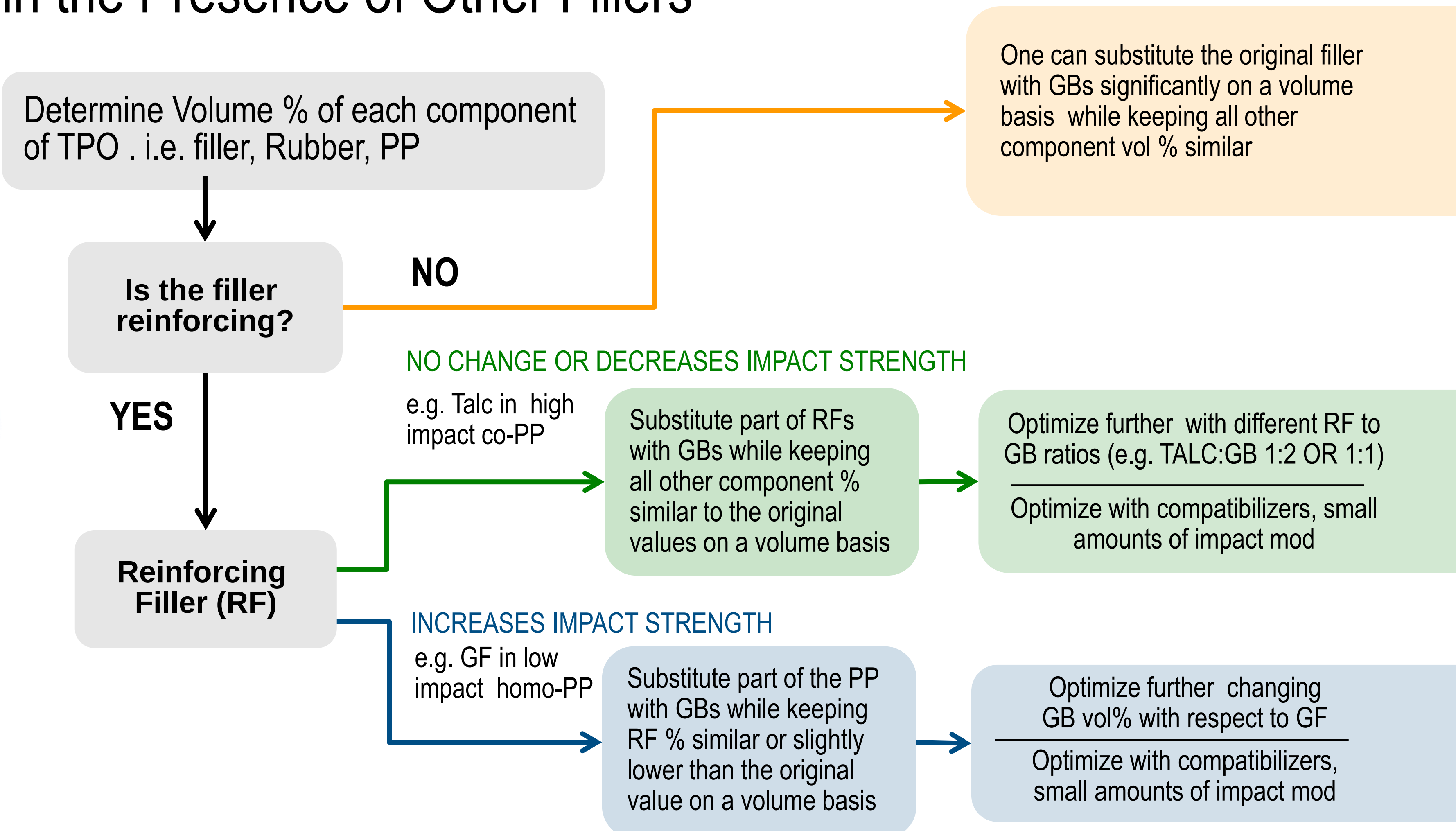
Glass Bubble Formulation Strategy in the Presence of Other Fillers



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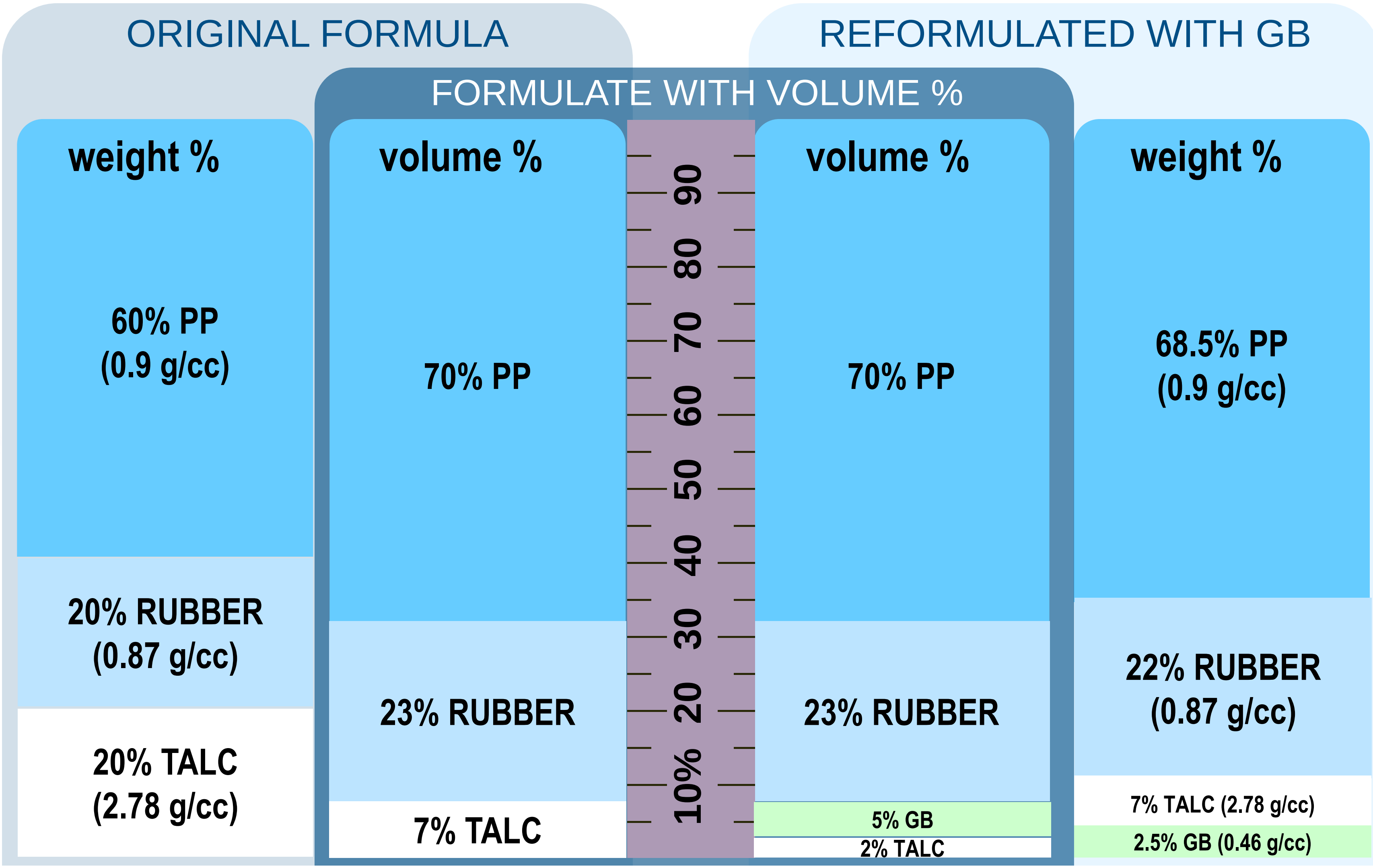
Formulation Scheme in the Presence of Talc



