

Polystyrene Foams. III. Structure–Tensile Properties Relationships

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ABSTRACT: In the previous two parts of this series of articles, the relations among the foaming conditions, microstructure, and impact properties of expanded microcellular polystyrene (EPS) were discussed. In this article, the effects of the foaming conditions and structure on the tensile properties of EPS were investigated. A systematic investigation was performed based on a statistical experimental design. Various processing conditions were used and a wide range of cellular structures was developed. Regression analysis was conducted on the data and expressions were developed to quantify the relationship between the tensile properties and the cellular structure. *Foaming time* and *foaming temper-*

ature were the most important processing parameters influencing the tensile modulus and strength. The tensile modulus and strength increased with an increasing foam density, but they decreased slightly when the cell size increased. Two different approaches were used to develop models relating the modulus to the foam density of EPS. Both models fitted the experimental data quite well. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 90: 1427–1434, 2003

Key words: polystyrene; mechanical properties; structure-property relations

INTRODUCTION

Previously, the relations among foaming conditions, structure, and impact properties of expanded microcellular polystyrene (EPS) made using CO₂ as a physical blowing agent were explored.^{1,2} It was shown that, by controlling the foaming conditions, a wide range of cellular structures could be produced. In particular, foam samples having the same foam density and different cell sizes were developed. Foaming time was found to be the most important factor determining the foam density, while saturation pressure was the most important factor controlling the cell size and cell density.

In general, foaming dramatically changes the properties of polymers. There is considerable literature on the mechanical properties of foams.^{3–7} So far, three approaches have been used to analyze the mechanical properties and behavior of cellular materials. Skochdopole and Rubens used phenomenological models to explain the compressive stress–strain behavior of closed-cell structures.⁸ Equations developed for com-

posite materials, such as the Nielsen, Halpin–Tsai, and Kerner equations, have also been widely used to analyze the mechanical properties of foams (e.g., see Throne⁹). In another approach, the load-deformation behavior of cellular materials was analyzed based on a micromechanical analysis of cell-wall deformation.

The mechanical properties of cellular polymers are basically influenced by properties of the base material (crystallinity, crosslinking, molecular orientation, etc.) and by cellular morphology. The *relative density*, *cell size*, *cell density*, and the *degree of openness* of the cells are the key structural parameters which control the properties. Mechanical properties are usually reported as a function of these structural parameters, or of processing parameters, such as foaming temperature and foaming time. In particular, since the foam density, ρ_r , can be measured easily, it is very common to report and analyze the mechanical properties of foams as a function of the foam density. Several empirical and theoretical relationships between mechanical properties and foam density have been established. The advantage of this approach is that the foam density can be related to the cellular structure and geometry of the foam.

Polymer foams are networks of solid struts and/or plates, which form the edges and faces of the cells of the foam. The cells may be closed or open. In an open-celled foam, the polymer is contained only in the edges and the cells are connected through open faces. In a closed-celled foam, the faces are solid and the cells are isolated from each other. These are ideal cases and

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polymer foams are usually partially open-celled or closed-celled.

Early studies by Martini¹⁰ showed that while a microcellular PS has a strength lower than that of the neat polymer its specific strength (strength-to-density ratio) was higher. Later, Waldman¹¹ showed a four-fold increase in the fracture toughness in microcellular high-impact polystyrene (HIPS) in comparison to neat plastic. A preliminary study of microcellular polyester composites was performed by Youn and Suh.¹² This study compared the flexural properties of a sheet-molding compound (SMC) and a microcellular composite with 42% lower density. The flexural strength of the foamed composite was found to be almost the same as that of the SMC. The specific strength was nearly 78% larger for the microcellular composite compared with the SMC, and the flexural toughness of the microcellular composite was three times that of the SMC.

Recently, Sun et al. studied the microcellular foaming and mechanical properties of polysulfones.¹³ They found increases in the tensile strength and modulus when the foam relative density increased, but did not report on the effect of cell size and cell density on the mechanical properties.

