

Microcellular Thin PET Sheet Foam Preparation by Compression Molding

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ABSTRACT: This study has created a new way of microcellular processing PET thin sheet foam using a conventional hydraulic press by compression molding and by setting the temperature of press plates differently. Comparing this study with our previous work, the emphasis is on the difference of plate temperature. The nonisothermal condition is used to control the foaming agent decomposition to lead to more uniform cell size microcellular foam. A variety of cell sizes, cell densities, and relative densities are obtained as a consequence of the different foaming temperature, time, pressure, and foaming agent content via isothermal and nonisothermal conditions. The effect of isothermal and

nonisothermal foaming on the cell size, cells density, and relative density has been discussed. The electrical properties of the microcellular poly(ethylene terephthalate) (PET) samples prepared in nonisothermal foaming have been investigated. The experimental results show that the microcellular PET foam has lower dielectric constant and dielectric loss and higher electric resistivity than unfoamed PET. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 93: 1698–1704, 2004

Key words: microcellular foam; polyester; compression; cell size; dielectric properties

INTRODUCTION

The microcellular foam was invented at the Massachusetts Institute of Technology (MIT) under the direction of Professor Nam P. Suh^{1,2} and has been studied extensively for the past 20 years.^{3–18} In the beginning, the microcellular foam was prepared in a two-stage batch process.¹⁷ In the first stage, the polymer is placed in a pressure vessel with a high-pressure and nonreacting gas at room temperature. Over time, the gas diffuses into the polymer and produces a “supersaturated” specimen that is thermodynamically unstable because of the excessive gas dissolved into the polymer. In the second stage, the specimen removed from the pressure vessel is heated to the foaming temperature that is above the glass transition temperature of the gas-polymer system in order for the bubble to nucleate and grow. This batch process is characterized by high pressure (such as 25 MPa), long processing cycle time (more than several hours, even days), and foaming in the solid state. Then, Suh et al. tried to use the extruder to perform the microcellular processing continuously and to prepare the microcellular foam in the melt state. The continuous extrusion microcellular foam is based on the concept of a rapid change in solubility created by thermodynamic instability. Supercritical

fluids of atmospheric gases are injected into a polymer melt under specified pressure profiles and mixed to create a homogeneous solution within the polymer melt. When the polymer melt solution comes through the die, a large pressure drop results in a thermodynamic change. The supercritical fluid precipitates from the solution and leads to cell nucleation and growth. Development and demonstration of continuous laboratory scale microcellular processing equipment was done at MIT. The continuous microcellular foam molding process was developed and commercialized at Trexel, Inc. (Woburn, MA). Until now, Trexel, Inc. has developed microcellular processing methods such as, extrusion, injection, and blow molding to produce polystyrene (PS), polyethylene, polypropylene, polyvinyl chloride, and thermoplastic elastomer, etc. microcellular foams.

Other researchers also have successfully prepared many kinds of microcellular polymer foams with various methods. Goel and Beckman¹⁹ used a pressure-quench method via supercritical carbon dioxide to prepare microcellular polymer. He et al.²⁰ followed the pressure-quenched method of Goel and Beckman, prepared the microcellular foams of PS / lighted sulfonated PS and PS / zinc-neutralized lighted sulfonated PS blends under high pressure and reduced the foaming time under pressure to 6–10 h. They found that both high-foaming temperature and lower pressure resulted in large cell size, lower cell density, and lower relative density for microcellular ionomers and blends and microcellular PS. Tomoko and Sato et al.²¹ prepared PS microcellular foam in a batch process.

