Impact of Microcellular Plastics on Industrial Practice and Academic Research

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Summary: Microcellular plastics (MCP) refer to any plastic with tiny bubbles of less than about 50 microns. It is made by subjecting polymers with a large amount of dissolved gas to a thermodynamic instability so as to nucleate a large number of cells instantaneously. MCP has been extruded and injection molded to make various industrial products. The fundamental theory for design of MCP and the processing methods are reviewed. It also discusses the design of equipment, including the die or mold, for MCP processing. The performance of MCP and the advantages of using MCP are presented. Some of industrial applications are also highlighted.

Keywords: cell density, microcellular plastics, MuCell, nucleation of cells, polymer processing

Introduction

Microcellular plastics (MCPs), which were first invented at the Massachusetts Institute of Technology in 1979,[1] refer to any polymeric materials that have closed cells of very small diameters, typically smaller than 50 microns. The cell density can be made to vary a great deal depending on the final application of a given microcellular plastic. MCPs can have as high as 10^{15} bubbles/cm^3 when the bubble diameter is 0.1 microns, 10^{12} bubbles/cm^3 for 1 micron, and 10^9 for 10 micron diameter cells. They can be created in thermoplastics, thermosetting plastics, and elastomers.

The original impetus for the invention of MCPs was to create a plastic consuming less material without sacrificing mechanical properties, especially toughness. The saving of material was achieved by creating voids and toughness was a result of making the diameter of the bubble smaller than a critical size. The central idea was to replace some of the polymers with a large number of very small bubbles that are smaller than the pre-existing flaws in polymers. Small bubbles can blunt the crack-tips and act as crazing initiation sites, this making the material tougher.

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The basic processing method for all microcellular plastics is the use of thermodynamic instability phenomena. A large amount of gas, typically CO_2 or N_2, is dissolved the in plastics under high pressure at the processing temperature so as to create a driving force for phase separation when the pressure is suddenly lowered.

Depending on the magnitude of the driving force, various nucleation sites are activated. The number of nucleation sites increases nearly exponentially with the amount of gas dissolved, when the polymer is super-saturated with the dissolved gas – relative to its equilibrium concentration at the pressure of one bar and the operating temperature. Micro-cells form for the following reasons: the amount of gas dissolved must be shared equally by an extremely large number of nucleated sites, since the cells nucleate nearly simultaneously, preventing the preferential diffusion of the gas to the sites that have nucleated first. Because the driving force is so large, homogeneous nucleation dominates even when there are second-phase particles that would be the preferred heterogeneous nucleation sites because of its low activation energy.

Microcellular plastics have unique processing characteristics. The processing temperature is substantially less than the conventional processes because the viscosity of plastics is substantially reduced due to the presence of gas between polymeric molecules. The throughput rate of a given extruder can be also greater because of the low viscosity. The cycle time of injection molding machines is also reduced because the processing temperature is lower and the phase separation of gas from polymer instantaneously increases the rigidity of plastics. Furthermore, there is no shrinkage of the injection-molded part because it is compensated by the internal expansion in the microcells, creating parts with minimal residual stress and warpage. Sometimes, depending on the color of the plastic and the smoothness of the molded surface, swirl marks may appear, which can be hidden through painting or texturing.

Certain properties of microcellular plastics, such as modulus and strength, follow the rule of mixture, whereas such properties as toughness and coefficient of thermal expansion do not. When the cell size is less than a few microns, the toughness of certain microcellular plastics should be equal to or better than the plastic without the cells. Small cells also lower the thermal conductivity when they are smaller than a critical size.

Many industrial firms worldwide are now making microcellular products through extrusion and injection molding.[2] It is very likely that the number of new applications that use the microcellular technology will continue to increase at a rapid rate in the years to come.

The field of microcellular plastic technology is in some ways in the early stages of research and development, notwithstanding its relatively long history. It has raised many interesting scientific and technological issues that can be the basis for thought provoking ideas and research. Many academic institutions worldwide are conducting their research in the field of microcellular plastics, which should further generate new ideas and applications. Many industrial firms are developing new applications for injection molding and extrusion processes.

