

Effects of the Die Geometry on the Expansion of Polystyrene Foams Blown with Carbon Dioxide

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ABSTRACT: This article presents the effects of the die geometry on the expansion ratio of extruded polystyrene foams blown with CO₂. Three groups of interchangeable filamentary dies were used to represent the die parameters. The experimental results reveal that a strong relationship exists between the expansion ratio of the extruded polystyrene

foams and the die geometry through its effects on the pressure-drop rate, the die pressure, the amount of premature cell growth, and the initial shape of the extrudate. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 3329–3336, 2008

Key words: foam extrusion; melt; nucleation; diffusion

INTRODUCTION

Most commercial styrenic foams are produced in an expansion process that occurs at the exit of a foaming die with a physical blowing agent dissolved or dispersed in the plasticated polymer matrix.¹ This foam expansion process typically comprises three fundamental steps: (1) nucleation of small discontinuities or cells, (2) growth of these cells to a desired volume, and (3) stabilization and retention of the expanded form obtained by physical or chemical means. All expansion foam fabrication processes are designed to promote and control the sequential development of each of these three steps.² Because volume expansion of polymeric foams takes place during the cell-growth stage, an established cell-growth control strategy is desirable to obtain a desired expansion ratio of polymeric foams.

Polystyrene (PS) has been widely recognized and demonstrated to be a favorable material for foaming because of its amorphous molecular structure; meanwhile, because of its desirable mechanical and thermally insulating properties, PS foams have been broadly used for insulation, packaging, and furniture.³ For example, a package made of PS foam can be used to hold hot or cold food; while keeping food warm or cold, it does not suffer from deformation or deterioration. In addition, the components of PS foams are recyclable. Over a million tons of this material are consumed per year, and the number tends to increase.⁴

With all their advantages, PS foams have intrinsic drawbacks: a lack of toughness and resilience. The brittleness of the product is inherent from the glassy base polymer. The lack of breakage resistance makes PS foams less suitable than polyolefin foams for cushion packaging, especially when protection against multiple impacts is needed. In addition, the rigid foams are not well suited for the insulation of curved surfaces.

To resolve these deficiencies of PS products, technologies need to be developed to amend the foam structure. This is because the properties of foams are basically determined by not only the kind of base polymer but also the cellular structure. The cellular structure is fundamentally determined by the cell density, the volume expansion ratio, the average cell size, and the cell size distribution of the foam. If the cell size is assumed to be uniform, only two of the first three parameters are independent. For example, for a fixed expansion ratio, the cell size becomes smaller as the cell density increases. On the other hand, when the cell density is constant, increasing the volume expansion ratio will increase the average cell size. Because plastic foams having a smaller cell size and a more uniform cell size distribution exhibit better properties^{5–12} and because foams having a large volume expansion ratio are lighter and use less material, substantial research efforts have been made to decrease the cell size with a large cell density and/or to increase the void fraction by increasing the volume expansion ratio. To obtain a desired expansion ratio, a cell-growth control strategy needs to be established.

Although the role of die geometry is significant in determining the cellular morphology, there has been no systematic study published in the literature on its

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effects on foaming. In this context, an experimental study has been conducted to systematically investigate the fundamental cell-growth mechanisms of extruded PS foams. In particular, this research has been focused on the effects of the die geometry on the volume-expansion behaviors of extruded PS foams blown with CO₂.

ANALYSIS OF PREMATURE CELL GROWTH ALONG THE DIE

In a continuous extrusion foaming process, cell nucleation usually occurs inside the die, and the nucleation rate is typically determined by the pressure-drop profile and the flow rate.^{13,14} Because cell nucleation occurs when the pressure of the polymer/gas solution drops below the solubility pressure, a certain amount of cell growth will occur inside the die from this moment of cell nucleation until the solution exits the die. This unavoidable and undesirable cell growth is called *premature cell growth* in this study. The amount of premature cell growth inside the die plays an important role in affecting the final expansion ratio.