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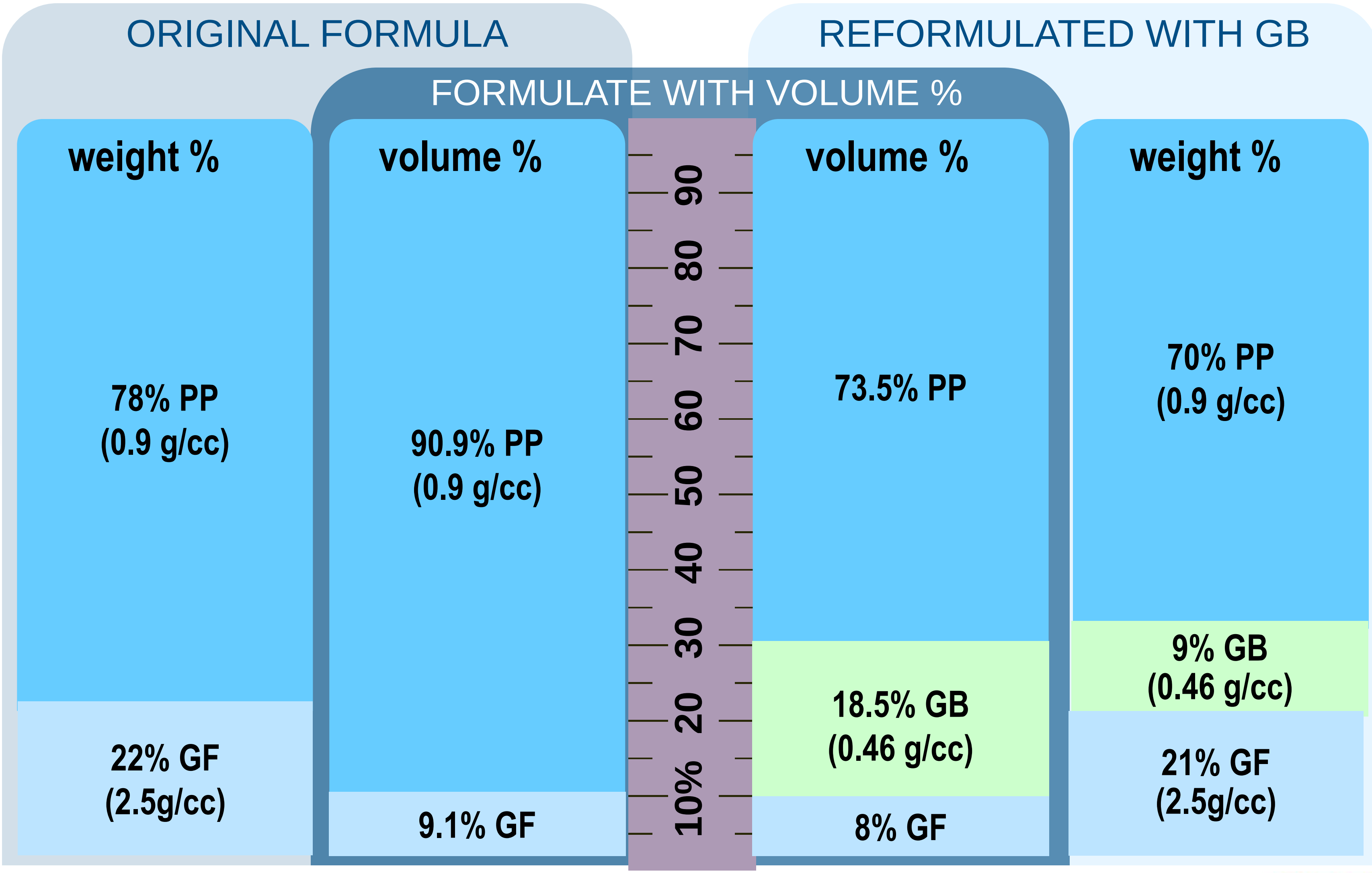
Formulation Scheme in the Presence of GF in a Low Impact PP



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Technical Data

Hollow Glass Microspheres

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Component	Formula 1		Formula 2		Formula 3		Formula 4		Formula 5	
	PP		PP-GB10		PP-GB10-mapp		PP-GB20		PP-GB20-mapp	
	Wt%	**Vol%**	**Wt%**	**Vol%**	**Wt%**	**Vol%**	**Wt%**	**Vol%**	**Wt%**	**Vol%**
PP homopolymer	100	100	90	83	87	80.60	81	69	76	65.84
GB-iM16K			10	17	10	16.65	19	31	20	30.78
Mapp compatibilizer					3	2.75			4	3.38
Final	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**
Density	0.9		0.835		0.835		0.785		0.785	
Tensile Strength @ RT (Mpa)	30.2		22.3		29.8		16.2		31.0	
Tensile Strength @ 90°C(Mpa)	10.1		8.6		11.6		7.7		13.5	
Tensile Modulus @RT (Mpa)	1195		1670		1513		1974		1830	
Tensile Modulus @ 90°C (Mpa)	162		230		230		340		300	
Flexural Strength (Mpa)	37.9		36.6		44.3		32.5		49.2	
Flexural Modulus @1 % secant (Mpa)	1063.		1410		1486		1585		1740	
Izod impact Strength at RT (J/m)	38.5		24.2		34.8		20.9		30.3	

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Impact Strength in Unfilled PP Copolymer

ASTM D256 Izod Pendulum impact strength –Test Method A- Notched (kj/m^2)	As Received Unfilled	**Part with IM16K**	**Part with iM16K**
Density g/cc	0.9	**0.86**	**0.83**
Borealis Standard PP (Homopolymer H503)	4.54	**4.16**	**3.36**
Borealis Copolymer (CP284)	19.16	**7.8**	5.4

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Homopolymer PP based TPOs Containing Talc

Partial Replacement

Component	Formula 1		Formula 2		Formula 3		Formula 4	
	PP-T20		PP-T10 GB4		PP-T10 GB4 -mapp		PP-GB20-mapp	
	Wt%	**Vol%**	**Wt%**	**Vol%**	**Wt%**	**Vol%**	**Wt%**	**Vol%**
PP homopolymer	80	92.5	86	88.6	83	85.5	77	65.84
GB-iM16K			4	8	4	8	20	30.78
Talc	20	7.5	10	3.4	10	3.4		
Mapp compatibilizer					3	3.1	4	3.38
Final	**100**	**100**	**100**	**100**	**100**	**100**	**100**	**100**
Density	1.046		0.942		0.943		0.785	
Tensile Strength @ RT (MPa)	31.7		27.0		32.7		31.0	
Tensile Strength @ 90 ºC (MPa)	12.5		11.4		13.5		13.5	
Tensile Elongation (%)	10		40		12		6	
Tensile Modulus@ RT (MPa)	2110		1900		1835		1830	
Tensile Modulus@ 90 ºC (MPa)	270		265		250		300	
Flexural Strength (MPa)	49		45		50		49.2	
Flexural Modulus @1 % secant (MPa)	1650		1620		1620		1740	
Izod impact Strength at RT (J/m)	32		28		39		30.3	

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High Impact Ductile TPOs Containing Talc

Partial Replacement

Component	Formula 1		Formula 2		Formula 3	
	Wt%	Vol%	Wt%	Vol%	Wt%	Vol%
High Impact PP containing rubber	100	100	80	92.74	87.68	91.92
Talc			20	7.26	9.41	3.20
iM16K-GB					2.91	4.88
Final	**100**	**100**	**100**	**100**	**100**	**100**
Density	0.877		1.021		0.926	
Tensile Strength (Mpa)	17.2		18.8		16.8	
Flexural Strength (Mpa)	26.9		33.1		29.7	
Flexural Modulus (Mpa)	915		1585		1310	
Izod impact Strength at RT (J/m)	725		645		430	

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PP Containing Glass Fibers

Component	Formula 1		Formula 2	
	Standard Injection Molding Control		Standard Injection Molding PP/GF/GB	
	Wt%	**Vol%**	**Wt%**	**Vol%**
HC- PP	78	91	68.2	70.14
GF	22	9	19.2	7.12
iM16K-GB			9.96	20.06
Compatibilizer			2.64	2.68
Final	**100**	**100**	**100**	**100**
Density	1.046		0.927	
Tensile Modulus (Mpa)	4050		3960	
Flexural Strength (Mpa)	100		94	
Flexural Modulus @1 % secant (Mpa)	2606		3020	
Izod impact Strength at RT (J/m)	54.5		51.8	

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Nylon 66 with 30wt.% Glass Fiber

Comparison of Glass Bubble Containing Systems

Component.	Formula 1		Formula 2		Formula 3	
	PA 6,6 Base Resin + 30w% Glass Fiber		PA 6,6 Base Resin + 30w% Glass Fiber + 5w% iM16K		PA 6,6 Base Resin + 30w% Glass Fiber + 10w% iM16K	
	Wt%	vol%	Wt%	vol%	Wt%	vol%
PA 6,6	70	83.9	65	71.4	60	60.8
Glass Fiber	30	16.1	30	14.7	30	13.6
iM30K-GB						
iM16K-GB			5	13.9	10	25.6
GB/GF VOL RATIO			0.94		1.9	
Final %	100		100	100	100	100
Vol.% Glass	16.1		28.6		39.2	
Density	1.37		1.25		1.15	
Tensile Strength (MPa)	191.4		176.9		164.1	
Tensile Elong. (%)	6.6		6.3		5.4	
Tensile Modulus (MPa)	4454		4434		4595	
Flexural Modulus (MPa)	7470		7598		8044	
RT Izod impact Strength (kJ/m2)	8.9		8.6		7.7	

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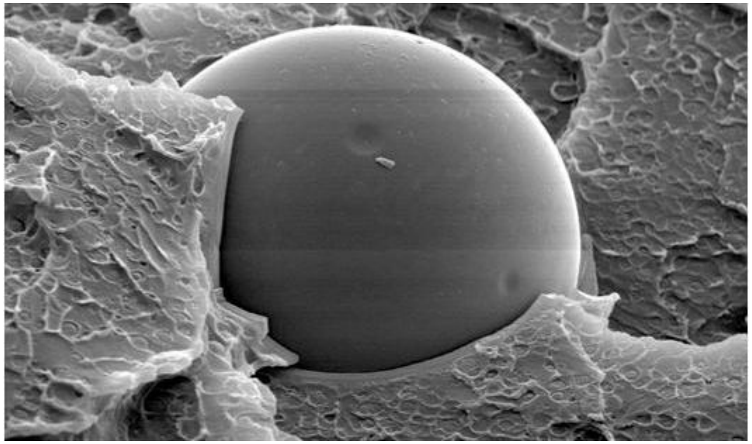
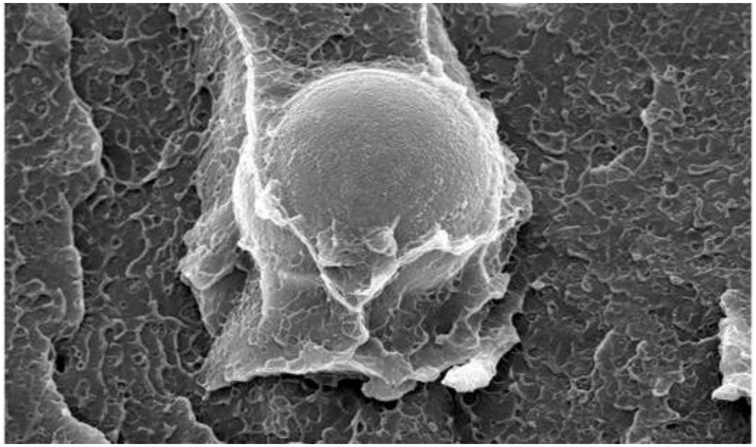
Silane Treated GBs