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The 1.3-mm thickness PS sheets were placed in a stainless steel saturation vessel and nitrogen saturation pressure was kept constant at 25 MPa. After saturating for a long time, the samples were heated quickly to 393–433 K to generate a microcellular structure with a cell size $\leq 100 \mu\text{m}$. Their results showed that with temperature increases, the nitrogen solubility increases, the average cell diameter and volume expansion ratios increase, and the cell density decreases. When the foaming temperature was 433 K, the cell structure shattered after microcellular processing; i.e., the foaming temperature for their method is lower than 433 K. Furthermore, the polymer-solvent phase separation also has been used to create microcellular structures.^{22,23} Therefore, from the state point of view, the microcellular polymer foams can be prepared under solid, melt, or solution state. One unique feature of the solid-state microcellular process is the ability to create an unfoamed skin of a desired thickness that is integral to the foam itself.²⁴

Looking at the development of microcellular polymer foams since 1980, we see that most of the research efforts have focused on innovative processing methods. Recently, we have created a new method, that is, using a conventional press to prepare thin poly(ethylene terephthalate) (PET) sheet foam under the isothermal foaming condition, which can not be microcellularly processed via extrusion and injection molding or via a traditional foaming process. Our method has the advantages of a very short processing cycle, being easy to operate, simplicity, a relatively low foaming pressure, and it is more economical compared with the pressure vessel methods.

In this study, we have modified our microcellular foaming method, i.e., we changed the isothermal foaming condition to nonisothermal foaming. The temperature of the two plates of the press is set differently, aiming to control the decomposition rate of the foaming agent and to produce more uniform cell-size microcellular PET foam. A series of samples was prepared under different foaming temperature, time, pressure, and foaming agent content in isothermal and nonisothermal conditions. The effects of the foregoing processing parameters on the cell size, cell density, and relative density have been discussed, and both isothermal and nonisothermal foaming results have been compared. The electrical properties of the samples prepared under nonisothermal conditions were investigated also.

EXPERIMENTAL

A commercial-grade PET sheet with a thickness of 0.15 mm from the Oriental Insulation Materials Factory of China (Mianyang, Sichuan, China) was used as received. A low-density polyethylene (LDPE) pellet from Maoming Factory of China (Maoming, Guangdong, China) was used as received. A commercial grade of azobisformamide (AC), a foaming agent, and

dicumyl peroxide (DCP) were used without any further purification.

The microcellular foam of the thin PET sheet was prepared by first mixing foaming gas substrate in a two-roll mill for a few minutes, and then the PET and the substrate were loaded into a $250 \times 250 \times 2$ -mm mold and put into a hydraulic press under desired experimental conditions. Once the required foaming time and other conditions were reached, the mold was taken out of the hot press and quenched at room temperature; thus, the microcellular PET foam samples were ready for characterization.

The cell size of the foamed samples was observed under Leitz optical microscopy (using a $100\times$ amplifying camera lens; Leitz Camera AG, Germany), followed by a procedure described previously by Kumar et al.²⁵ and eqs. (1) and (2),⁴ where V_f is the void fraction of the foamed material, D_c is the average cell size measured from the photographs, N_f is the cell density relative to the foamed material, and ρ_c is the cell density relative to the neat material:

$$V_f = \pi D_c^3 N_f / 6 \quad (1)$$

$$\rho_c = N_f / (1 - V_f) \quad (2)$$

Apparent density of the foamed samples was measured followed by the immersion liquid method (GB 1033–86) and eq. (3), where ρ_t is the apparent density at measured temperature, ρ_x is the immersion liquid density, a is the mass of the sample and copper wire, w is the mass of copper wire, b is the apparent mass of the sample and copper wire inside of the immersion liquid, and c is the apparent mass of the copper wire and metal hammer inside of the immersion liquid. The relative density is calculated according to eq. (4), where ρ_R is the relative density and ρ_0 is the unfoamed PET density at measured temperature:

$$\rho_t = (a - w)\rho_x / (a - b + c) \quad (3)$$

$$\rho_R = \rho_t / \rho_0 \quad (4)$$

The electrical properties, including insulating property, electric resistivity, dielectric constant, and dielectric strength, were measured. The insulating property was performed in a Zc-36 $10^7 \Omega$ superhigh resistance instrument, following the method of volume resistivity and surface resistivity, with aluminum foil as the electrode material, test voltage 500 V at 23°C, and 86% relative humidity (RH). Dielectric property was performed in a Schering bridge, following the method of measuring the relative dielectric constant and dielectric loss factor of solid material under working, audio, and high frequencies. The sample size is 50×50 mm, and the measurement condition is set at 1 kV voltage, a 50-Hz frequency, and 25°C. The dielectric strength