Design of Microcellular Plastics and Processing Techniques

Review of the Design of Microcellular Plastics

Microcellular plastics were designed to satisfy the following three functional requirements (FRs) based on axiomatic design:[1,3] FR1 = Reduce plastics consumption, FR2 = Maintain the toughness of plastics, and FR3 = Make three dimensional parts.

To satisfy these FRs, the concept of microcellular plastics was created by envisioning plastics with tiny bubbles.[1] Then the design parameters (DPs) of microcellular plastics are the following: DP1 = Total volume of cells (i.e., bubbles), V; DP2 = Diameter of cells, d; DP3 = Die or Mold.

The design equation that relates the FRs to the DPs of microcellular plastics may be written as

$$\begin{Bmatrix} FR1 \\ FR2 \\ FR3 \end{Bmatrix} = \begin{bmatrix} X & X & 0 \\ 0 & X & 0 \\ 0 & 0 & X \end{bmatrix} \begin{Bmatrix} DP1 \\ DP2 \\ DP3 \end{Bmatrix} \qquad (1)$$

Equation (1) indicates that the design of an MCP is a decoupled design. It indicates that the bubble size must be determined first before setting the total volume of the bubbles.

In an ideal microcellular plastic, where spherical bubbles are packed in body- centered cubic structure, the bubble size can be directly related to the bubble density. In a 1-cm cube of foamed material, the number of cells is inversely proportional to the cube of the bubble diameter. Therefore, a microcellular plastic with 10-mm bubbles has approximately 10^9 bubbles per cm^3 of unfoamed material, whereas microcellular plastics with 1-μm and 0.1-μm size

bubbles have approximately 10^{12} and 10^{15} bubbles per cm^3 of unfoamed material, respectively. Since the volume taken by spherical bubbles in an ideal, closely packed hexagonal or cubic structure is approximately 74%, the plastic occupying the interstitial space is 26%. Therefore, the cell density of an ideal closely packed spherical microcellular plastic is equal to (1/cell size)^3 times (1/0.26). For a microcellular plastic with 1-μm cell diameter, the bubble density is 3.85×10^{12} cells/cm^3 of the solid plastic. The overall density of foam can decrease further when these cells expand, thinning the wall diameter and reducing the interstitial materials between the cells.

Dissolution of Gases in Polymers

The basic physics involved are as follows:[4]

(1) The plastic must be supersaturated with sufficient gas such as N_2 and $CO_2$to nucleate a large number of cells simultaneously.

(2) The temperature of the plastic must be set so as to control the flow of plastics during processing.

(3) A gas with a suitable solubility and diffusivity for the plastic must be selected.

(4) Homogeneous nucleation must dominate the nucleation process to create a large number of microcells even when heterogeneous nucleation sites are available by providing sufficient driving force with a sufficient amount of dissolved gas.

The processing technique consists of forming a polymer/gas solution and then suddenly inducing a thermodynamic instability by either lowering the pressure or raising the temperature to change the solubility *S*. The solubility of gas in polymers decreases with an increase in temperature. The solubility of N_2 is considerably less than that of CO_2. Since the amount of gas that can be dissolved is a function of the saturation pressure and since the gas diffusion rate is the rate-limiting process, we can use supercritical CO_2 to enhance the solubility and diffusion rate. CO_2 is supercritical at pressures and temperatures greater than 7.4 MPa and 31.1°C.

With dissolution of a large number of gas molecules in polymers, the glass transition temperature and viscosity decrease with the increase in gas concentration. The change in the glass transition temperature is quite substantial at high gas concentrations. These changes affect

the processibility of polymers.