FOR FURTHER COMPATIBILITY WITH VARIOUS RESINS, SILANE TREATED GLASS BUBBLES ARE AVAILABLE



Untreated Bubble



Treated Bubble

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Polymer Processing with 3M™ Glass Bubbles

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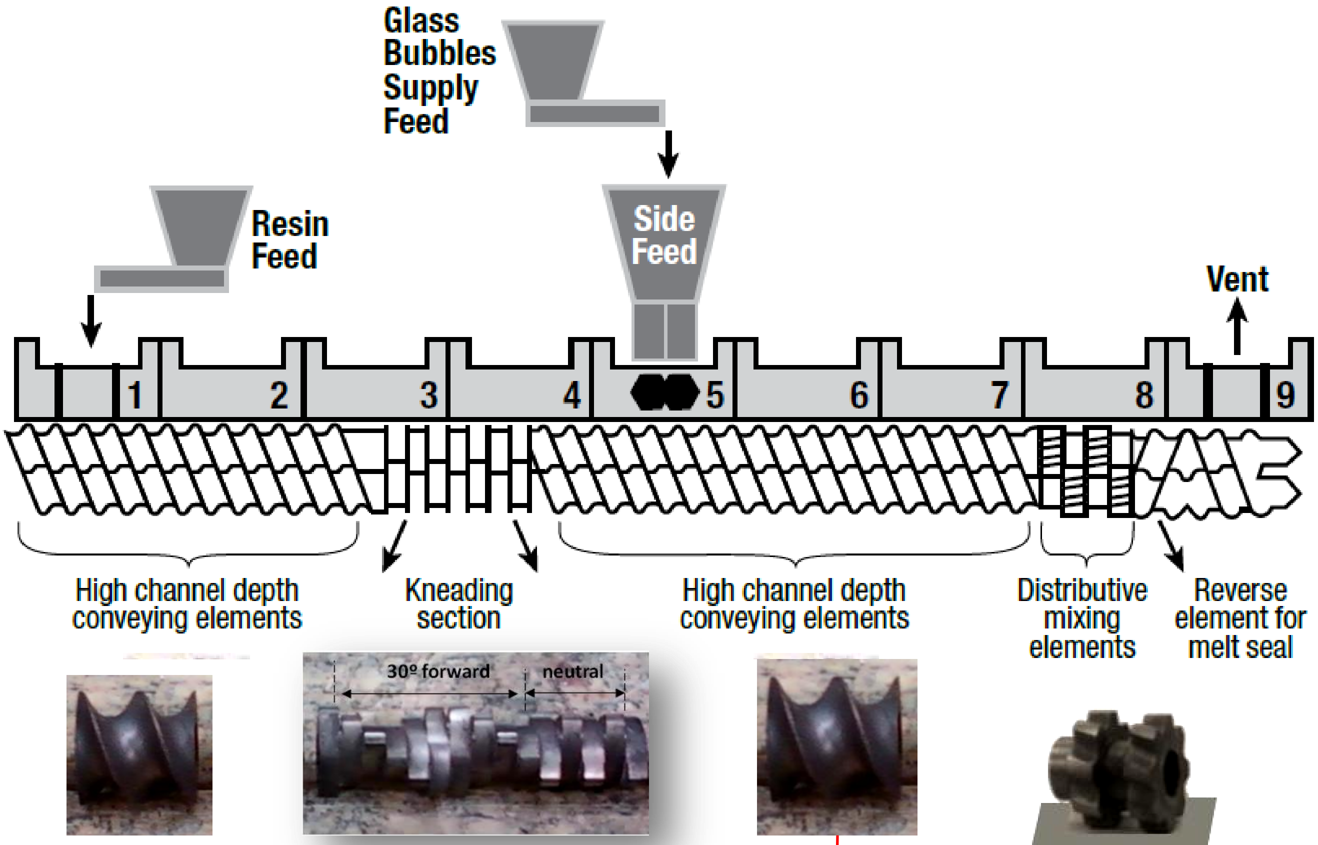
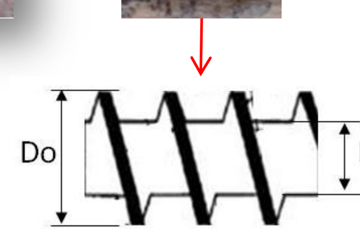
Incorporation of 3M™ Glass Bubbles via Twin Screw Extrusion



- Add Glass Bubbles downstream into fully molten polymer
- After addition use high channel depth conveying elements



Do/Di= 1.75 to 1.9

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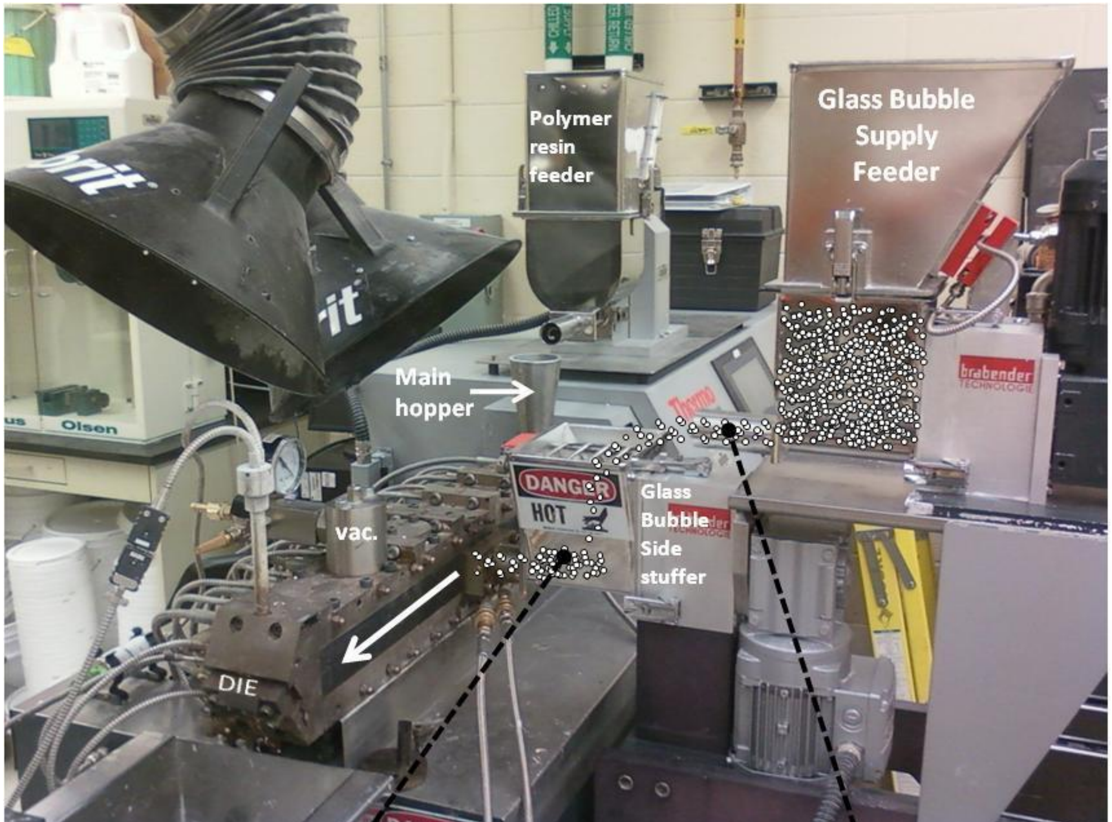

Screw of side stuffer

Screw of supply feeder

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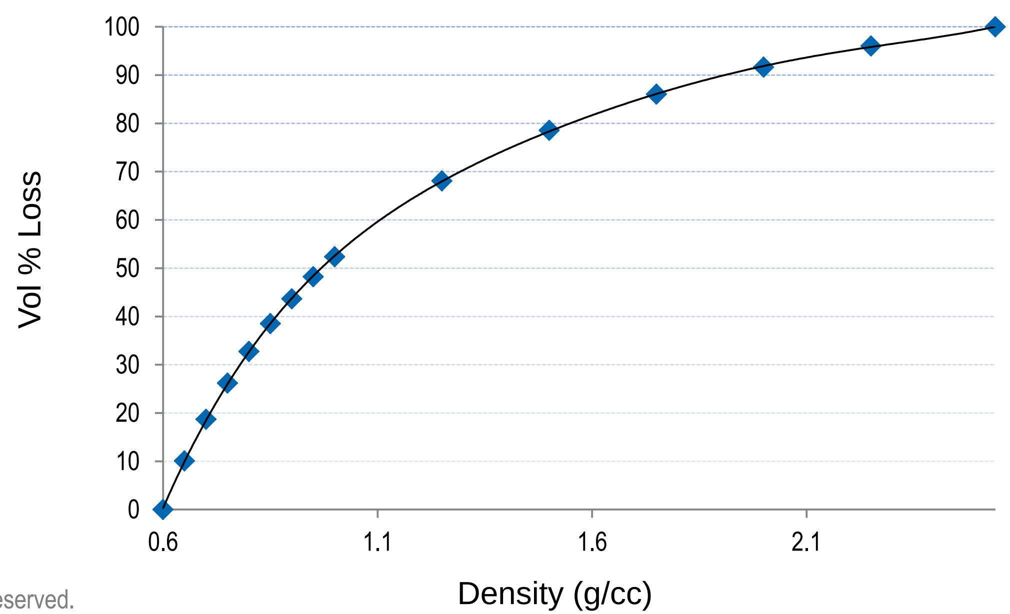
Determining Glass Bubble Concentration and Survival Rate

$$\text{Weight\% of GBs} = \frac{\text{Mass of residual inorganics after burn}}{\text{Mass of compounded material before burn}} \times 100$$

$$\text{\% Volume Loss} = \frac{\left(\frac{1}{\text{Initial Density of GB}} - \frac{1}{\text{Density of Solid Glass}}\right) - \left(\frac{1}{\text{Measured Density of GB}} - \frac{1}{\text{Density of Solid Glass}}\right)}{\frac{1}{\text{Initial Density of GB}} - \frac{1}{\text{Density of Solid Glass}}} \times 100$$