Once cell nucleation occurs, the stabilized cells tend to grow with the gas molecules that diffuse into the nucleated cells from the melt. The amount of growth for each cell is well correlated to the total amount of gas that has diffused into the nucleus from the polymer melt unless the melt stiffness is too strongly governing the cell-growth rate.¹⁵ This amount of diffused gas for each cell in the early stage of cell growth can be well approximated by the amount of gas that is initially dissolved in a polymer volume around the nucleated cell and participates in the diffusion. The gas concentration in the polymer matrix around the cell is rather complex and highly nonlinear,¹⁶ and the polymer volume that is involved in gas diffusion for cell growth increases over time. However, a hypothetical volume, called a depleted region, in which the gas concentration is too low to nucleate additional cells,^{17,18} can be defined to estimate the amount of gas that is used in the early-stage cell growth inside the die:

$$m \approx C_s \times V_{\text{depleted}} \approx C_s \times \frac{4}{3} \pi \ell_{\text{diffusion}}^3 \quad (1)$$

where m is the amount of gas that is used in the early-stage cell growth inside the die, C_s is the gas concentration per unit of volume, V_{depleted} is the volume of the hypothetical depleted region, and $\ell_{\text{diffusion}}$ is the diffusion distance of a gas molecule at time t .

The amount of diffused gas for all the cells per unit of volume of the polymer/gas mixture ($M_{\text{premature}}$) is

$$M_{\text{premature}} = Nm + M_0 \approx \frac{4}{3} \pi N C_s \ell_{\text{diffusion}}^3 + M_0 \quad (2)$$

where N and M_0 are the cell density and amount of undissolved gas, respectively.

The diffusion distance (ℓ) in eq. (2) can be approximated by¹⁵

$$\ell \approx \sqrt{D t_{\text{premature}}} \quad (3)$$

where D is the diffusivity of the gas and $t_{\text{premature}}$ is the premature cell-growth time that elapses from the cell nucleation point until the polymer/gas mixture comes out of the die. Now, the amount of premature cell growth can be estimated as follows:

$$M_{\text{premature}} \approx \frac{4}{3} \pi N C_s \left(D t_{\text{premature}} \right)^{\frac{3}{2}} + M_0 \quad (4)$$

Equation (4) indicates that a high cell density, a high gas concentration, a high diffusivity, and a high premature cell-growth time will in general promote premature cell growth that occurs inside the die. The gas concentration and the diffusivity will not be affected by the die geometry as long as the die pressure is high enough to dissolve all the injected gas in the polymer melt. However, the cell density¹⁴ and the premature cell-growth time are strongly dependent on the die geometry. The following paragraph demonstrates how the die geometry affects the premature cell-growth time.

The premature cell-growth time can be derived as a function of the gas concentration, the die geometry, the flow rate, and the temperature. First, it is assumed that cell growth inside the die does not affect the velocity of the melt flow significantly, (i.e., the volume flow rate is constant along the die channel), although the velocity is actually increased. It is also assumed that the pressure drops linearly along the die despite the occurrence of cell nucleation.¹⁹ If the entrance and exit pressure losses are ignored, the following expression can be obtained (see Fig. 1):

$$\frac{P_{\text{solubility}}}{P_{\text{die}}} = \frac{t_{\text{premature}}}{t_{\text{residence}}} \quad (5)$$

where $P_{\text{solubility}}$ is the solubility pressure for a given amount of gas in the given polymer, P_{die} is the die pressure at a processing condition, and $t_{\text{residence}}$ is the total residence time of the melt through the die. The residence time can be expressed in terms of the die geometry and the flow rate:

$$t_{\text{residence}} = \frac{L}{v} = \frac{\pi R^2 L}{Q} \quad (6)$$

where R and L are the radius and length of the filament die, respectively; v is the average velocity of the polymer/gas solution; and Q is the volume flow

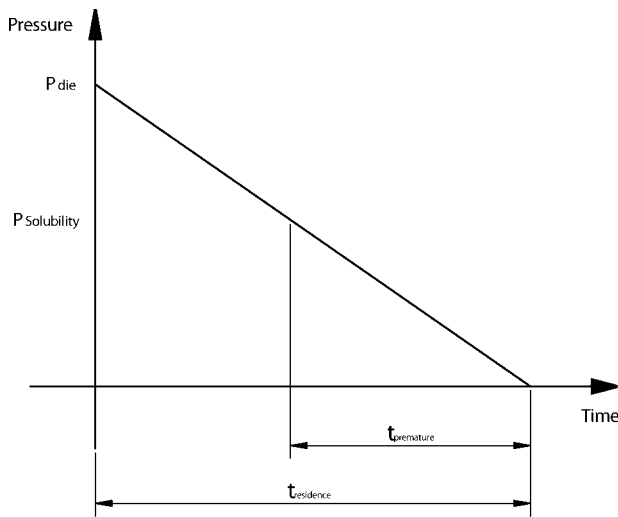


Figure 1 Relationship between the die pressure (P_{die}), solubility pressure ($P_{solubility}$), residence time ($t_{residence}$), and premature cell-growth time ($t_{premature}$).