The tensile response of solid foams resembles that for honeycomb structures.⁵ There is an initial linear elastic deformation, which is caused by cell wall bending and stretching. At larger strains, the structure is geometrically nonlinear, as the cell walls rotate toward the tensile axis, increasing the stiffness. In brittle foams, a crack nucleates at a weak cell wall or preexisting flaw and propagates, giving a fast brittle fracture.⁵

The effects of the cell size on the mechanical properties of foamed materials have also been studied. These studies were conducted mainly on rigid foams. Brezny and Green¹⁴ investigated the effect of the cell size on the mechanical behavior of cellular reticulated vitreous carbon, made by carbonizing an open-cell foamed polymer. In their work, the samples contained cells in the range of 0.4–4.5 mm and the elastic modulus was found to be independent of the cell size. Hagiwara and Green¹⁵ observed an increase in the elastic modulus with an increasing cell size in an open-cell alumina foam. This behavior was in terms of the differences in the macrostructural variations caused by changing the cell size range. A similar observation was made by Dam et al.¹⁶ with open-cell ceramic foam and was explained in terms of microstructural variations with the cell size. Work by Morgan et al.¹⁷ on closed-cell cellular glass showed that the elastic modulus was independent of the cell size in that material. Kumar and Weller also examined the effect of the cell size on the tensile behavior of microcellular polycarbonate (PC).¹⁸ It was determined that the tensile behavior of microcellular PC was not af-

ected by the average cell size, for the range of sizes studied. They found the bulk foam density as the only variable determining the tensile strength. A number of other authors have tried to relate tensile and compression properties of foams to the foam relative density and properties of the foam matrix.^{19–24}

The objective of this work was to relate the tensile properties of EPS to the foaming conditions and structural parameters. Samples of polystyrene (PS) sheets were saturated with CO₂ at room temperature and relatively high pressure and expanded by heating the saturated samples after releasing pressure. Standard samples were cut from expanded sheets and their tensile properties were examined.

EXPERIMENTAL

The details of the materials, foaming process, and sample preparation were explained in part I of this series of articles.¹ Specimens for tensile testing were prepared by cutting compression-molded sheets. Tensile tests were performed on a Sintech Model 20 testing machine according to ASTM standard method D-638, using specimen type M-I. The drawing speed was 5.08 mm/min. Tensile properties and statistical data were calculated by the Testworks program (version 2.10, Sintech Inc., USA). For each sample, five to eight specimens were tested.

The experiments were performed based on a statistical experimental design. In this design, the processing variables were the *saturation pressure*, the *foaming temperature*, and the *foaming time*. The large number of samples required for a classical factorial design suggested the use of a central composite design plan. Table I presents the design for three variables at three levels. Four replicates were used to estimate the experimental error. The regression model relating the response y (representing tensile strength or tensile modulus) to x_1 (representing the *pressure*), x_2 (representing the *foaming temperature*), and x_3 (representing the *foaming time*) that is supported by this design is

$$y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_3x_3 + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \beta_{23}x_2x_3 + \beta_{11}x_1^2 + \beta_{22}x_2^2 + \beta_{33}x_3^2 + \varepsilon$$

RESULTS AND DISCUSSION

The effects of the processing parameters and the structures of the resulting foams on the elastic moduli and tensile strengths of EPS are reported below. Regression analyses were performed on the experimental data to find the effects of the processing variables, including the *saturation pressure* (P), *foaming temperature* (T), and *foaming time* (t) on the tensile modulus and tensile strength.

TABLE I
Design Matrix for Three Variables at Three Levels Based on Central Composite and Data
(Replicates Are Designated with an Asterisk)

Sample no.	Pressure (MPa)	Temperature (°C)	Time (s)	Relative density	Cell size (μm)	Tensile modulus (MPa)	Tensile strength (MPa)
13	3	105	10	0.5663	92	380	12.90
17*	3	105	10	0.5547	81	345	12.58
6	3	105	30	0.2414	163	129	2.81
9	3	110	20	0.2107	118	151	3.76
14	3	120	10	0.2424	27	250	5.85
12	3	120	30	0.1275	341	50	2.28
7	4.5	105	20	0.2911	74	268	5.71
5	4.5	110	10	0.3809	77	243	5.95
3	4.5	110	20	0.1836	112	115	2.68
16*	4.5	110	20	0.1745	114	118	2.70
8	4.5	110	30	0.1098	132	60	1.88
19*	4.5	110	30	0.1133	148	62	1.69
2	4.5	120	20	0.1212	145	99	2.60
4*	4.5	120	20	0.128	133	99	2.63
1	6	105	10	0.3361	3	197	5.01
15	6	105	30	0.0952	29	125	2.69
18	6	110	20	0.1004	15	123	2.99
11	6	120	10	0.1927	23	106	3.38
10	6	120	30	0.0318	57	33	0.33