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Parameters that are Influential in GB Survival During Compounding

- Pelletizing
- Polymer Viscosity
- HGM Loading –Fill Ratio
- Back Pressure
- Channel Depth in the screw channels

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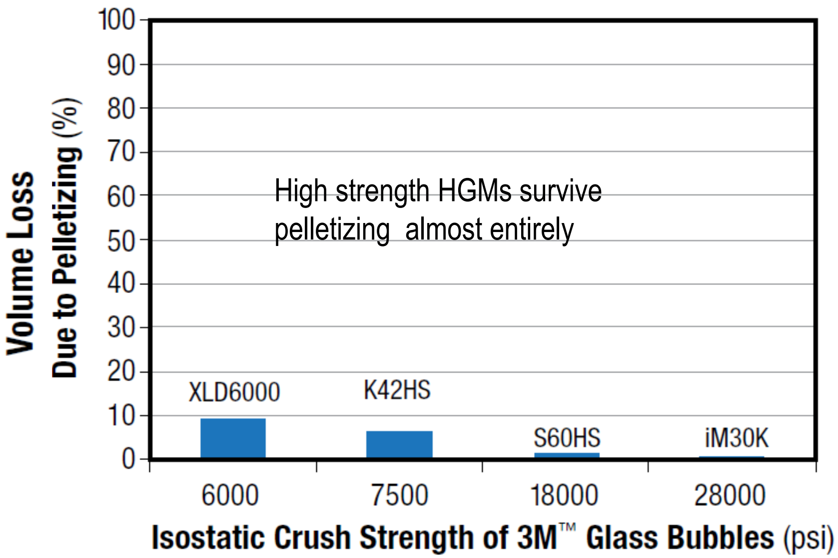
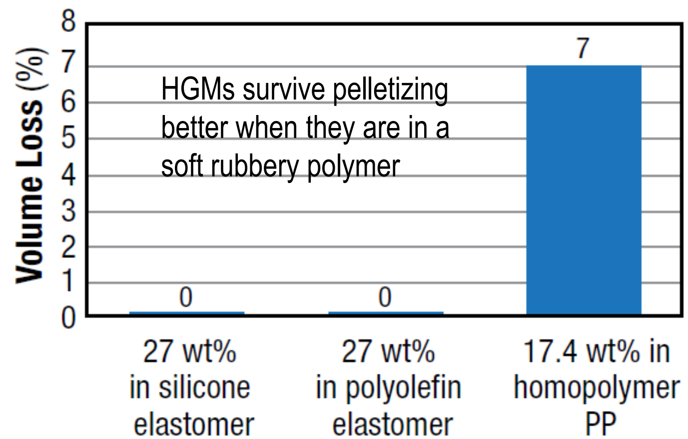
Parameters that are influential in HGM Survival During Compounding -Effect of Pelletizing



% HGM volume loss due to pelletizing as a function of isostatic crush strength - in homopolymer polypropylene with an MFI of 4 g/10 min at 230ºC, 2.16 kg



% Glass bubble volume loss due to pelletizing as a function of resin system used. In elastomeric soft resins, glass bubble survival is higher during pelletizing (glass bubble with 6000 psi crush strength, 0.3 g/cc)

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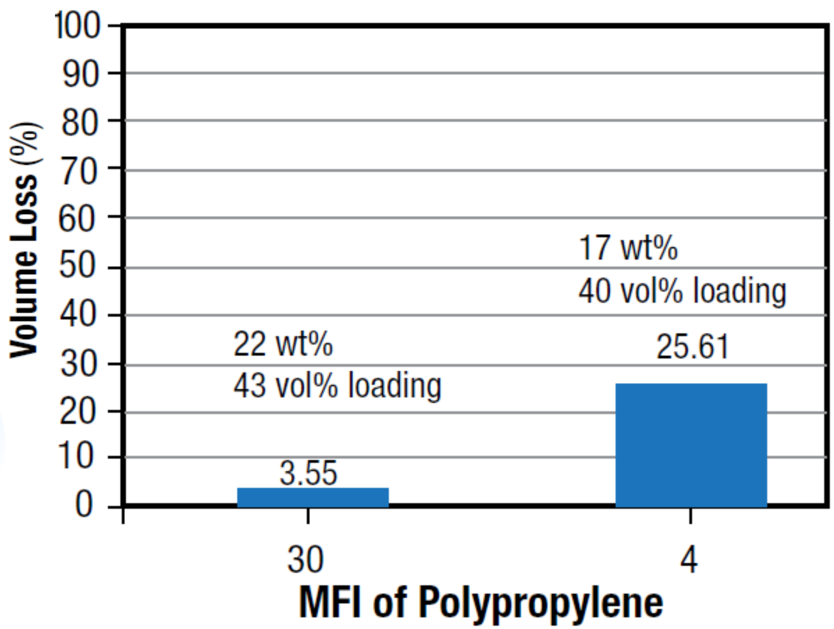
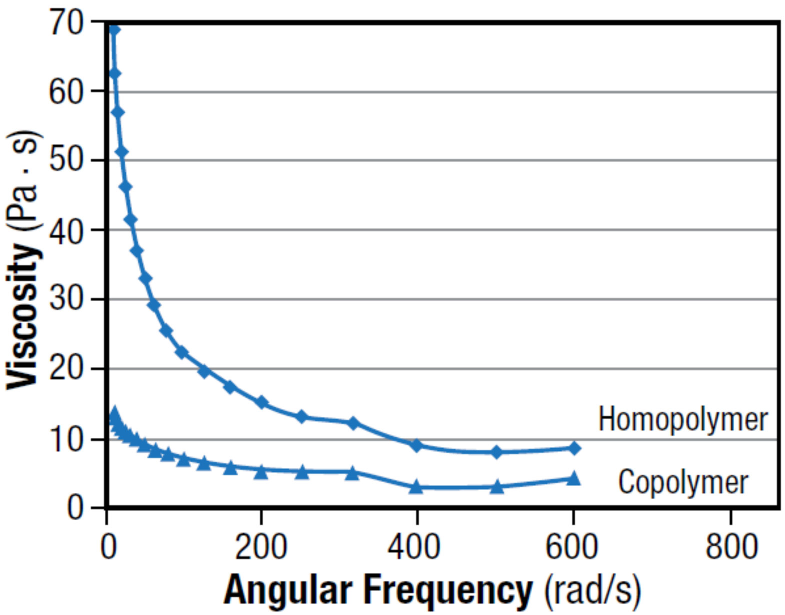
Parameters that are influential in HGM Survival During Compounding -Effect of Polymer Viscosity





HGM(6000 psi collapse strength) % volume loss in polypropylene as a function of melt flow index (MFI). LyondellBasell Pro-fax™ 6523. MFI (230ºC/2.16 kg): 4 g/10 min and LyondellBasell Pro-fax™ SG899 MFI (230ºC/2.16 kg): 30 g/10 min.

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Parameters that are influential in HGM Survival During Compounding- Effect of HGM Concentration

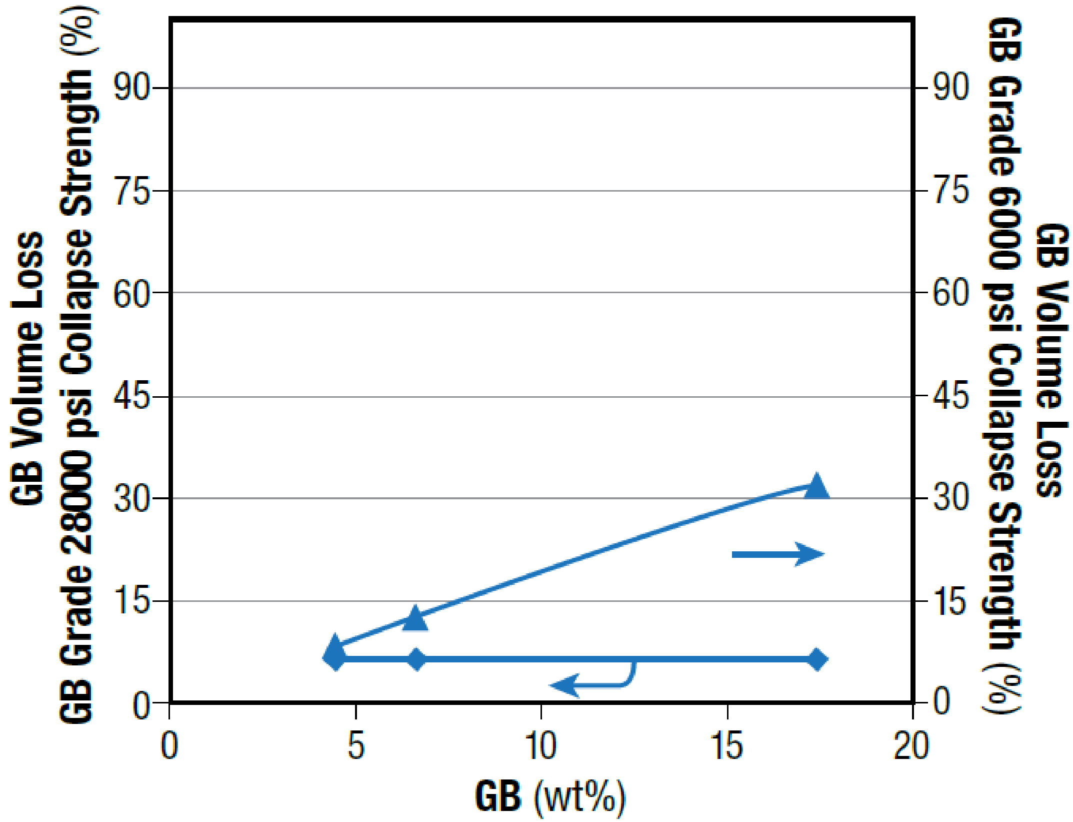

Figure 7. GB % volume loss as a function of loading in LyondellBasell Pro-fax™ 6523, MFI 4 g/10 min, homopolymer PP