To decrease the solubility and induce the thermodynamic instability, either the pressure must be decreased (i.e., $\Delta p<0$) or the temperature must be increased (i.e., $\Delta T>0$). Furthermore, regardless of whether the process is continuous or batch type, the thermodynamic instability must be induced quickly so that the cells will nucleate simultaneously before significant diffusion of gas has taken place. Therefore, the higher the temperature of the polymer, the quicker nucleation has to occur since the diffusion of the gas occurs faster at higher temperatures. Such simultaneous cell nucleation will assure a uniform cell size distribution. The following two dimensionless numbers must be less than one for this to happen:

$$\frac{\text{Characteristic nucleation time}}{\text{Characteristic diffusion time}} \propto \frac{\alpha}{\frac{dN}{dt} d_c} << 1 \qquad (2)$$

$$\frac{\text{Characteristic gas diffusion distance}}{\text{Characteristic spacing between stable nuclei}} \propto 2\rho_c^{1/3}(\alpha\, t_D)^{1/2} << 1 \qquad (3)$$

The number of cells nucleated is a function of the supersaturation level relative to the equilibrium concentration at ambient pressure at the processing temperature. The higher the supersaturation level, the greater is the number of cells nucleated. Furthermore, since the amount of dissolved gas that fills the nucleated cells is finite, and since all the cells are nucleated almost simultaneously, the gas distributes more or less evenly among all these cells -- a condition for making microcellular plastics. The final bubble size is then determined by the total gas per bubble, and by the flow characteristics of the polymer at the nucleation temperature.

To create a continuous process, we designed processes and associated equipment to perform the following functions in extrusion and injection molding: (1) Rapid dissolution of gas into molten flowing polymer to form a polymer/gas solution; (2) Nucleation of a large number of cells; (3) Control of the cell size; and (4) Control of the geometry of the final product.

To produce the microcellular plastics at an acceptable production rate through a continuous process, we must dissolve the gas in polymers quickly despite the slow diffusion rate. The

diffusivity increases with temperature by an Arrhenius relationship. The time for gas diffusion is proportional to the thickness of the plastic ℓ as $t \propto \frac{\ell^2}{\alpha}$. The diffusivity of CO_2 and N_2 are nearly the same and it takes a long time to diffuse gas into a polymer at room temperature. For example, the diffusivity of CO_2 in most thermoplastics at room temperature is in the range of $5x10^{-8}$ cm^2/s and the diffusion time is approximately 14 hours when ℓ is 0.5 mm. The diffusivity at 200°C is 3 to 4 orders of magnitude greater than that at room temperature. Even at high temperatures, the diffusion rate is still the rate-limiting step in continuous processes.

To accelerate the diffusion rate and shorten the time for the formation of gas/polymer solutions, we must raise the temperature and shorten the diffusion distance. This is done by deforming the two-phase mixture of polymer and gas through shear distortion to decrease the diffusion path. This type of deformation occurs in an extruder under laminar flow conditions. The bubbles are stretched by the shear field of the two-phase mixture and eventually break up to minimize the surface energy when a critical Weber number is reached.[5] The disintegrated bubble size is calculated to be about 1 mm and the initial striation thickness after bubble disintegration is calculated to be about twice the bubble diameter.[6] This striation thickness decreases with further shear, and the gas diffusion occurs faster as a result of the increase in the surface area and the decrease in striation thickness. The striation thickness in an extruder is estimated to decrease to about 100 μm. At this thickness, the diffusion time is in the range of 1 minute in PET, from 10 to 20 seconds in polystyrene (PS), polyvinylchloride (PVC), and high density polyethylene (HDPE), and in the range of a few seconds in low density polyethylene (LDPE).

Nucleation

The key idea in the formation of an MCP is the nucleation of an extremely large number of bubbles (cells). Although cells can nucleate either homogeneously or heterogeneously, the driving force is so high due to such a large amount of supersaturation of the gas in the polymer that the both homogeneous and heterogeneous nucleation sites are expected to be nucleated. This can be seen from micrographs, which show that cells are nucleated both at and away from the heterogeneous sites.