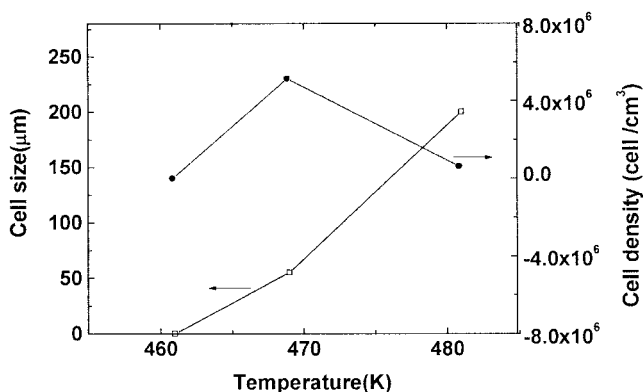


Figure 1 Effect of foaming temperature on microcellular PET cell size and density [$p = 16$ MPa (AC) = 12%; $t = 3$ min].

measurement was done based on the strength test method of the working frequency electronics of insulation material, continuously increasing voltage at 1 kV/sec, 23°C, and 86% RH. The sample size is the same as aforementioned, and the measurement was performed in the medium of air and pure transformer oil separately.

RESULTS AND DISCUSSION

The microcellular foaming method we used is in the $T_g - T_m$ temperature range and close to the PET melting point side. The foaming occurs in the solid state of PET material. Actually, it is in the amorphous, intercrystalline, and introcrystalline regions of semicrystalline PET, which we proved by our previous work.²⁶ The advantages of using compression molding in the microcellular processing of PET sheets are a greatly shortened foaming time, simplicity, and it is easy to operate and more economical. Furthermore, for very thin sheets, it is very difficult to use the conventional extrusion or injection molding to prepare microcellular foam. The conventional polymer foams that typically have cell sizes of ≥ 0.25 mm are not suitable for using in an application involving a thin wall product because of excessive loss of strength.¹⁷ Thus, our method of preparing thin sheet PET microcellular foam has very important significance in theory and application, and it has the potential to be industrialized in the near future.

During microcellular processing of PET thin sheet foam, the cell size, cell density, and relative density are affected by the foaming temperature, time, pressure, and foaming agent content under isothermal and nonisothermal foaming conditions.

Isothermal foaming

In this process, the temperatures of the upper and lower plate of the press are set the same, so the foam-

ing is achieved isothermally. The decomposition of the foaming agent is controlled by a single temperature. When the foaming temperature is lower than 461 K, no detectable cells can be observed. One possible reason is that the foaming agent AC can not decompose to provide enough gas to diffuse into the PET sheet to initiate cell nucleation at that temperature. Another reason is that at a lower foaming temperature, the matrix stiffness may be sufficiently large to prevent nucleated cells from growing to a detectable size even though the allotted foaming time would allow all of the available gas to diffuse into the cells. However, when the foaming temperature is higher than 485 K, the cell structure can not be observed clearly under an optical microscopy, suggesting that the very soft matrix and high chain moving ability can not provide enough stiffness to fix the formed microcells, and some of them collapsed and melted together. Figure 1 shows the temperature effect on the cell size and density. From Figure 1, we can see that the cell size increases quickly when the foaming temperature increases from 470 to 480 K. When the foaming temperature variation is small, the cell size increases and cell density decreases greatly. The cell nucleation is depressed on increasing temperature. Thus, increasing foaming temperature favors the cell growth and forms large-size cells under isothermal foaming condition.

As depicted in Figure 2, the foaming time plays an important role in the microcellular PET cell size and density. When foaming time reaches 2 min, the minimum cell size and the maximum cell density have been obtained. After that, the cell size increases and the cell density decreases with increasing foaming time. This indicates that the effect of increasing the foaming time actually is increasing the heating time and chain moving ability, leading to cell growth and large cell size. Long foaming time can result in large cell size and cause degradation, which results in brittle samples. Therefore, the foaming time should not be over 10 min when using the compression molding to prepare microcellular foam.