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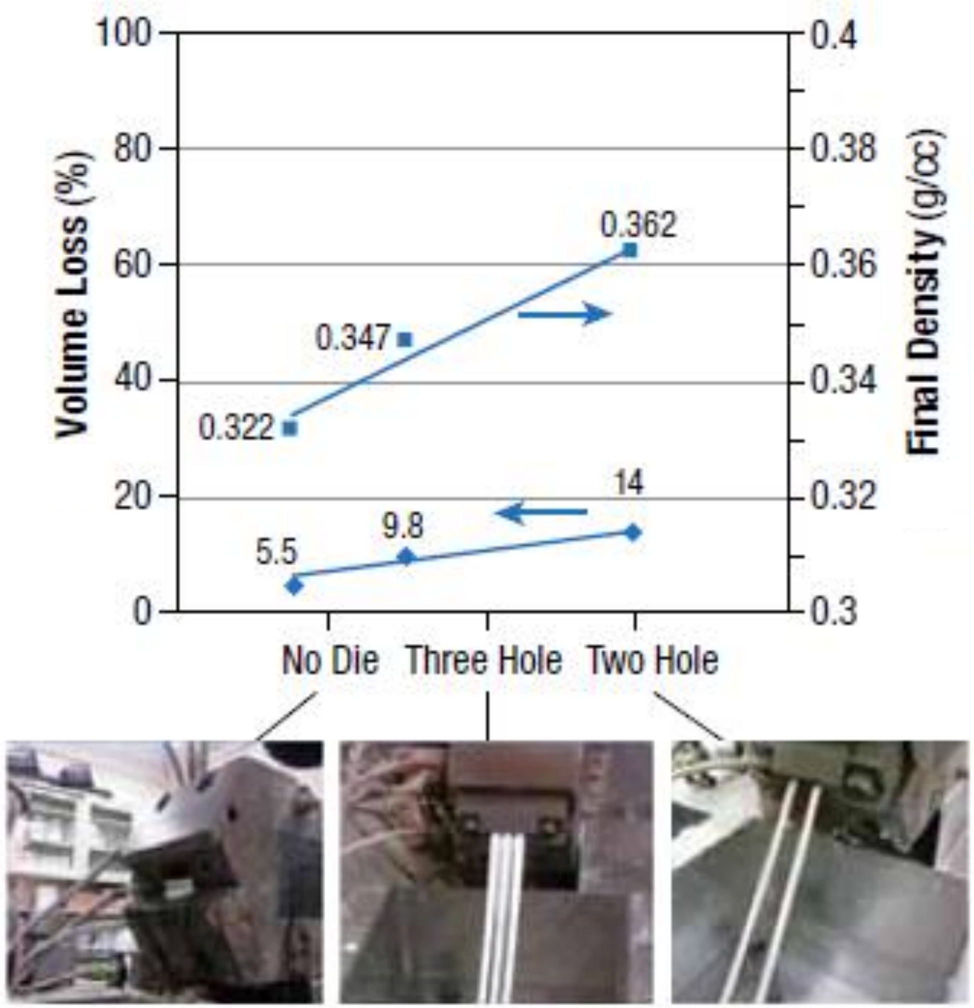
Parameters that are influential in HGM Survival During Compounding Effect of Back Pressure



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Summary of Important Things to Consider During Compounding

1. Twin screw co-rotating intermeshing extruders
2. Add HGMs into an already molten polymer at a downstream port via a side or top feeder (side feeder is preferred).
3. A side feeder should be fed via a supply feeder. This will ensure starve feeding of bubbles into the polymer melt and allow various volume % loadings to be prepared. If the bubbles are flood fed into the hopper of a side feeder, clogging and bridging may occur.
4. Inlet design of the side feeder into the extruder is very important, especially if high volume percentages of glass bubbles are formulated. The screw elements in the inlet section should be of the conveying type with a very high OD/ID ratio, such as 1.75 or more.
5. Pre-heating of glass bubbles, although not mandatory, could help prevent rapid temperature decrease of the polymer melt, which could cause rapid increase in viscosity.
6. After the glass bubbles are added into the molten polymer, they should be conveyed via standard conveying screw elements for a while before entering distributive block sections (if any need to be used).
7. Minimal back pressure is preferred during compounding with glass bubbles. In this respect, a die design that creates low back pressure is important. Likewise, screens with too large mesh sizes should be avoided.
8. An underwater pelletizer is the preferred method of pelletizing
9. If possible, resin parameters should be considered to prevent breakage – lower viscosity, higher MFI resins are preferred as well as materials that are softer and more elastic.

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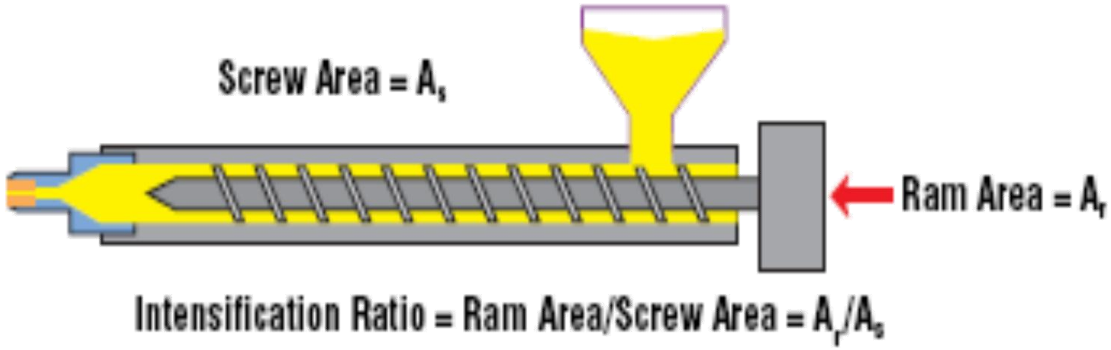
Injection Molding Considerations



- Maintain Low Back pressure
- Set low screw rotation speed (RPM) and injection rate
- Maintain injection pressure x intensification ratio < Isostatic Crush Strength
- General-purpose injection screw

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Benefits of Hollow Glass Microspheres

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The Power to Do More

- Reduced density
- Increased flexural and tensile modulus
- Since they are isotropic with an aspect ratio of 1 (minimal surface area) and do not orient themselves or cause orientation to the polymer molecules, injection molded parts have uniform shrinkage leading to **improved warpage and dimensional stability**
- Since they take up resin volume and in some cases provide nucleation sites for crystalline polymers, **increased productivity** can be achieved for injection molded or thick extruded profiles due to **fast cooling cycles**.
- Decreased CLTEs, decreased thermal conductivity
- Increased bulk, compressive modulus, increased hardness and scratch resistance, increased HDT

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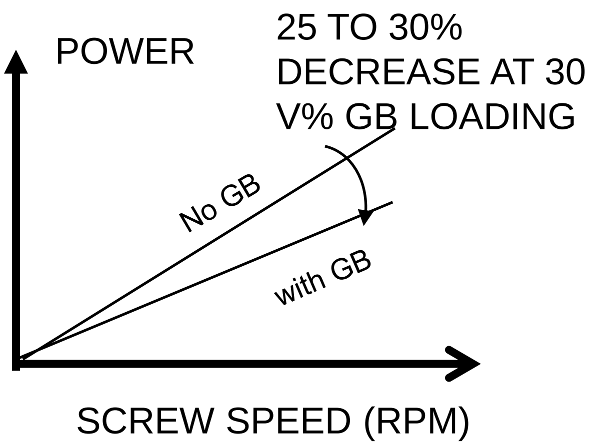
Reduced Power Requirement During Compounding

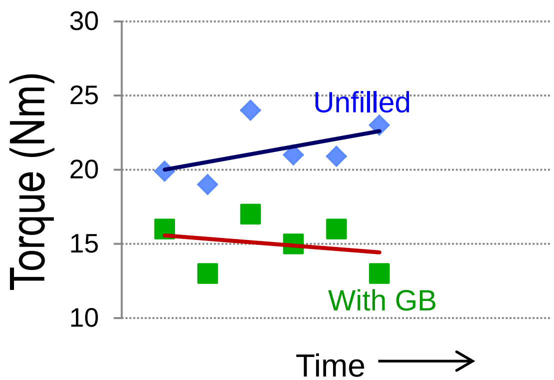
POWER = TORQUE X 2π SCREW SPEED (RPM)