rate. From eq. (5), the premature cell-growth time can be estimated as follows:

$$t_{premature} = \frac{P_{solubility}}{P_{die}} \times \frac{\pi R^2 L}{Q} \quad (7)$$

Apparently, the premature cell-growth time is determined by the die pressure during foam processing because the volume flow rate is normally set to be a desired value (e.g., with a gear pump), the solubility pressure is almost determined for a given amount of gas, and the die geometry is fixed during the operation. It is well known that the die pressure is governed by the melt and die temperatures,^{20,21} and so the premature cell-growth time increases as the melt and die temperatures increase. The die pressure can be approximated in terms of the die geometry and the flow rate on the basis of the power-law model:²²

$$P_{die} = \frac{2kL}{R} \left[\frac{Q(3 + \frac{1}{n})}{\pi R^3} \right]^n \quad (8)$$

where k is a consistency constant, and n is power law index. Therefore, the premature cell-growth time is expressed by

$$t_{premature} = \frac{P_{solubility} \left(\frac{\pi R^2}{Q} \right)^{n+1}}{2k \left(3 + \frac{1}{n} \right)^n} \quad (9)$$

The pressure-drop rate can be estimated by^{14,23}

$$\frac{dp}{dt} \approx 2k \left(3 + \frac{1}{n} \right)^n \left(\frac{Q}{\pi R^3} \right)^{n+1} \quad (10)$$

where dp/dt is the pressure-drop rate. The premature cell-growth time becomes a function of the solubility pressure and the pressure-drop rate:

$$t_{premature} = \frac{P_{solubility}}{dp/dt} \quad (11)$$

The solubility pressure can be estimated with Henry's law²⁴ for a given gas concentration and temperature. Henry's law constant [cm^3 (STP)/kg MPa], determined by Sato et al.²⁵ for the PS/ CO_2 system, and the critical temperature (304.1 K) for CO_2 , from ref. 26, were used in this study. The solubility pressure can be expressed as follows:

$$P_{solubility} = C_s \exp \left(- \left(6.400 + 2.537 \times \left(\frac{304.1}{T} \right)^2 \right) \right) \quad (12)$$

where C_s is the saturation concentration of gas in the polymer (cm^3/g or g of gas/ g of polymer), and T is the melt temperature. Finally, eq. (11) becomes

$$t_{premature} = \frac{C_s}{dp/dt} \exp \left(- \left(6.400 + 2.537 \times \left(\frac{304.1}{T} \right)^2 \right) \right) \quad (13)$$

Combining eqs. (4) and (13) leads to

$$M_{premature} = \frac{4}{3} \pi N C_{s,0}^3 \cdot D^3 \left(\frac{\exp \left(- \left(6.400 + 2.537 \times \left(\frac{304.1}{T} \right)^2 \right) \right)}{dp/dt} \right)^{n+1} + M_0$$

$$= \frac{4}{3} \pi^{\frac{5+3n}{2}} N C_s^3 D^{\frac{3}{2}} \cdot \frac{R^{\frac{9}{2}(n+1)}}{(2k)^{\frac{3}{2}} \left(3 + \frac{1}{n} \right)^{\frac{3n}{2}} Q^{\frac{3(n+1)}{2}}} \cdot \exp \left(- \left(9.600 + 3.8055 \times \left(\frac{304.1}{T} \right)^2 \right) \right) + M_0 \quad (14)$$

Equations (13) and (14) indicate that only the radius in the elementary die geometry affects the amount of premature cell growth as long as the length of the die is long enough to make the die pressure higher

than the solubility pressure. Figure 2 schematically shows how the premature cell-growth time changes with different die geometries. Figure 2(a) indicates that when two dies have the same pressure-drop rate