The behavior of foam in tension is important in many engineering applications. Figure 1 shows stress-strain curves of neat PS and EPS, saturated at a pressure of 4.5 MPa and foamed at 110°C for 10 s, which produced a foam with a relative density equal to 0.38. In this plot, the specific stress-strain curve of EPS is also shown as a dotted line. Specific properties are obtained by dividing the property by the fraction of the foam volume occupied by the solid polymer: the relative density. The stress-strain curves showed an initial period of linearity followed by a change in slope. For PS, this occurred at about 2.5% strain, while EPS showed a change in slope at a strain of 1%. In the

second region, the stress-strain relationship was non-linear and all specimens failed by fast fracture. The ultimate strength and modulus of elasticity of all EPS samples were less than those of unfoamed PS. However, the EPS specimens showed greater elongation at yield and at break, because the cell walls of the foam undergo both bending and axial deformations. The slope of the stress-strain curve (proportional to the modulus) in closed-cell foams is determined mainly by edge bending, face stretching, and pressure of the gas in the cells.⁵

Tensile modulus

The dependence of the tensile modulus on the processing parameters (foaming time, foaming temperature, and saturation pressure) and on the structural parameters (including foam density, cell size, and cell density) were studied. The results of the tensile modulus measurements are given in Table I. Analysis of variance showed that six terms were significant with coefficient confidence levels of more than 95%. Foaming time and foaming temperature were found to be the most important parameters controlling the tensile modulus, while saturation pressure was found to be less important. The fitted regression model for tensile modulus (*E*) is

$$E = 1706.1 - 41.3P - 10.8T - 10.0t + 85.0\bar{T}^2 + 68.7\bar{P}\bar{t} \quad (5)$$

where $\bar{P} = (P - 4.5)/1.5$, $\bar{T} = (T - 112.5)/7.5$, and $\bar{t} = (t - 20)/10$ are scaled forms of *P*, *T*, and *t*, respec-

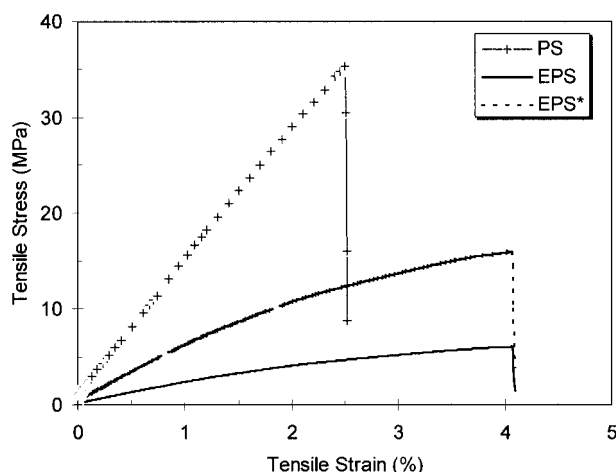


Figure 1 Uniaxial tensile stress-strain curves for PS and EPS with a relative density of 0.38. For the foam, both stress and the specific stress (designated by a star) are shown. The unit for specific stress is MPa cm³ g⁻¹.