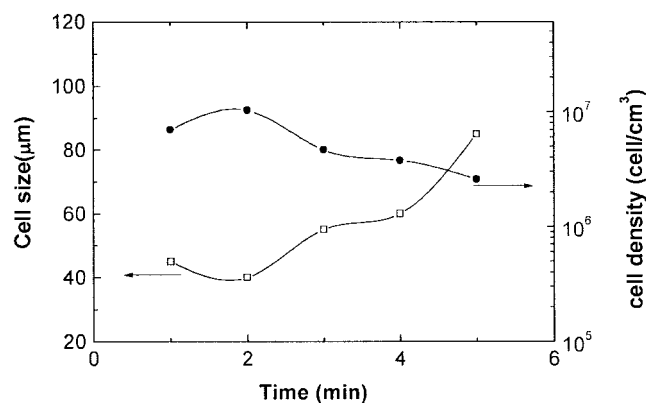


Figure 2 Effect of foaming time on microcellular PET cell size and density [$p = 16$ MPa (AC) = 12%; $T = 470$ K].

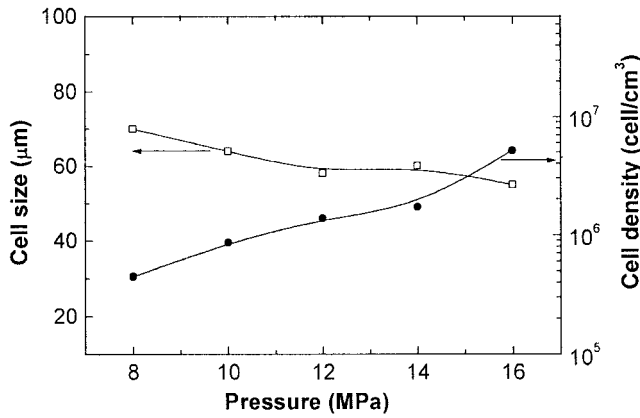


Figure 3 Effect of foaming pressure on microcellular PET cell size and density [$T = 470$ K (AC) = 12%; $t = 3$ min].

In Figure 3, the influence of the foaming pressure is depicted. From this figure, it is evident that the foaming pressure plays a significant role in the cell size and density. When the foaming pressure is < 8 MPa, no detectable cell structure can be observed as the produced gas may largely escape out of the mold rather than diffuse into the PET sheet to initiate nucleation. When increasing the foaming pressure, the cell size decreases and the cell density increases all the way. Apparently, the increased pressure favors cell nucleation and so, according to Sanchez-Lacombe's^{27,28} equation of state and the experimental data from Sato et al.,^{29,30} the solubility of nitrogen in polymer increases linearly with pressure up to ~ 25 MPa. Therefore, increasing the foaming pressure actually is increasing the foaming gas concentration and reducing the temperature and the chain moving ability inside of the PET sample, leading to the decreases in cell size and the increases in cell density.⁴

Figure 4 explicitly shows the influence of the foaming agent AC content on the microcellular PET foam cell size and density. It is evident from this figure that the foaming agent content has a larger

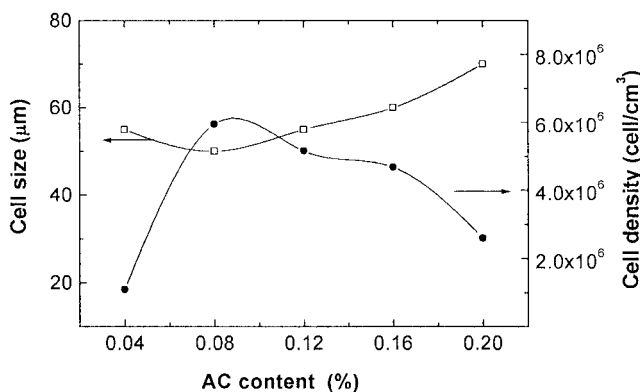


Figure 4 Effect of AC content on microcellular PET cell size and density ($p = 16$ MPa; $T = 470$ K; $t = 3$ min).