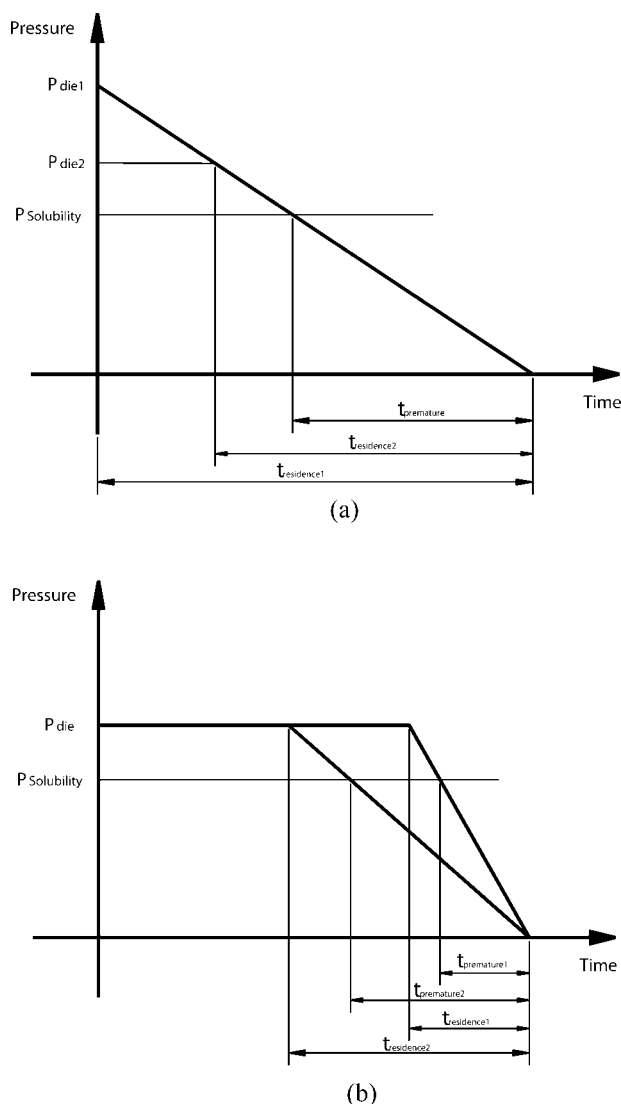


Figure 2 Schematic comparing the premature cell-growth time of two dies: (a) the same pressure-drop rate and different die pressures and (b) the same die pressure and different pressure-drop rates.

but different die pressures, they provide the same premature cell-growth time. Because the cell densities of the two dies having the same pressure-drop rate are the same,¹⁴ these two dies have the same amount of premature cell growth according to eq. (4). Figure 2(b) illustrates that when two dies have the same die pressure but different pressure-drop rates, the die with a higher pressure-drop rate will provide a shorter premature cell-growth time. Even though a higher pressure-drop rate will result in a higher cell density, the scale of the resultant increase in the cell density is smaller than the required increase in the pressure-drop rate; on the other hand, according to eq. (14), the pressure-drop rate has a higher impact on premature cell growth than the cell density (i.e., an order of 1.5 vs 1), so less pre-

mature cell growth is expected for the die with a higher pressure-drop rate. This implies that the die geometry needs to be carefully chosen to obtain proper control of premature cell growth.

The amount of premature cell growth can affect the final expansion ratio of the extruded foam significantly. Naguib et al.²⁷ identified the expansion mechanisms of extruded foam at various temperatures. As they identified the effect of the talc content on the expanding behaviors of extruded foam through the timing of an initial hump, they also observed that the initial hump is detrimental to the expansion behavior.²⁸ Because a large amount of premature cell growth can cause the formation of the initial hump, efforts need to be made to suppress premature cell growth as much as possible. In other words, the volume expansion ratio can be substantially sacrificed with a large amount of premature cell growth even when the temperature is optimized. Therefore, both the temperature and die geometry need to be carefully selected to maximize the expansion ratio.

EXPERIMENTAL

Materials

The plastic material used in this study was PS 101 from Nova Chemicals (Calgary-Alberta, Canada). The melt flow index of this material was 2.2 dg/min. The blowing agent used in the experiments was CO_2 (Matheson, 99.5%). The amount of CO_2 was fixed at 10 wt % in all the experiments. No nucleating agent was used.