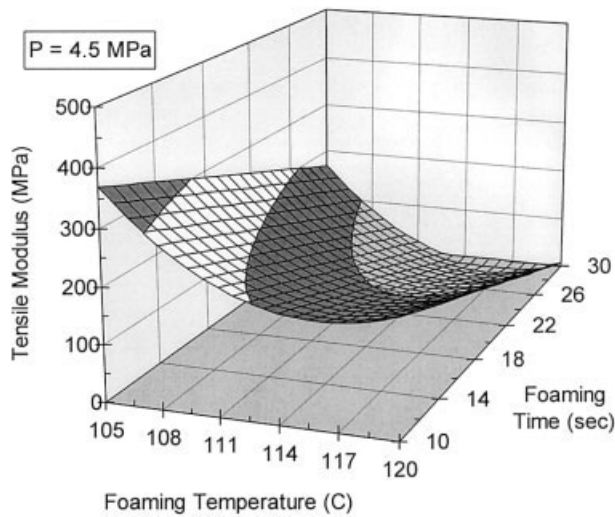


Figure 2 Dependence of tensile modulus on foaming conditions. The plotted surface represents the fitted regression equation [eq. (5)].

tively. This equation is plotted in Figure 2 against foaming conditions.

The specific tensile moduli of EPS (E_{sp}) were also statistically analyzed and eq. (6) was obtained:

$$E_{sp} = 10^5 \times (58.6 - 10.9P + 1.1T + 25.9\bar{t}^2 - 54.1\overline{Pt})^{-1} \quad (6)$$

In this analysis, the saturation pressure was found to be the most important parameter determining the specific tensile modulus, followed by the foaming temperature. The foaming time was not found to be significant, but the term $P \times t$, which represents the interaction between the saturation pressure and the foaming time, did have a significant effect on the specific tensile modulus.

Figure 2 demonstrates how the tensile modulus varies with the foaming conditions. While the tensile modulus was reduced at higher foaming times, the specific modulus did not significantly change. In other words, the decrease in the modulus as a result of foaming resulted entirely from a decrease in the foam density.

As the foaming temperature increased, the modulus showed an initial decrease and then leveled off. The specific tensile modulus slightly decreased at elevated foaming temperatures. This behavior can be attributed to a decrease in the foam density when the foaming temperature increased. While there was a decrease in the modulus with an increasing saturation pressure, the specific modulus showed two different trends, slightly decreasing at shorter foaming times and increasing at longer foaming times. It can be seen that increasing the saturation pressure, which corresponds

to more expansion and a decrease in foam density, decreases the tensile modulus.

The differences between the tensile modulus and the specific modulus and their variation under different foaming conditions can be explained by analyzing the foam structures. In the foam literature, mechanical properties are often related to the relative density using a simple empirical equation: *mechanical property* $\propto A\rho_r^n$. The constant A and the exponent n are generally determined empirically. The coefficient A is primarily related to the properties of the foam material, whereas n depends on the deformation mode. The exponent n typically lies between 1 and 2. The value of n for rigid foams, such as EPS, was found by various researchers to be approximately 2 (see, e.g., refs. 3 and 5).

Figure 3 illustrates the plot of the relative modulus of EPS versus the relative density. The two theoretical models in Figure 3 will be discussed later in the text. A simple polynomial regression curve fitting (with an R^2 value of 0.922) relates the relative modulus (E_r) to the foam relative density (ρ_r):

$$E_r = 0.58\rho_r^2 + 0.39\rho_r \quad (7)$$

From the plots of the modulus versus foam density and the cell size, it can be concluded that both of these variables influence the modulus. It is possible to select a subset of data where only one of these variables is changing while the other is held relatively constant. The values of the relative modulus of the foam specimens having similar cell sizes versus the foam density are shown graphically in Figure 4. The relative moduli of these samples follow the same trend as that observed for the entire data set, shown in Figure 3. Furthermore, the specific modulus of samples with

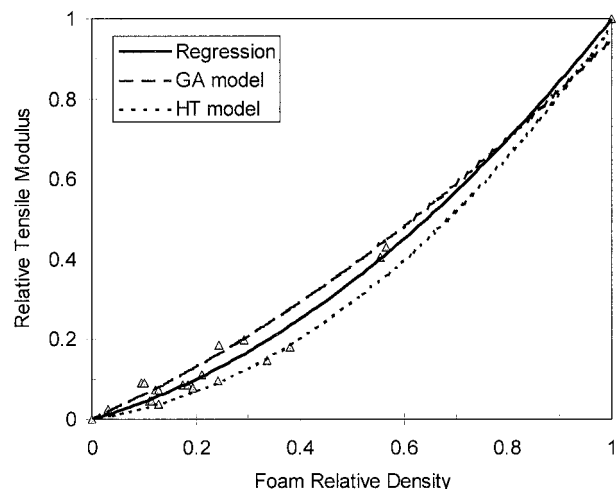


Figure 3 Relative tensile modulus of EPS versus relative density. The dotted line, dashed line, and solid line curves represent the HT model [eq. (12)], GA model [eq. (16)], and regression equation [eq. (7)], respectively.