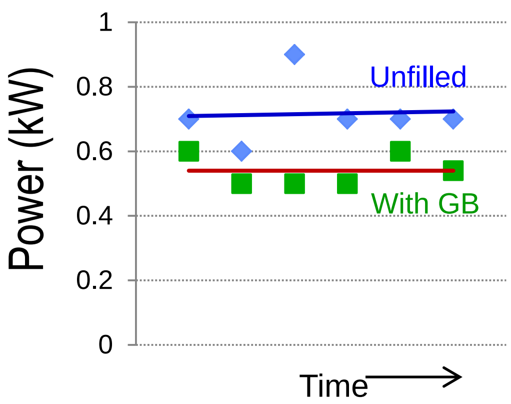
Achieve higher volumetric throughput with glass bubbles or reduce torque and hence power requirements for a given volumetric throughput

EXAMPLE : HOMOPOLYMER PP WITH AND WITHOUT GB AT 30 VOL%



24 MM & L/D 28:1
(PRISM TSE 24 MC)
Measurements were taken at ~ 20 % torque level for unfilled resin





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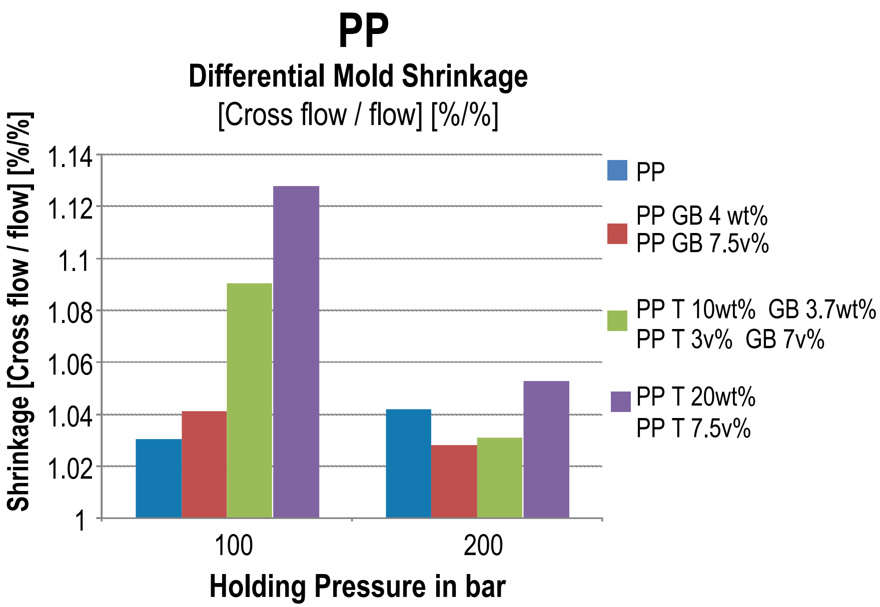
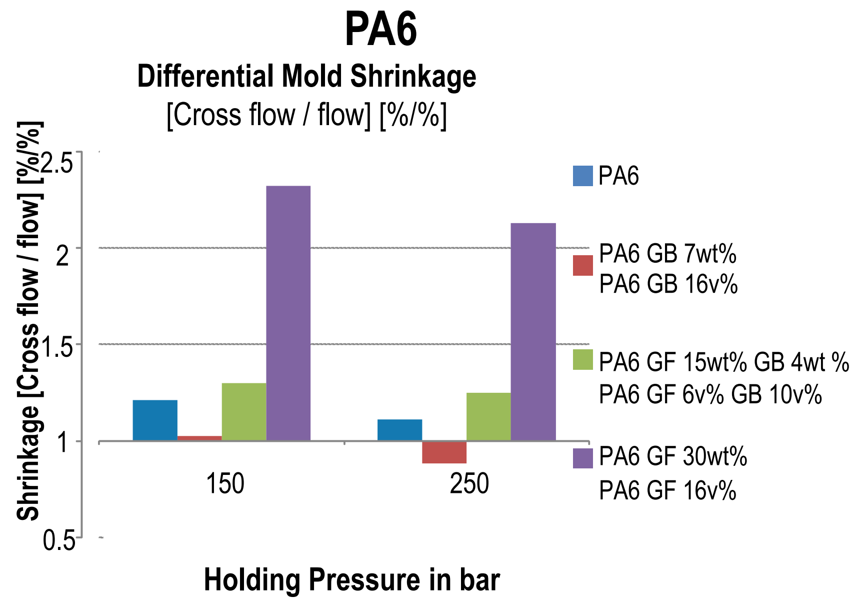
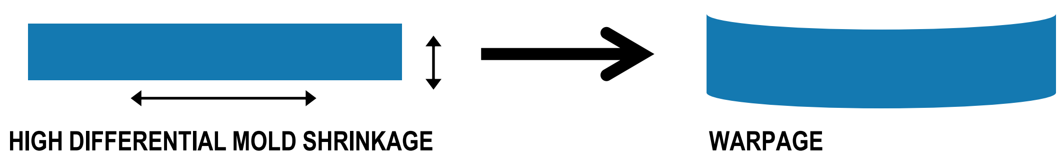
Dimensional Stability in PP and PA6

Independent Study by SKZ Institute, Germany
According to DIN EN ISO 294-4





Improve dimensional stability of molded parts by decreasing differential mold shrinkage with 3M™ Glass Bubbles iM16K



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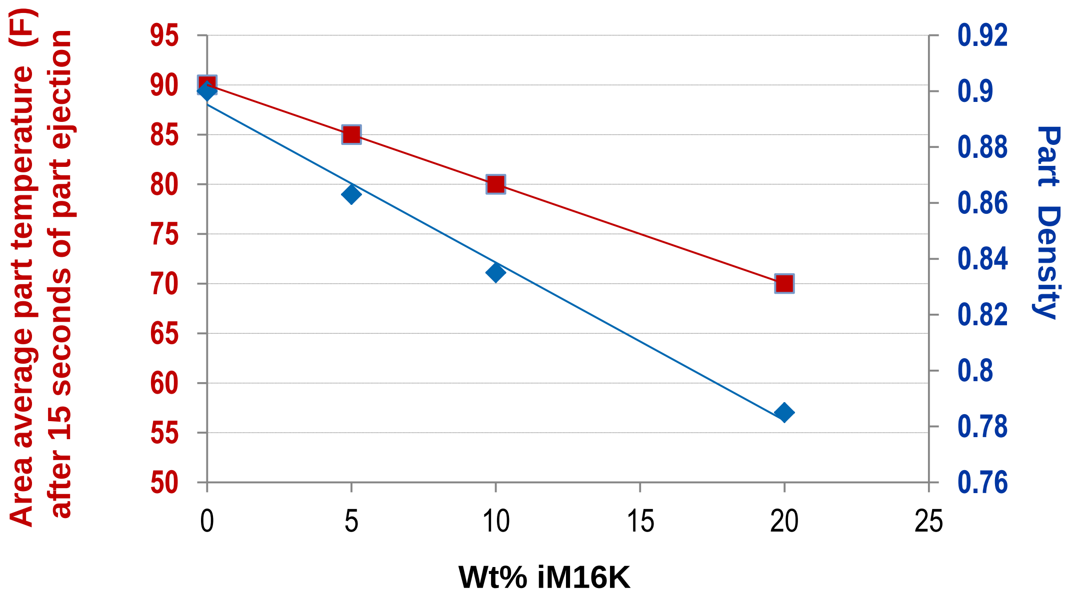
Effect of Glass Bubbles on Injection Molding Part Cooling Rate

Injection mold temp: 135 F
Holding Pressure: 3000 Psi
Mold: ASTM Mold
Base Polymer : PP Homopolymer



Trivial

$\alpha = k / \rho(Cp_{glass}$ wt fraction glass+ Cp_{void} wt fraction void $+Cp_{resin}$ wt fraction Resin)

Where α is thermal diffusivity, k thermal conductivity, ρ density and Cp is the specific heat

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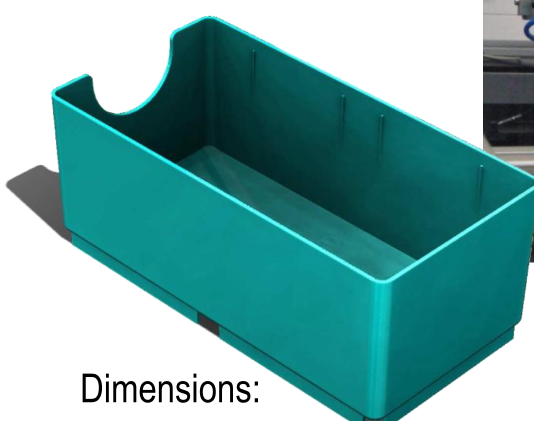
Inj. Molding Cycle Time Reduction in PA6 Containing iM16K

Independent Study by SKZ Institute, Germany

Material	Total Cycle Time t_G [s]	Cycle Time Reduction in [%]
PA6	40.2	**–**
PA6 GB–16 v%	35.2	**12**
PA6 GF 15–6 v% GB–10 v%	38.2	**5**