Experimental setup

A schematic of the experimental extrusion setup used in this study is shown in Figure 3. The setup consisted of a 7.5-hp variable-speed drive (EPL-V7752 plasticorder, Brabender, South Hackensack, NJ), a 3/4" extruder (05-25-000, Brabender) with a mixing screw with a 30 : 1 length/diameter ratio (05-00-144, Brabender) for plasticating the polymer pellets and dispersing the injected blowing gas throughout the melt, a positive displacement pump for injecting the blowing gas, a six-element static mixer with a 6.8-mm diameter (FMX-84441-S, Omega, Stamford, CT) for enhancing the polymer/gas solution formation, a gear pump (PEP-II 1.2 cc/rev, Zenith, Monroe, NC) for controlling the melt flow rate, a heat exchanger containing homogenizing static mixers (model H-04669-12, Labcore) for homogenizing the melt temperature, a gas injection port for injecting the blowing gas, six band heaters, four pressure transducers (PT462B-10M-6/18, Dynisco, Franklin, MA) for detecting the pressures

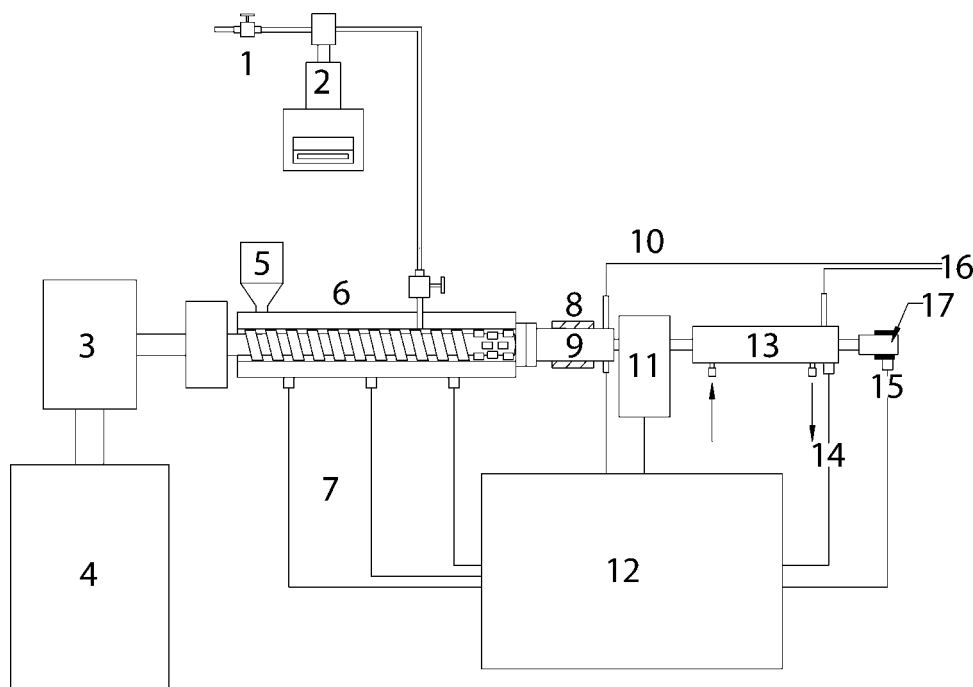


Figure 3 Schematic of the experimental system setup: (1) blowing agent supply valve, (2) positive displacement pump, (3) gear box, (4) variable-speed direct-current motor, (5) hopper, (6) extruder, (7) thermocouples, (8) heater, (9) mixing device, (10) pressure transducers, (11) gear pump, (12) control panel, (13) heat exchanger, (14) melt temperature, (15) nozzle temperature, (16) connection to control panel, and (17) nucleation die.

at various important locations, and six temperature controllers and six thermocouples for controlling the temperatures of the extrusion barrel, the mixer, the heat exchanger, the gear pump, and the die.

Design and calibration of the dies

To study the effects of the die geometry on the volume expansion behavior of extruded foams, three distinct groups of filamentary dies were designed and implemented in this study. The rationales and detailed procedure for the die design and calibration are described in our previous publication.¹⁴ The designed die geometry and characteristics of these dies are briefly described here. Each die group had the same pressure-drop rate by having the same diameter, whereas there were three different levels of pressure drops [13.8 MPa (2000 psi), 20.7 MPa (3000 psi), and 27.6 MPa (4000 psi)] in each die group with a different die length for each die. Die groups 1, 2, and 3 had values of 4.16, 0.416, and 0.082 GPa/s, respectively. All the die geometry information is summarized in Table I and Figure 4.¹⁴

RESULTS AND DISCUSSION

Extensive foaming experiments were conducted at various die temperatures in the range of 120–170°C; the temperature was changed by decrements of 5°C

while the die geometry was varied within the three die groups. The obtained results were then plotted for various die geometry and processing parameter combinations. Figures 5 and 6 illustrate the experimentally obtained volume expansion ratios and cell densities as a function of the die temperature for all the dies.