For nucleation to occur, a finite energy barrier has to be overcome. The energy barrier depends on two competing factors: (a) the energy available in the gas diffused into the embryo of the

cell and (b) the surface energy that must be supplied to form the surface of the cell. There is a critical cell size beyond which the cell becomes stable and grows, and below which the cell embryo collapses. Typically the cell nucleation rate is expressed as:

$$\frac{dN}{dt} = N_0 f e^{\frac{-\Delta G}{kT}} \tag{4}$$

where N = the number of cells; N_0 = the number of available sites for nucleation; f = the frequency of atomic or molecular lattice vibration; ΔG = the activation energy barrier; k = the Boltzmann constant; T = the absolute temperature. A variety of different nucleation sites may be nucleated when the driving force is very large, the most prominent of which are the free volume sites. Table 1 shows the potential activation sites and the expected cell density when these sites become activated.

Table 1. Potential activation sites for cells and rough estimates of potential cell density.

Solid/polymer interface	10^5 to 10^6 cells/cc
Non-polar polymer/polar polymer interface	--
High strain region	10^9 cells/cc
Free volume	10^9 cells/cc
Crystalline/amorphous interface in a polymer	10^{12} cells/cc
Interface between crystallites	10^{18} cells/cc
Morphological defects in a polymer	--
Polar groups of polymers	10^{22} cells/cc

The activation energy associated with each one of these potential activation sites is expected to be substantially different, probably increasing with the available sites. The activation energy may be represented in terms of its probability density function as shown in Figure 1. The activation energy also changes when the gas is dissolved.

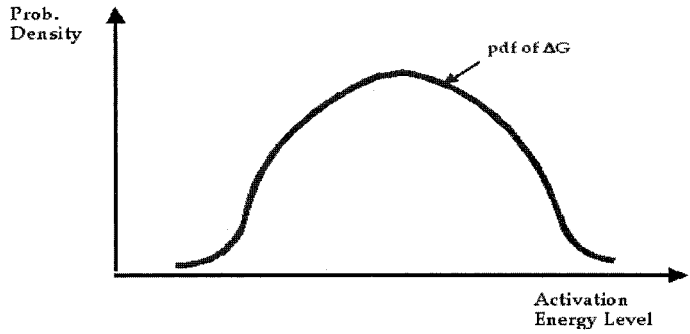

Fig. 1. Probability distribution of ΔG. Note as the amount of dissolved gas increases, the sites with high activation energy are expected to be activated.

The number of the available sites N_0 is also affected by the gas dissolved since the gas changes the intermolecular forces as indirectly evidenced by the change in the viscosity and melting point of the polymer/gas solution (see Figure 2). N_0 is a function of both the original activation energy ΔG and the amount of the gas dissolved. Although there is no data available, the N_0 is expected to increase with higher activation energy since it appears that there are more activation sites at these higher-level activation energies, which is represented schematically as shown in Figure 3.

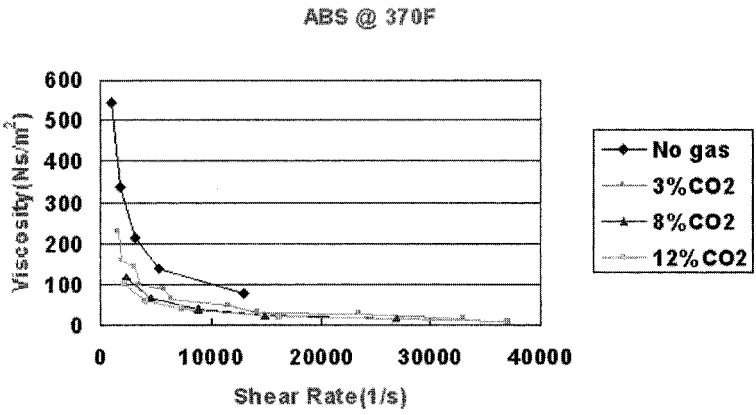

Fig. 2. Viscosity of ABS as a function of CO_2 concentration and shear rate at 370F (Courtesy of Trexel, Inc.). Note that the relative viscosity change is most pronounced at low shear rates.

Fig. 3. Number of available sites for cell nucleation as a function of the gas dissolved. It is conjectured that $(N_0)_{max}$ is greater as the activation energy increases.