Part Molded



Dimensions:
60 x 60 x 2 mm

Experimental Setup:
Ejection Temp measured by IR

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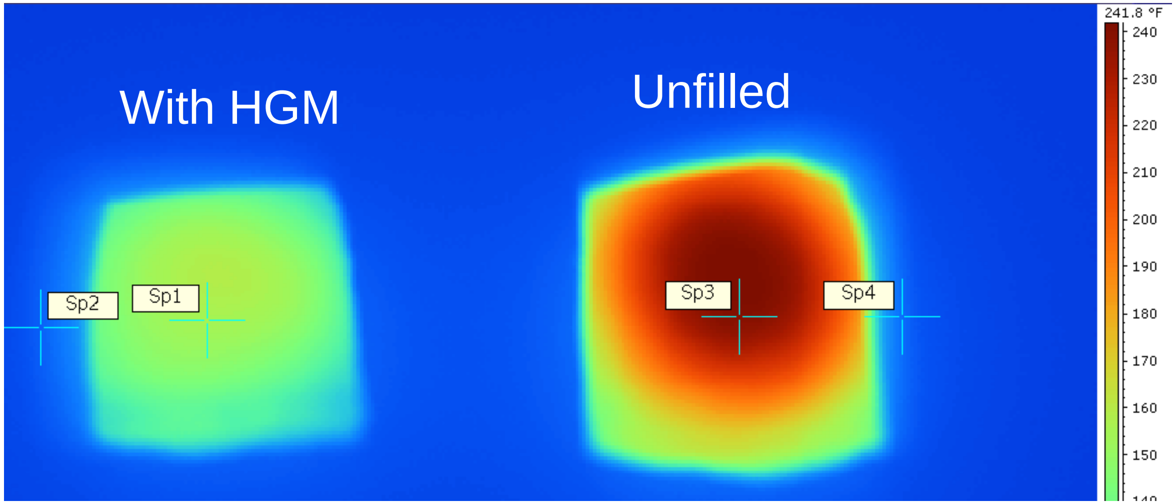
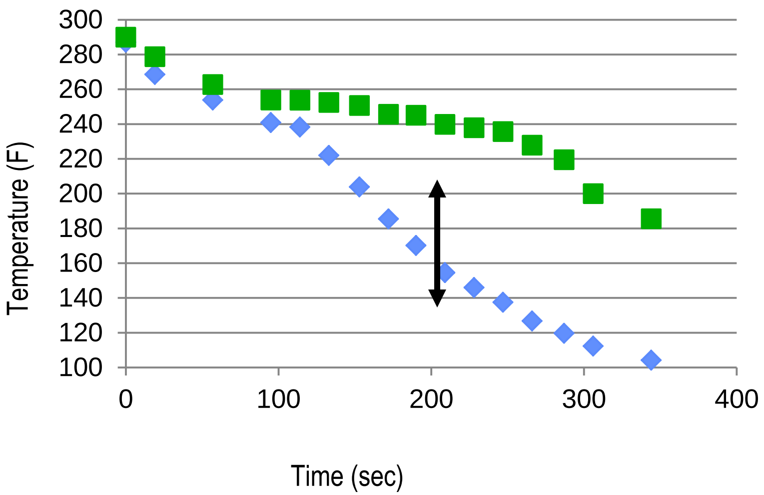
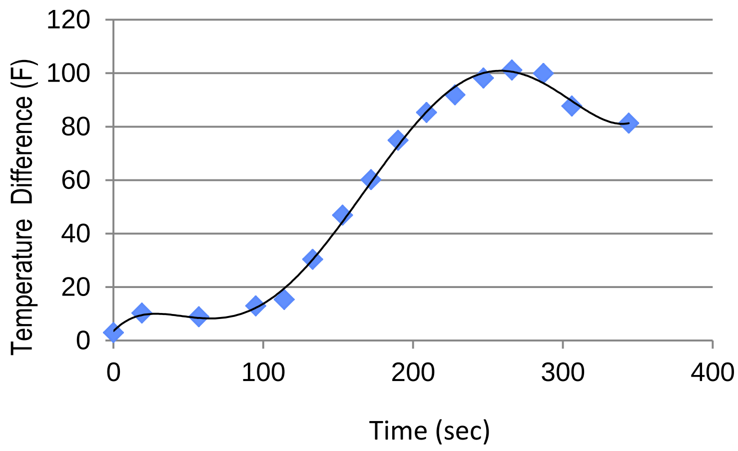
Effects of HGMs on Cooling from the Melt







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Application Examples

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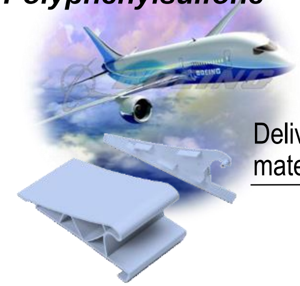
DO MORE for Customers

Polyetherimide, Polyphenylsulfone



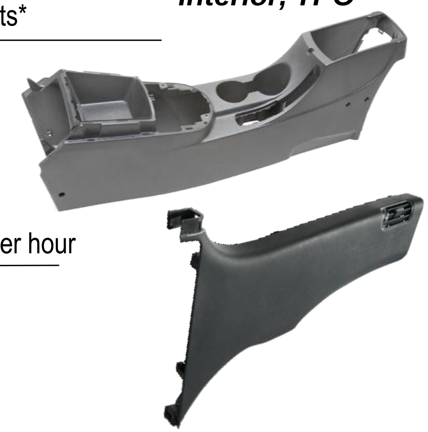
BMC headlamp

Flexible PVC baseboard

- **Problem Solving** — Lightweighting, warpage, shrinkage
- **Innovation** — Delivering new and unique materials solutions
- **Collaboration** — Global technical expertise
- **Online** — Online training, shop 3M.com, webinars, YouTube videos, thought leadership papers
- **Sustainability** — Neutral impact on carbon footprint of finished plastic parts*
- **For Less Money** — Reduction of total systems costs in plastics and shipping costs for parts
- **Production** — Make more parts per hour

PP Buoyancy Modules

Automotive Interior, TPO



PVC Boots

Butyl Rubber (IIR) Mountain hiking shoes

*A summary is available of the results of a single site cradle-to-gate carbon footprint study conducted by 3M in which polymers commonly used in transportation applications were evaluated.

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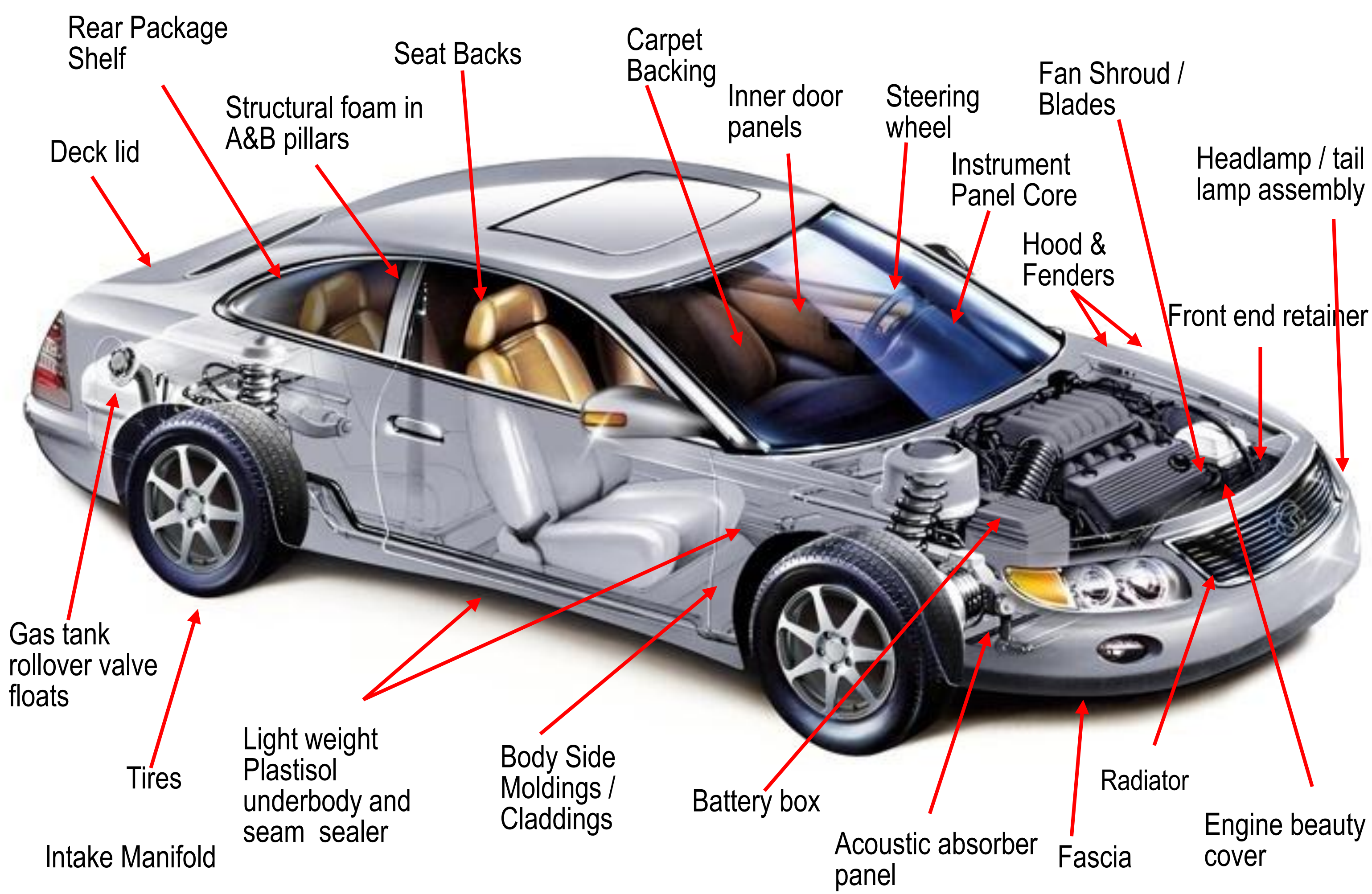
Hollow Glass Microspheres - Current & Potential Automotive Applications



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Examples of Commercial Glass Bubbles Applied Parts

MAGNA
EXTERIORS & INTERIORS

HANIL E-HWA CO., LTD

- 12–30% weight savings
- Reduced warpage & shrinkage
- Improved throughput by 15%
- Reduced cooling times
- Helping automotive OEMs meet greenhouse gas reduction targets

Photos provided by Magna, Hyundai Engineering Plastics and Visteon with customer claims as portrayed at NPE 2012.
Photo provided by Hanil e-Hwa with customer claims in 2010.