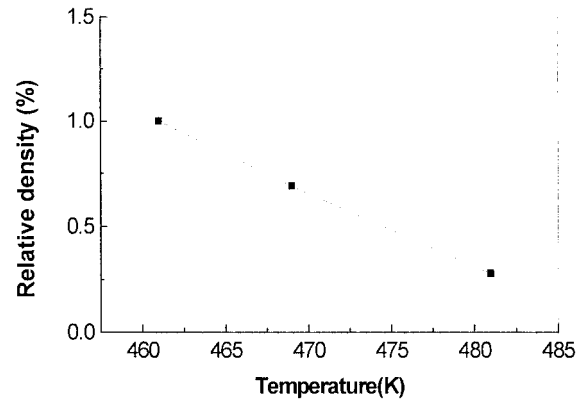


Figure 5 Effect of foaming temperature on microcellular PET relative density [$p = 16$ MPa (AC) = 12%; $t = 3$ min].

effect on the cell density than the cell size. With the foaming agent content increasing, the cell size first decreases slightly and then increases, while the cell density first increases largely and then decreases. Apparently, 8% AC content can provide the needed gas to initiate the satisfied cell nucleation. But considering the experimental deviation, we actually select 12% AC content as the fixed foaming agent content throughout all of our experiments when achieving isothermal foaming.

Figures 5–8 show the foaming condition effects on the microcellular PET relative density. With the foaming temperature, time, pressure, and foaming agent content increasing, the PET relative density decreases. Of these, the foaming temperature reduces the relative density significantly.

Nonisothermal foaming

From the foregoing isothermal foaming processing, we know that the small variation of the foaming temperature will change the cell size largely. Other processing parameters also play a significant role in the

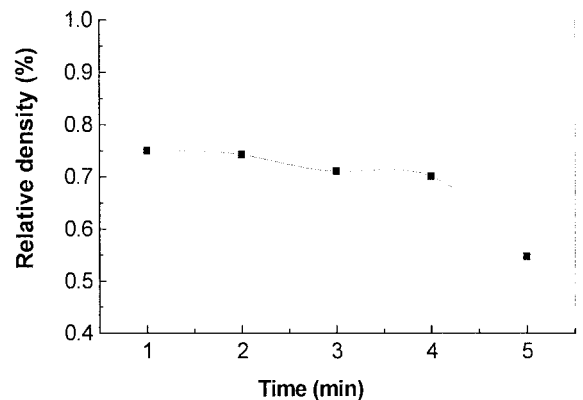


Figure 6 Effect of foaming time on microcellular PET relative density [$p = 16$ MPa (AC) = 12%; $T = 470$ K].

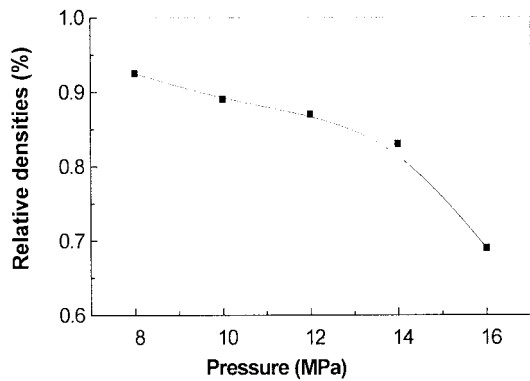


Figure 7 Effect of foaming pressure on microcellular PET relative density [$t = 470$ K (AC) = 12%; $t = 3$ min].

cell size. The processing parameters have affected the cell size largely, and the processing window is relatively narrow in isothermal foaming. To widen the processing window and obtain a more uniform cell size like the general pressure vessel did, we developed a new way to prepare the microcellular PET thin sheet foam by compression molding. That is, to set the upper-plate and lower-plate temperatures of the press differently to form a temperature gradient to control the foaming agent decomposing rate, we call it nonisothermal foaming. In nonisothermal foaming, the lower-plate temperature is set at 423 K, and only the upper-plate temperature of the press is altered.