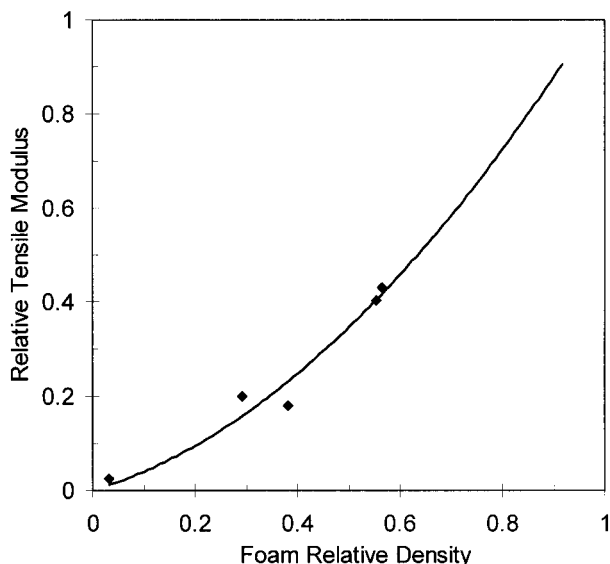


Figure 4 Relative modulus versus foam density of EPS samples with similar cell sizes.

similar cell sizes did not show any significant change with the density in the range of the foam density examined.

The same procedure was followed to construct plots of the modulus of samples having the same densities but differing cell sizes (Fig. 5). In this case, the modulus was found to decrease as the cell size increased. The cause of this scaling effect is not obvious but may have to do with strain-rate effects during cell-wall formation. It is known that if a sample of an amorphous or a semicrystalline polymer is heated to a temperature above its T_g and then subjected to large tensile strains, the molecules will tend to align themselves in the loading direction. If the polymer is then cooled below its T_g , while the molecules are still under stress, the molecules will become frozen in an oriented state. Such orientation can have significant effects on the properties of the polymer. If a filament of PS is heated, stretched, and frozen, a thinner filament will be produced with aligned molecules and a tensile strength as much as five times that of the unoriented PS.²⁵ During the foaming process, cell walls are stretched biaxially, and this would lead to molecular orientation and an elevated modulus in all directions in the plane of the cell wall. The degree of orientation depends on the strain rate and the cooling rate and thus may be related to cell size.

Work by Brezny and Green¹⁴ on the effect of the cell size on the mechanical behavior of cellular materials showed results different than those described by Figure 4. The material selected for that work was reticulated vitreous carbon, made by carbonizing an open-cell foamed polymer, thereby converting it to a glassy carbon with an entirely open cell structure. In their work, the samples contained cells in the range of

0.4–4.5 mm, and the elastic modulus was found to be independent of the cell size. It should be noted that the properties of solid foams strongly depend on the structure and the properties of the matrix material. For instance, the results obtained from brittle foams or foams with open cells cannot be generalized to foams of ductile materials or foams with closed cells.

A quadratic equation was found to fit the tensile modulus data versus foam density quite well. The proposed equation to relate the tensile modulus to the foam density does not take into account the cell geometry and orientation. During the foaming process, the bubbles expand in all directions, but, due to differences in the resistance against expansion induced by mold constraint, the bubbles tend to orient in preferred directions, transverse to the specimen. A similar correlation was found for the relationship between the tensile modulus of polysulfone microcellular foams and the foam density.¹³