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Injection Molded Parts

Materials used:

Talc Filled PC/ABS Deck Lid

Enabling Features:

- Reduced weight
  - Control – 5.5 lbs
  - 7.5 wt. % GBs – 5.0 lbs
- Class A Paint-able Surface
- Same Process Conditions

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Fuel Floats

Materials used:

- Nylon 6,6

Enabling Features:

- Reduced weight
- 33 Wt. % S60HS
- Final density of 0.90 g/cc
- Allowed use of existing tooling
- Material specification

The following ASTM callout has been developed for GB1430-N :

ASTM D 4000 PA0120KD044PM018UM034YI095Z01Z02Z03Z04

Callout	ISO Method	Unit	Value
PA0120 (heat stabilized nylon 66)			
KD044 (tensile strength)	ISO 527	Mpa	44 min.
PM018 (Izod impact)	ISO 180	kJ/m2	1.8 min.
UM034 (flexural modulus)	ISO 178	Mpa	3400 min.
YI095 (HDT @ 1.8 Mpa. flat)	ISO 75	deg. C	95 min.
Z01= (filler hollow glass bubbles)			
Z02= (ash)	ISO 3451	%	33 +/- 4
Z03= (density)	ISO 1183	g/cm3	.90 +/- .02
Z04= (viscosity)	ISO 307	%	1.45-1.85

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Extruded Profiles

Materials Used:

- PVC or Filled PVC

Enabling Features:

- Increased throughput due to lower density/reduced cooling time (10 – 15%)
- Reduced weight
- Reduced warpage
- Improved feeding
- Meets flammability requirements

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REHAU®

Unlimited Polymer Solutions

- 5kg weight savings per aircraft
- 1000 liters fuel savings/year
- Reduction in carbon emissions

Low-density polymer systems specifically for the aircraft industry have been developed using 3M™ Glass Bubbles in high temperature high performance polymers such as Polyether imide (PEI) and polyphenylsulfone (PPSU).

Photo provided by REHAU as portrayed at NPE 2012.

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Photo provided by Kohshin Rubber Co., Ltd. as portrayed at NPE 2012.

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Glass Bubbles in Shoe Soles

- FEATURE
  - KOLON SPORT ™ FEATHER
  - 3M™ Performance Additives iM30K incorporated
  - Floats in the water
  - Work with research institute (KIFLT)

- ADVANTAGE
  - Weight reduced (20%)
  - Control 1.18g/cc → under 1.0g/cc at 11% iM30K
  - Improved dimensional stability
  - Water & chemical resistant

- BENEFIT
  - Less weight and comfort
  - Less fatigue

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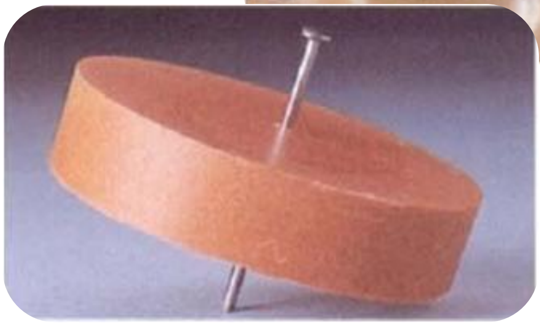
Polymer Wood Composites

Materials used:

- Virgin and Recycled HDPE, PVC

Enabling Features:

- Reduced Weight (5 – 15%)
- Reduced Cooling Time
- Improved Nail-ability
- Reduced Flammability



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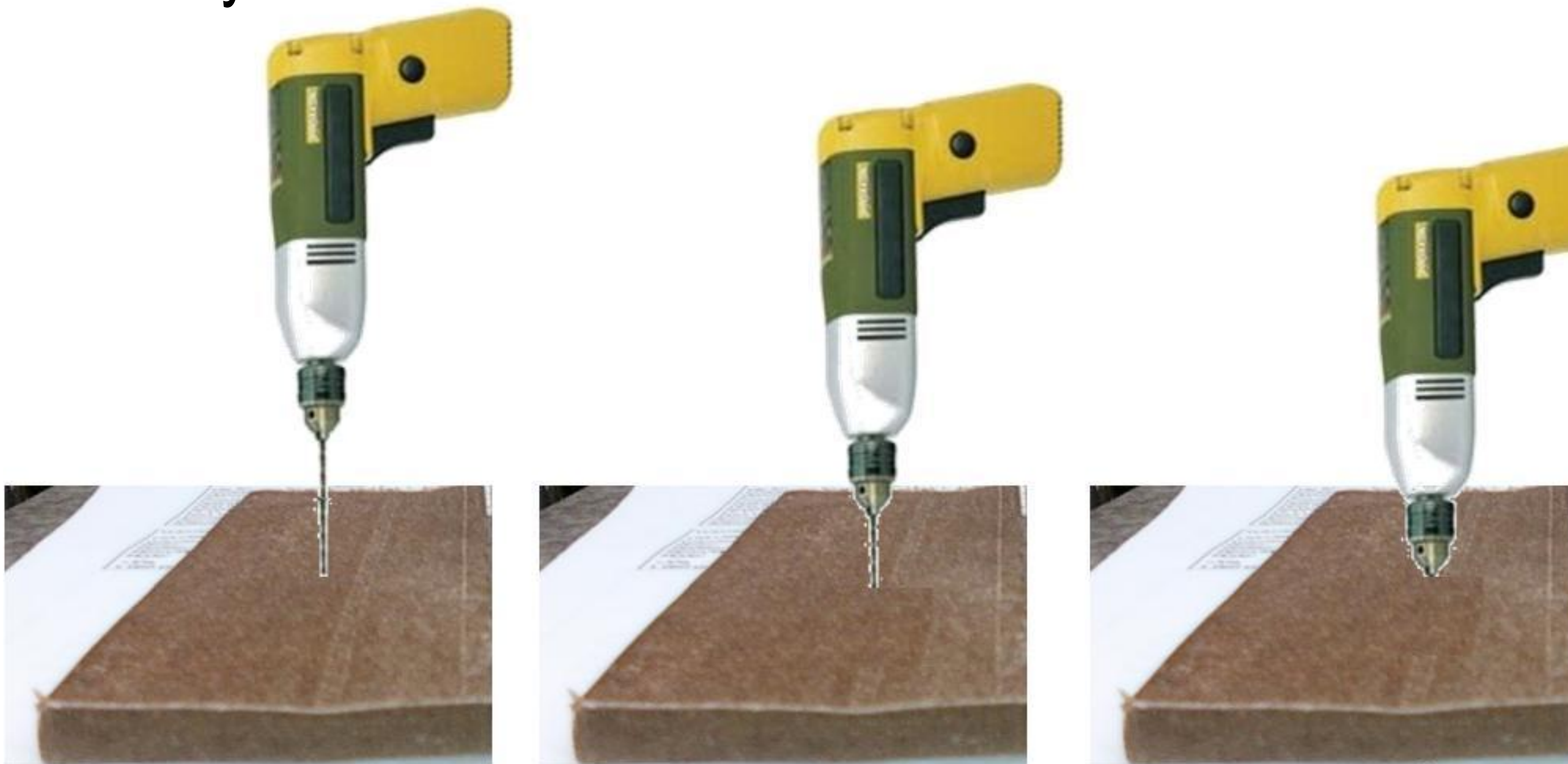
Drillability



Pressure at the wood-drill bit contact point by dividing the weight of the drill by the cross sectional area of the ¼ " drill bit. 1800 grams drill weight applies a pressure of ~80 psi at the contact point.

75 SECONDS VS 20 SECONDS WITH GLASS BUBBLES (18 wt% GB)

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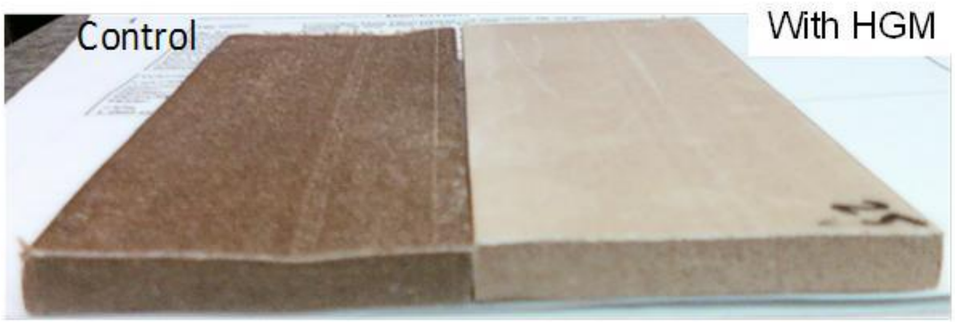
Improved Dimensional Stability



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Reduced Differential Weighting

Materials used:

- Polypropylene

Enabling Features:

- Reduced/differential weighting
- Improved buoyancy
- Even wear
- Improved material flowability in mold
- Allowed use of existing tooling

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Pressure-resistant thermal insulation and buoyancy

Materials used:

- PP, PU, Epoxy, Silicon

Enabling Features:

- Reduced weight
- Density change
- Thermal conductivity
- High loading of GBs

Glass Bubble	W/m K	Btu in/h ft2 F
K1	0.047	0.327
K15	0.055	0.38
K20	0.07	0.486
S22	0.076	0.529
K25	0.085	0.593
S32	0.108	0.746
S35	0.117	0.813
K37	0.124	0.858
S38/ S38HS/ S38XHS	0.127	0.88
K46	0.153	1.062
S60/ S60HS	0.2	1.39

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Conclusion

Hollow Glass Microspheres can reduce density of materials while providing significant processing benefits with improved properties.

Smart formulation is key in attaining an acceptable balance of weight and final properties.

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Thank you!

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