Foaming temperature and foaming time influences on the cell size are shown in Figure 9. At 470 and 480 K foaming temperature of the upper plate of the press, no detectable cell can be observed when foaming time is <3 min, where 1-min foaming time can result in a microcellular PET sample in isothermal foaming. The possible reason for this is because at this time, there is still not enough gas to initiate the cell nucleation. Even when the foaming temperature of the upper plate reached 495 K, it still needed at least 2 min to produce the microcellular PET sample. At the 470 K foaming

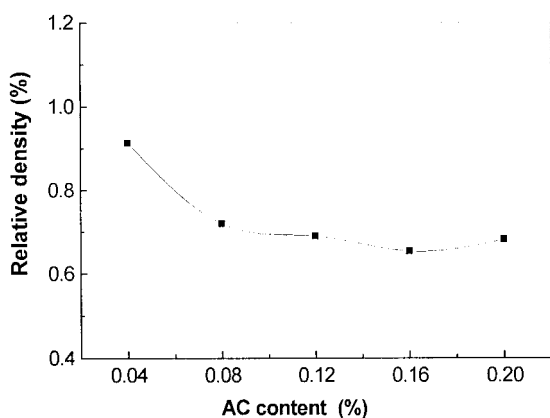


Figure 8 Effect of AC content on microcellular PET relative density ($p = 16$ MPa; $T = 470$ K; $t = 3$ min).

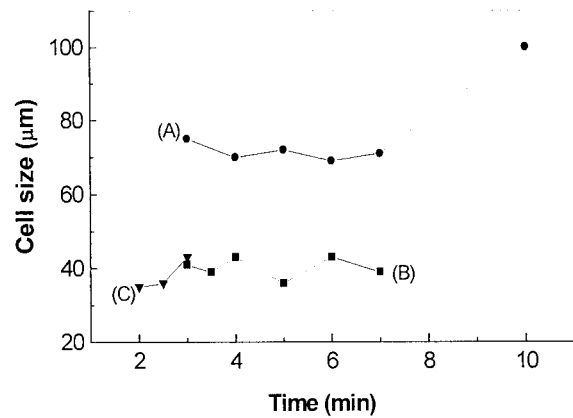


Figure 9 Effect of foaming temperature and time on microcellular PET cell size; (A) $T = 470$ K, (B) $T = 480$ K, and (C) $T = 495$ K [$p = 16$ MPa (AC) = 12%].

temperature, the foaming time can last 8 min to get the satisfied microcellular PET samples, compared with 4 min in isothermal foaming at the same foaming temperature. When the foaming temperature increases from 470 to 495 K, the cell size decreases rather than increases as in Figure 1 isothermal foaming. The higher the foaming temperature, the narrower the foaming time window. The foaming temperature can be as high as 495 K in nonisothermal foaming, whereas the highest in isothermal foaming is 480 K. In addition, Figure 9 also shows that as the foaming time increases, the cell size changes slightly. The cell size became larger only when the foaming time reached 10 min. The possible reason for this is that the lower temperature of the lower plate of the press resulted in the AC foaming agent decomposing gradually along the temperature gradient of the upper and lower plates to provide a constant foaming gas in a relatively long foaming time; and at the same time, the higher upper-plate temperature is facilitating the cell nucleation rather than cell growth in the narrow foaming

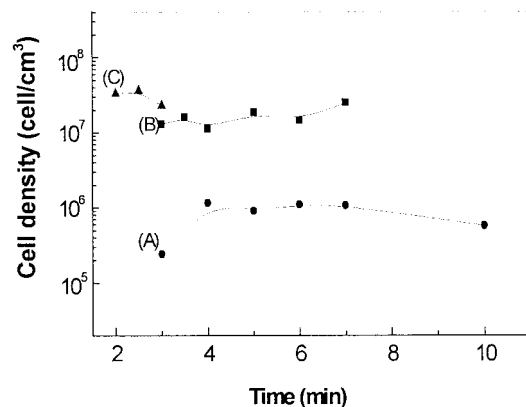


Figure 10 Effect of foaming temperature and time on microcellular PET cell density; (A) $T = 470$ K, (B) $T = 480$ K, and (C) $T = 495$ K [$p = 16$ MPa, (AC) = 12%].