Figure 7 shows the pressure changes as a function of the temperature for all the dies. Although dies 1, 4, and 7, dies 2, 5, and 8, and dies 3, 6, and 9 were supposed to have the same pressures,¹⁴ there were some variations in the actual pressures measured from these dies. The dies in each die group (e.g., dies 1, 2, and 3) had the same pressure-drop rate because they had the same diameter¹⁴ [see eq. (10)].

TABLE I
Calibrated Geometry of the Dies for PS with Pressure Drops of 27.6, 20.7, and 13.8 MPa

Die group	Die number	Die diameter	Die length
1	1	0.4572 mm (0.018 in.)	6.79 mm (0.27 in.)
	2	0.4572 mm (0.018 in.)	5.59 mm (0.22 in.)
	3	0.4572 mm (0.018 in.)	3.81 mm (0.15 in.)
2	4	0.8128 mm (0.032 in.)	35.56 mm (1.4 in.)
	5	0.8128 mm (0.032 in.)	21.59 mm (0.85 in.)
	6	0.8128 mm (0.032 in.)	14.22 mm (0.56 in.)
3	7	1.2192 mm (0.048 in.)	68.58 mm (2.7 in.)
	8	1.2192 mm (0.048 in.)	50.80 mm (2.0 in.)
	9	1.2192 mm (0.048 in.)	34.04 mm (1.34 in.)

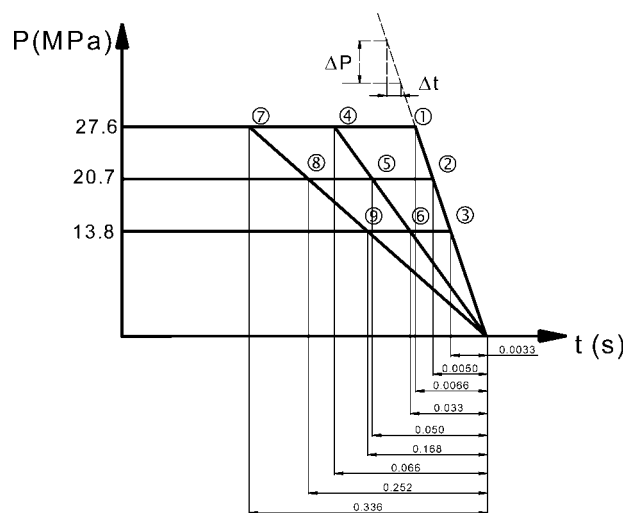


Figure 4 Design of a series of 9 dies and a comparison of the pressure-drop rates pertaining to the different dies (ΔP = change in pressure; Δt = change in time; P = pressure; t = time).

Effect of the die temperature on the volume expansion

The results revealed that as the die temperature decreased, the volume expansion ratio initially increased and then decreased. An optimum temperature for attaining the maximum volume expansion ratio could be consistently observed with respect to each die. It appeared that, in the high-temperature region, the loss of the blowing agent governed the expansion ratio because of the high diffusivity of the gas and the weak melt strength of the polymer.^{20,27} Especially at a very high temperature, gas loss was so severe that a very low volume expansion ratio was obtained regardless of the die geometry; on the other hand, in the low-temperature region, stiffening

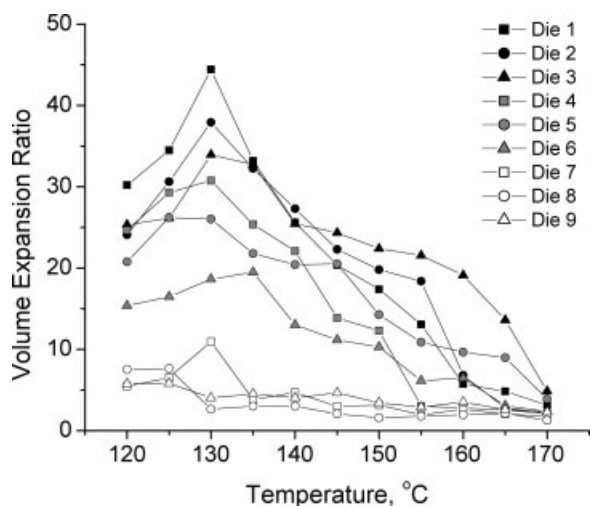


Figure 5 Volume expansion ratios obtained at various die temperatures.