Cell Growth

Immediately after the cells are nucleated, the pressure in the bubble is equal to the saturation pressure. Therefore, the cells expand if the polymer matrix is soft enough to undergo viscoelastic-plastic deformation. The cell expands until the final pressure inside the cell is equal to the pressure required to be in equilibrium with the surface forces and the stress in the viscoelastic cell wall. Unlike in conventional foaming, in the case of microcellular plastics, there are so many cells nucleated and the diffusion length is so short that the diffusion of the gas to the cell growth stops relatively quickly. In practice, the temperature of the surface of the extrudate changes as a result of heat transfer, and thus, the expansion of the cell is constrained by the outer stiff layer. Also some of the gases from the cells near the surface escape, reducing the tendency to expand.

Cell Density and Cell Size

The cell density is a function of both the pressure drop and the pressure drop rate. During the cell nucleation stage, there is a competition for gas between cell nucleation and cell growth if the cells do not nucleate simultaneously. When some cells nucleate before others, the gas in the solution will preferentially diffuse to the nucleated cells to lower the free energy of the system. As the gas diffuses to these cells, low gas concentration regions where nucleation cannot occur

are generated adjacent to the stable nuclei. As the solution pressure drops further, the system will either both nucleate additional microcells and expand the existing cells by gas diffusion or only expand the existing cells. Therefore, when the pressure drop occurs rapidly, the gas-depleted region where nucleation cannot occur will be smaller and a more uniform cell distribution will result. It has been determined experimentally that a drop rate of 2 GPa/second is the minimum pressure drop rate required for microcellular plastics processing. Figure 4 and show examples of the cell nucleation density as a function of the gas pressure.

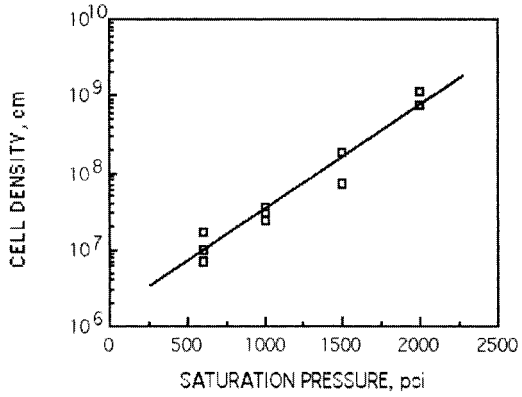

Fig. 4. Cell nucleation density as a function of N_2 pressure in polystyrene.[7]

Equipment and Die Design

The role of the extruder (or the plasticating unit of an injection-molding machine) is to melt the plastic, create a single phase polymer/gas solution, and pump the solution through a die or inject it into a mold. To achieve these functions, high-pressure CO_2 or N_2 gas is introduced into the extruder barrel by metering the exact amount of CO_2 or N_2 at pressures greater than 2,000 psi. The flow rate of CO_2 into the extruder can be controlled using a special metering pump. The gas forms a large bubble in the extruder since the flow of the gas is briefly interrupted whenever the screw flight wipes over the barrel. Then to diffuse the gas in the bubble quickly in the molten plastic, the polymer/gas interfacial area is increased and the striation thickness of polymers between the gas bubbles decreased. This is done by elongating the bubble in the

barrel through the shear deformation of the two-phase mixture of the polymer and gas. The approximate residency time required for diffusion and solution formation in the extruder is estimated to be as follows: less than 100 seconds for PET and less than 10 seconds for polystyrene at typical operating temperatures.

To design the process, the FRs are selected as: FR1 = Reduce the amount of plastic used; FR2 = Increase the toughness of the plastic product; FR3 = Make three-dimensional geometrical shape. The DPs are chosen as: DP1 = Microcellular plastics (uniform cell distribution in large numbers); DP2 = Diameter of microcells; DP3 = Die shape. The process variables (PVs) for the process described that can satisfy the DPs given are: PV1 = Supersaturation of the plastic with a large amount of gas and sudden pressure change (dp/dt); PV2 = Temperature of the molten polymer to control the expansion of cells at the die; PV3 = Cross-sectional dimensions.