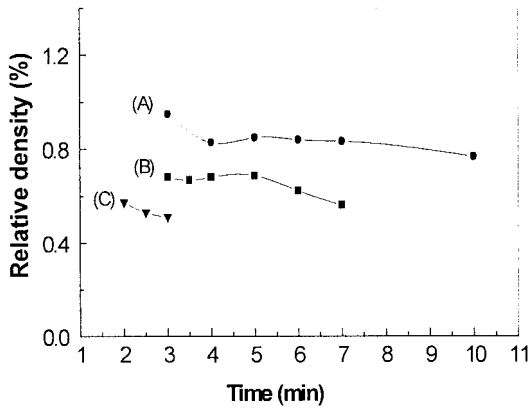


Figure 11 Effect of foaming temperature and time on microcellular PET relative density; (A) $T = 470$ K, (B) $T = 480$ K, (C) $T = 495$ K [$p = 16$ MPa (AC) = 12%].

time window (at 495 K); therefore, the cell size varies in a small amount and the smaller cell size can be obtained at a higher foaming temperature in nonisothermal foaming.

The effect of the foaming temperature and time on the cell density is depicted in Figure 10. It clearly shows that the foaming time has a small effect on the cell density, similar to that on the cell size in Figure 9. However, the foaming temperature affects the cell density significantly. The higher the foaming temperature is, the higher the cell density is. The higher foaming temperature promotes nucleation as Figure 10 shows. The corresponding PET relative density is shown in Figure 11; they agree with the cell size and cell density variations in Figures 9 and 10. At a foaming temperature of 495 K, the microcellular PET foam exhibits the lowest relative density.

The foaming agent content effect on the cell size is shown in Figure 12, which shows that the higher the AC content, the smaller the cell size, contrary to the

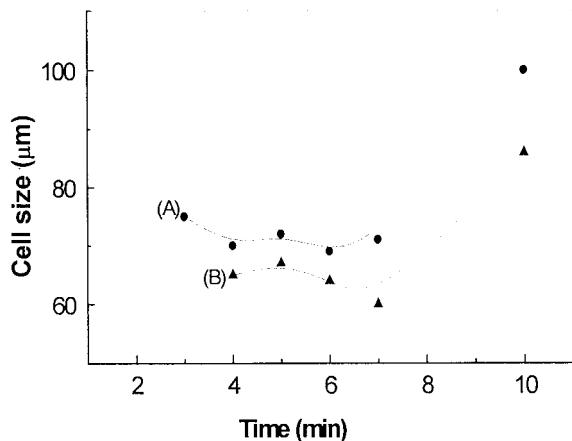


Figure 12 Effect of foaming agent and time on microcellular PET cell size; (A) [AC] = 12%, (B) [AC] = 20%; ($P = 16$ MPa, up plate temperature = 470K)

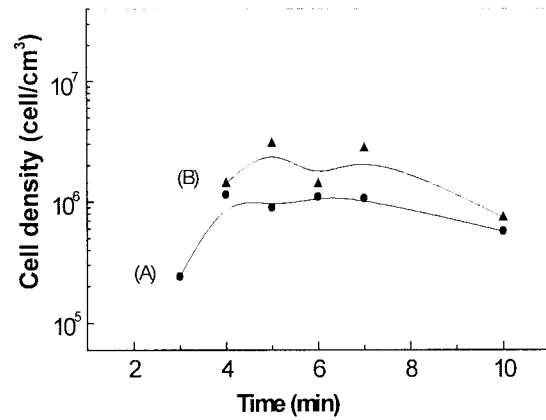


Figure 13 Effect of foaming agent and time on microcellular PET cell density; (A) AC = 12%, (B) AC = 20% ($p = 16$ MPa; upper plate temperature = 470 K).

isothermal foaming in Figure 4. Higher foaming agent AC content promotes nucleation and leads to higher cell density (Figure 13). The microcellular PET foam relative density is shown in Figure 14. Both Figures 13 and 14 reveal that the foaming time variation causes a smaller change in the cell density and the relative density. Hence, the more uniform cell size, cell density, and relative density microcellular PET has been obtained via nonisothermal foaming of compression molding.