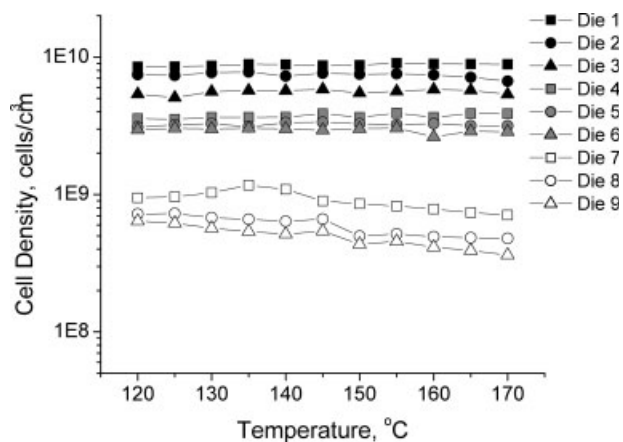


Figure 6 Cell densities obtained at various die temperature.

of the polymer dominated the cell growth because of the increased melt stiffness and the low diffusivity of the gas.^{20,27} All these results confirmed the previous observation on the effect of temperature on the final expansion ratio for other polymer and blowing agent combinations.

Effect of the die geometry on the volume expansion through its influence on the die pressure

The pressure profile along the die is a dependent-state variable and is governed by multiple factors, such as the die geometry, temperature, plastic material, blowing-agent concentration, nucleating-agent concentration, and flow rate (if a gear pump is used). Therefore, the mechanism by which the die pressure affects the volume expansion ratio could be rather complex. To investigate the effect of the die pressure through the die geometry on the volume expansion ratio, all other parameters should be fixed, and only the die geometry should be varied.

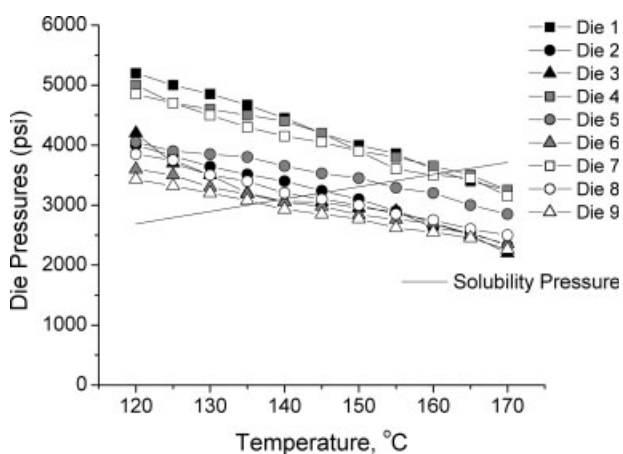


Figure 7 Die pressures measured at various temperatures.

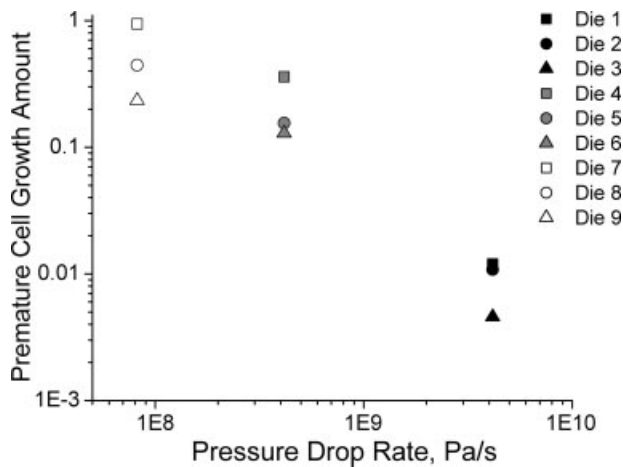


Figure 8 Amount of premature cell growth obtained at the optimum temperature as a function of the pressure-drop rate.

Figure 2(a) indicates that pressure would not affect the premature cell-growth time if the die radius (and thereby the pressure-drop rate) were the same [see eqs. (9) and (11)]. Because the cell density would not be a sensitive function of pressure either¹⁴ (see Fig. 6), the premature cell growth would not be affected by pressure via its effects on cell density. The gas concentration and diffusivity terms in eq. (4) would be invariant with respect to the pressure. The gas concentration in the polymer melt would be the same as long as the pressure were higher than the solubility pressure and all the gas dissolved in the polymer. The diffusivity would also be the same if the polymer molecules relaxed quickly as the pressure decreased. This would be a reasonable assumption at a high processing temperature. However, if not, the gas diffusivity at a higher initial pressure would be lower because of the smaller free volume of the polymer matrix. The relaxation rate of the polymer/gas mixture and the effect of the free volume on the diffusivity need to be studied to clarify this issue. However, it seems that the diffusivity would not differ by an order of magnitude because of the lubrication effect of the supercritical fluid in the polymer matrix. This contemplation indicates that all the dies in each group are supposed to have the same amount of premature cell growth. Although Figure 8 indicates some difference in the premature cell growth within the same die group, it is believed that the difference in the calculated premature cell growth amount among the dies in the same die group resulted from the variation of the actual cell densities obtained from the dies in the same group. The variation of the volume expansion ratios for the same die group shown in Figure 5 would be not from the amount of premature cell growth but from another factor (or factors) such as varied cell density.