A simple model was developed by Mehta and Colomb²⁶ using a modified version of the Halpin–Tsai (HT) equation [eq. (8)] (also known as the Kerner equation), in which the geometry and orientation of the bubbles were considered. Initially, the modified HT equation was presented for the prediction of a composite's elastic moduli given the properties of the filler and matrix.²⁷ The HT equation is actually a modification of the rule of mixtures in which two empirical constants to account for fiber/matrix interactions have been included²⁸:

$$E_c/E_m = (1 + ABV_f)/(1 - BV_f) \quad (8)$$

where E_c and E_m are moduli of the composite and matrix; A , a constant and a function of the filler's geometry; and V_f , the volume fraction of the inclusion, and B is related to the relative moduli of the filler and polymer and is defined as

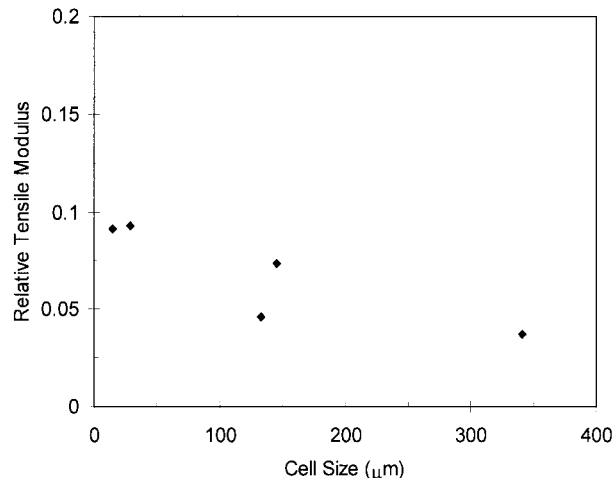


Figure 5 Tensile modulus versus cell size of EPS samples with similar foam density.

$$B = (E_f/E_m - 1)/(E_f/E_m + A)$$

where E_f is the modulus of the filler. In an expanded polymer, the bubbles were treated as particles with a stiffness or modulus of zero. Therefore, by substituting zero for E_f , the HT equation yields

$$B = -1/A$$

Substituting B in eq. (8) gives

$$E_r = (1 - V_f)/(1 + V_f/A) = V_p/(1 + V_f/A) \quad (9)$$

where E_r is the relative modulus of the expanded polymer and V_p is the volume fraction of polymer phase, which corresponds to the foam relative density (i.e., $V_p = \rho_r$). Substituting in eq. (9) results in

$$E_r^{-1} = (-1/A) + (1 + 1/A)\rho_r^{-1} \quad (10)$$

Equation (10) gives the relative modulus of the foam as a function of the relative density. The structure factor A depends on the cell geometry and orientation—in other words, on the aspect ratio of the bubbles—and can be determined experimentally. Approximate values for A for expanded polymers with different densities were found⁹ to be in the range of 0.5–0.6. Equation (10) can be rewritten in another form:

$$E_r = A\rho_r/(1 + A - \rho_r) \quad (11)$$

To evaluate the factor A , values of E_r^{-1} were plotted against ρ_r^{-1} . The slope of the fitted line equals $(1 + 1/A)$, which results in a value of 0.55 for the factor A . Hence, the HT model leads to eq. (12):

$$E_r = 0.55\rho_r/(1.55 - \rho_r) \quad (12)$$

This equation was plotted along with the data points and a regression-fitted curve in Figure 3. Comparison of the curves reveals that the modified HT equation models the data quite adequately.

Gibson and Ashby⁵ used another approach and developed a theoretical model to relate the mechanical properties of cellular materials to their relative densities. The proposed model by Gibson and Ashby (GA model) does not provide any information on the dependence of the mechanical properties on the cell size. The GA model is based on a cubic unit cell in which the deformation is controlled by the bending of the individual struts within the unit cell. Using the cubic cell model, Gibson and Ashby proposed an expression to calculate relative density of low-density foams with open cells from the unit dimension n :

$$\rho_r = C_1(d/l)^2 \quad (13)$$

where d and l are the cell strut thickness and the strut length, respectively. The strut length is sometimes used to represent the cell size. A general expression of $C_i \rho_r^n$ was employed to connect the relative mechanical properties of open-cell foams to the relative density, where C_i is a geometric constant characteristic of the unit cell shape and n depends on the deformation mode of the struts. The elastic modulus was derived by considering the deflection and fracture of a centrally loaded beam:

$$E_r = C_2\rho_r^2 \quad (14)$$

where C_2 is a constant characteristic of the cell geometry. Curve fitting to the data of a wide variety of open-cell foams⁵ showed that $C_2 = 1$.