Electric properties

Considering the application of microcellular foam, such as in electrical industry, several microcellular PET samples prepared via nonisothermal foaming have been selected to measure their electrical properties. As presented in Table I, the dielectric constant and dielectric loss of the microcellular PET are smaller than the unfoamed PET. Thus, the microcellular PET foam is more suitable to use in electrical industry

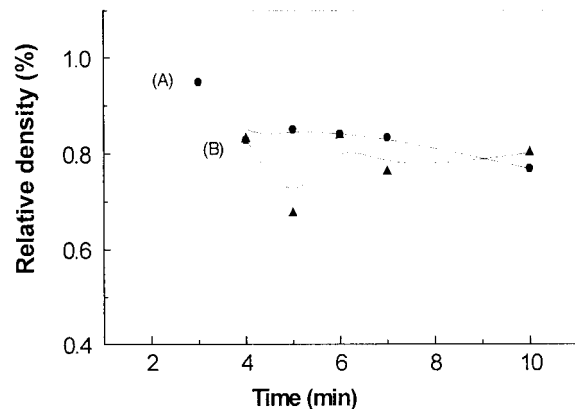


Figure 14 Effect of foaming agent and time on microcellular PET relative density; (A) AC = 12%, (B) AC = 20% ($p = 16$ MPa; upper plate temperature = 470 K).

TABLE I
Electrical Properties of Microcellular PET ($p = 16$ MPA, Nonisothermal Foaming)

Code Property	1	2	3	4	5	6	
Foaming condition	AC = 12% $T = 470$ K $t = 5$ min	AC = 12% $T = 470$ K $t = 5$ min	AC = 12% $T = 470$ K $t = 5$ min	AC = 12% $T = 470$ K $t = 5$ min	AC = 12% $T = 470$ K $t = 5$ min	AC = 12% $T = 470$ K $t = 5$ min	Unfoamed
Cell size (μm)	58	43	36	43	86	0	
Dielectric constant (50 Hz)	1.76	1.09	1.49	1.67	1.97	3.20	
Dielectric loss (50 Hz)	4.5×10^{-3}	1.2×10^{-3}	2.6×10^{-3}	2.4×10^{-3}	4.1×10^{-3}	5.0×10^{-3}	
Surface resistivity (Ω)	2.90×10^{13}	2.94×10^{13}	6.91×10^{12}	2.11×10^{13}	3.29×10^{13}	1.74×10^{12}	
Dry dielectric strength (kV/mm)	86.6	84.2	44.3	45.7	74.2	93.3	
Wet dielectric strength (kV/mm)	89.4	87.4	47.9	37.1	74.2	83.3	

Note: volume resistivity for unfoamed PET sample is $5.5 \times 10^{13} \Omega\cdot\text{cm}$.

because its heat-producing capacity is low. Table I shows that the microcellular PET has higher surface resistivity than unfoamed PET. Its volume resistivity has not been obtained because of the instrument limitation. However, the volume resistivity of air is $1.6 \times 10^{17} \Omega\cdot\text{cm}$ at room temperature, and the unfoamed PET is $5.5 \times 10^{13} \Omega\cdot\text{cm}$ from measurement; hence, it is reasonable to think that the volume resistivity of microcellular PET should be higher than the unfoamed PET; i.e., the microcellular PET has better insulation property. After microcellular processing, the dry and wet dielectric strength of PET also changed as listed in Table I.

CONCLUSION

A new method, nonisothermal foaming, was used to prepare microcellular PET thin sheet foam in a conventional hydraulic press. Compared with isothermal foaming, nonisothermal foaming results in more uniform cell size microcellular PET foam due to controlling the foaming agent decomposition rate and providing nearly constant gas in a relative long foaming time. In addition, the nonisothermal foaming widens the foaming temperature and foaming time processing windows, which the cell size and cell density vary slightly in a relatively wide processing window. In nonisothermal foaming processing, the cell size is smaller even at a higher foaming temperature and higher foaming agent content. In isothermal foaming processing, the foaming temperature, time, and foaming agent content have greater effects on the cell size, cell density, and relative density. After microcellular processing, the dielectric constant and dielectric loss of PET decreases, the electric resistivity increases, and the dielectric strength changes as well.

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