On the other hand, if the pressure were too low (i.e., below the solubility pressure), then undissolved gas pockets would be formed in the polymer melt, and this would cause an initial hump to be formed at the die exit for the same reason described earlier.

Effect of the die geometry on the volume expansion through its influence on the pressure-drop rate

Figure 5 shows that a high pressure-drop rate is favorable for obtaining a large expansion ratio for the PS-CO₂ system. As the pressure-drop rate decreased from die group 1 to die group 3, the expansion ratio decreased. There must have been a number of factors that affected the volume ratio.

First of all, it is believed that the amount of premature cell growth affected the expansion ratio significantly. Despite the largest cell densities of die group 1, as shown in Figure 6, the highest pressure-drop rate (or equivalently the smallest radius) in eq. (14) made the amount of premature cell growth of die group 1 the smallest because a higher pressure-drop rate results in a shorter premature cell-growth time, and the pressure-drop rate has a higher impact on premature cell growth than cell density. Figure 8 shows the amount of premature cell growth (obtained at the optimum temperature for each die) decreased as the pressure-drop rate increased. Figure 9 shows the maximum expansion ratio of each die obtained at the optimum temperature versus the amount of premature cell growth. It seems that a larger amount of premature cell growth negatively affected the maximum expansion ratio achievable.²⁷ As the amount of premature cell growth increased (from die group 1 to die group 3), the maximum expansion ratio obtained from each die decreased.

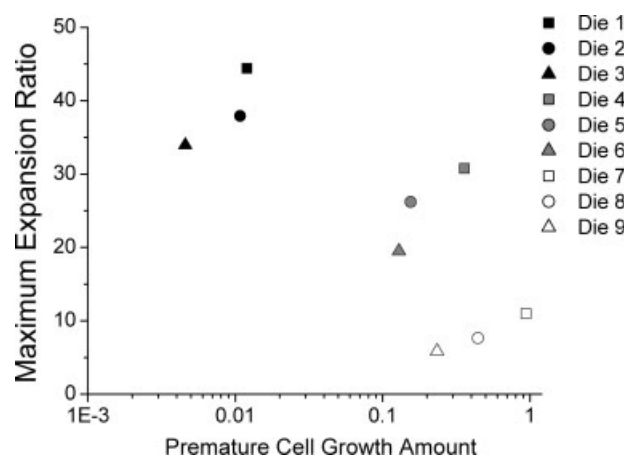


Figure 9 Maximum expansion ratios obtained at the optimum temperature versus the amount of premature cell growth.

There could have been some other factors such as the large number of cell layers in the cross section of the extruded foam²⁹ due to a large cell density generated at a high pressure-drop rate.^{13,14} Lee and Ramesh²⁹ observed an increase in the expansion ratio of extruded low-density polyethylene foams with a high cell density due to the localized gas loss in the foam surface area. However, the number of cell layers in our foams was large enough to ignore the localized gas loss effect because of the relatively high cell densities obtained even from die group 3. Therefore, it is contemplated that the amount of premature cell growth inside the die played the most significant role in determining the maximum expansion ratio.

CONCLUSIONS

We investigated the effects of the die geometry and the temperature through the pressure, pressure-drop rate, and premature cell growth on the volume expansion behavior of extruded PS foam. Three groups of carefully calibrated dies with different pressures and pressure-drop rates by the variation of the die geometry were used in this study.

The amount of premature cell growth apparently played a critical role in determining the expansion ratio of the extruded PS foams blown with CO₂. The high pressure-drop rate dies were favorable in promoting the expansion ratio. For each die geometry, there was an optimal temperature to maximize the expansion ratio of the PS foam. However, apart from the temperature, the die geometry was also observed to be one of the most critical parameters for the expansion behavior.

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