In expanded polymers, often, most of the cells are closed and are separated from each other by cell edges. Therefore, the model based on strut bending is not appropriate. Gibson and Ashby considered the contributions of cell edge bending and face stretching and proposed a more complicated model for closed-cell foams, eq. (15):

$$E_r = \Phi^2\rho_r^2 + (1 - \Phi)\rho_r \quad (15)$$

where $1 - \Phi$ is the fraction of the solid which is in the face and is a measure of the degree of the openness of the cells. Kumar and VanderWel suggested Φ to be proportional to the foam density.²⁹ Assuming this proportionality, we reach eq. (16):

$$E_r = \rho_r^4 - \rho_r^2 + \rho_r \quad (16)$$

This equation was plotted in Figure 3 to compare it with the regression-fitted curve and HT model. Comparing the GA and HT models with the data reveals that both models can represent the variation of the elastic modulus with the foam density. An important point for all of these models is that they consider only foam density and do not consider the contribution of cell size to the elastic modulus. In comparing the results of this work with investigations on other materials reveals that the dependence of the modulus on the cell size is a strong function of the material.

Tensile strength

The experimental design described earlier was also employed to analyze the effect of the foaming conditions on the tensile strength of EPS. The fitted regression equation for the tensile strength, which represents the relations between the tensile strength and the foaming conditions, is

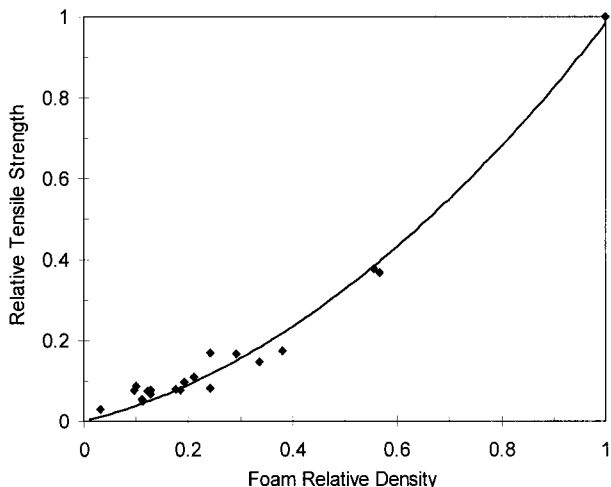


Figure 6 Relative tensile strength versus relative density of EPS.

$$\sigma_u = 33.79 - 0.97P - 0.18T - 0.25t + 1.37\overline{Pt} \quad (17)$$

In this equation, all terms with a percent confidence for significance greater than or equal to 95% were selected. The foaming time had the greatest influence on the tensile strength. The effect of saturation pressure was also statistically significant, but less important than the foaming time. The effect of the foaming temperature was less than that of the foaming time and saturation pressure.

Equation (18) was fit to the data for specific tensile strength versus foaming conditions. The model term ranking in the analysis of variance revealed that the specific tensile strength depended mainly on the saturation pressure:

$$\sigma_{u,sp} = 8.831 + 1.908P + 4.043\overline{P^2} + 5.380\overline{Pt} \quad (18)$$

While the tensile strength decreased significantly at longer foaming times, the change in the specific tensile strength was not significant. The foam's tensile strength decreased at a higher saturation pressure, due mainly to a drop in the foam density. However, the tensile strength normalized by mass increased at a higher saturation pressure. This may result from the finer structure (smaller cell size) generated at higher pressures. An increase in the foaming temperature also generated more expansion, which, as a result, caused a decrease in the tensile strengths of the foam specimens. The foaming temperature did not produce a consistent effect on the specific tensile strength.

The tensile strength of the polymer decreased as a result of foaming, since less solid material was available to bear the load. The specific tensile strengths did not change significantly and this observation leads to the conclusion that the quantity of the solid material is the main parameter determining the tensile strength.