The design equation for the extrusion process may be written as

$$\begin{Bmatrix} DP1 \\ DP2 \\ DP3 \end{Bmatrix} = \begin{bmatrix} X & 0 & 0 \\ X & X & 0 \\ 0 & 0 & X \end{bmatrix} \begin{Bmatrix} PV1 \\ PV2 \\ PV3 \end{Bmatrix} \tag{5}$$

Equation (5) shows that the process design is also a decoupled design. Therefore, each design satisfies the Independence Axiom.

DP1 and PV1 can be further decomposed as: DP11 = Large number of nucleated cells; DP12 = Uniform-size cells; PV11 = The level of supersaturation of CO_2; PV12 = Rapid pressure drop dp/dt. The design matrix for this design may be represented as

$$\begin{Bmatrix} DP11 \\ DP12 \end{Bmatrix} = \begin{bmatrix} X & x \\ X & X \end{bmatrix} \begin{Bmatrix} PV11 \\ PV12 \end{Bmatrix} \tag{6}$$

Equation (6) states that DP11 and DP12 are coupled slightly in that if the pressure drop rate is really slow, we cannot get a large number of cells and uniform-sized cells. In most cases, the effect of dp/dt on the number of cells is negligible.

The role of the plasticating section of the injection molding machine or the extruder is to melt the plastic and dissolve the gas in the polymer. The extruder must be under high pressure to

maintain a single-phase solution. The cell density is primarily controlled by the amount of the dissolved gas and also partly by the pressure drop rate.

The die must be designed to control the pressure drop rate, which controls the uniformity of cell size. The desired pressure drop rate is greater than 1 GP/s. It also removes the thermal energy from the molded part. The die also creates 3D shapes in the case of injection molding or the profile in the case of extrusion. It can be seen that the die design is as important as the extruder or the injection molding design.

The highest-level FRs and DPs are given as; FR1 = Control cell size; FR2 = Control the number of cells; FR3 = Control the geometry of the extrudate. DP1 = P_i*; DP2 = dp/dt; DP3 = Die shape & Accessories.

The design equation is given by

$$\begin{Bmatrix} \text{Cell size} \\ \text{Cell density} \\ \text{Geometry} \end{Bmatrix} = \begin{bmatrix} X & x & 0 \\ X & X & 0 \\ x & 0 & X \end{bmatrix} \begin{Bmatrix} P_i \\ dp/dt \\ Die\ \&\ Acc. \end{Bmatrix} \tag{7}$$

The corresponding PVs are chosen as: PV1 = Extruder RPM; PV2 = Die lip length; PV3 = Means of controlling the profile.

The design equation for the process is given by

$$\begin{Bmatrix} P_i \\ dp/dt \\ Die\ \&\ Acc. \end{Bmatrix} = \begin{bmatrix} X & x & 0 \\ X & X & 0 \\ 0 & 0 & X \end{bmatrix} \begin{Bmatrix} \Omega \\ L \\ \text{Profile} \end{Bmatrix} \tag{8}$$

Advantages of Injection Molding with Microcellular Plastics

The injection pressure of injection-molding process decreases due to the presence of dissolved gas, which lowers the viscosity. The cycle time is also reduced due to the elimination of the "hold and pack" time and also due to about 25% reduction in cooling time. Table 2 presents a comparison of the injection molding process with and without the dissolved gas.

Table 2. Comparison of injection-molding process for various products with and without microcellular structure (Courtesy of Mar Lee Companies).

Air Bag Canister (33% glass filled Nylon)		**Conventional**	**MCP**	**%**
	Part Weight	365 g	252 g	30.9
	Cycle Time	45 sec	35 sec	22.2
	Clamp.Tonnage	150 tons	15 tons	90
Connector (polycarbonate)	***Part Weight***	48.8 g	42.9 g	10.6
	Cycle Time	17.5 sec	15.9 sec	9.1
	Clamp. Tonnage	140 tons	20 tons	85.7
Battery cover (poypropylene)	***Part Weight***	201 g	159 g	20.8
	Cycle Time	60 sec	37 sec	38.3
	Clamp. Tonnage	200 tons	15 tons	92.5

Figure 5 shows an injection-molded printer chassis for inkjet printers made of glass filled engineering plastic (PPO/HIPS). The chassis made of microcellular plastics has 50 % less warpage, 25% reduction in cycle time, and 8% weight reduction. The microcellular plastic also had higher toughness -- 9.0 ft-lb vs 6.7 ft-lb by drop weight test, and 9.7 kJ/m^2 vs 7.3 kJ/m^2 by notched Izod impact test.

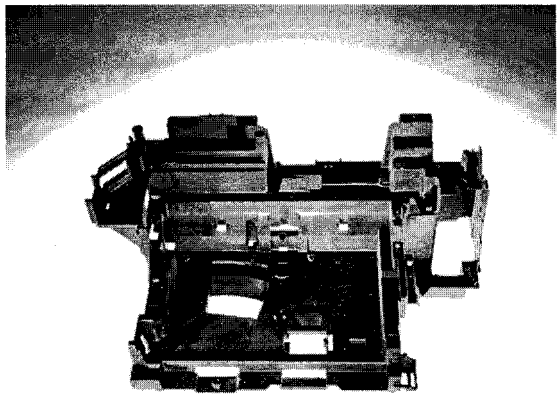

Fig. 5. Injection-molded Printer Chassis with Microcellular Plastics (Courtesy of Trexel, Inc.).

There are a large number of advantages of using the microcellular plastics: reduction of material consumption (between 5% and 30 %); faster cycle time; higher productivity; greater toughness in some plastics; low residual stress; dimensional accuracy; minimal residual stress, dimensional stability; reduction in warping of injection molded parts; appearance (no visible cells); thin sections; no sink marks; low temperature process; low pressure process; large number of cavities or smaller machines; most polymers; use of non-hydrocarbon solvents; no additives for nucleation; and no special equipment other than gas supply system.

Performance and Applications of Microcellular Plastics

Since the cell size is extremely small, the cells cannot be seen by the naked eye. Therefore, the foamed plastic resembles a solid plastic, having a good physical appearance. At conventional cell sizes, they are opaque without the need to introduce pigments such as titanium dioxide.

MCPs save money for manufacturers due to the use of less material and faster cycle time. Since about 70% of the cost of foamed plastic goods is the material cost and since up to 50% weight reduction is possible for some applications, the cost of plastic parts can be reduced by as much as 35%.

MCPs are environmentally acceptable since they are processed using carbon dioxide (CO_2) or nitrogen (N_2), instead of hydrocarbons or fluorinated materials. Since smaller amounts of plastics are used in a given product, there is less material to recycle or dispose. Furthermore, less raw material and energy are used to make the same plastic article.

MCPs find many applications in housing and construction, sporting goods, vehicles, electrical and electronic products, chemical and biochemical applications, and the textile and apparel industry. They can be used in siding, pipes, electrical wire, automotive seats and other parts, airplane parts, filters, shoe soles, office equipment housing, artificial paper, food containers, polishing cloth, thermal insulation around pipes, and other uses as well.

MCPs are processed at lower processing temperatures, since the glass-transition and melting temperatures and the viscosity of plastics decrease with the increase in dissolved gas. As the gas is formed in the bubble during the nucleation and cell growth phase, the viscosity and the melting temperature of plastics increases, reverting back to the original state. Therefore, MCPs

"solidify" much more quickly – by almost a factor of two – and therefore, injection-molded MCP parts can be taken out of the mold quickly.

Conclusions

Because of their advantages in performance, cost, and processing microcellular plastics show great promise in changing the polymer processing industry. A great deal is known about MCPs and their processing techniques, but the field is still full of research and development opportunities to satisfy the diverse requirements of the polymer processing field.

[1] See for detailed historical account and a review, Suh, N. P. "Microcellular Plastics," *Innovation in Polymer Processing: Molding*, (Ed. by J. Stevenson), SPE Books of Hanser Publishers, New York, **1996**. (ISBN 3-446-17433-8)

[2] Under the license from Trexel, Inc. Trexel, as the sole licensee of M.I.T., has developed the MIT technology further for commercial applications. The trade name is MuCell.

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