As Figure 6 shows, the tensile strengths of the foam specimens with a wide range of densities increased with an increasing foam density. A quadratic expression fitted the relative tensile strength data with R^2 equal to 0.983:

$$\sigma_r = 0.66\rho_r^2 + 0.33\rho_r \quad (19)$$

When the cell size increased, both the tensile strength and the specific tensile strength slightly decreased. This behavior can be attributed to the greater difficulty in bending the cell walls and struts within the unit cell in the foams with a smaller cell size.

To separate the effects of the foam density and the cell size on the tensile strength, two subsets of data were extracted. The plots in Figure 7 show the influence of the foam density on the tensile strength for samples having similar cell sizes. These data show a similar trend to that observed for the whole data set (shown in Fig. 6). Equation (20) fitted the data points in Figure 7, which is very close to eq. (19). The R^2 value for this curve fitting was 0.973. The tensile strengths of samples having the same foam density were almost independent of the cell size (Fig. 8); however, there was a clear decrease in specific tensile strength as the cell size increased. This may be related to strain-rate effects and tensile drawing of the cell walls as discussed earlier:

$$\sigma_r = 0.62\rho_r^2 + 0.31\rho_r \quad (20)$$

The behavior of expanded polymers in tension and its dependence on the cellular structure is complex. Some explanations of this relationship have been reported. The behavior of expanded polymers in compression has recently received much more attention, since foam is more often used in compression. But the behavior of foam in tension, particularly at failure,

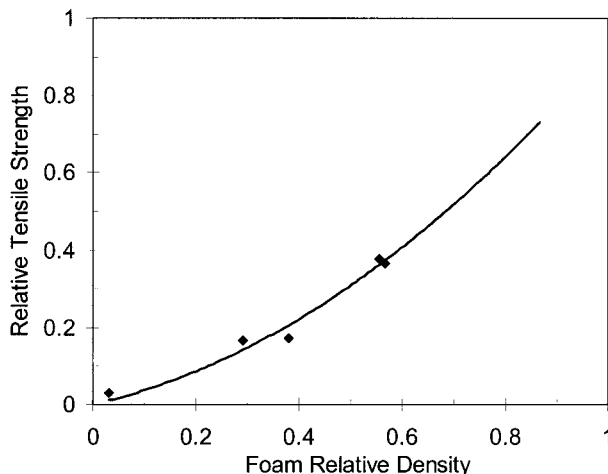


Figure 7 Relative tensile strength versus relative density of EPS samples having similar cell sizes.

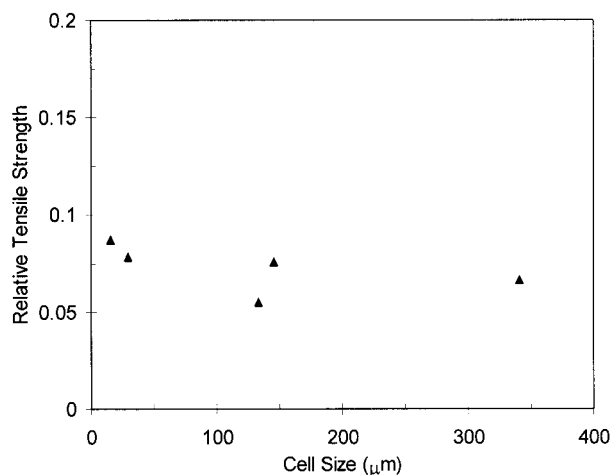


Figure 8 Relative tensile strength versus cell size of EPS samples having similar relative density.

cannot be deduced from studies of compression. An expanded polymer can be fractured rapidly by tensile loading, whereas compression generally causes progressive and slow crushing.

CONCLUSIONS

PS samples were expanded after saturating with CO₂ at pressures close to critical conditions. The tensile properties of the EPS samples were measured. Statistical analysis of the data showed that *foaming time* and *foaming temperature* play the most important roles in controlling the tensile modulus and strength. As expected, the tensile modulus and strength increased with an increasing relative density. The tensile modulus and strength of EPS are effectively determined by the foam density and only slightly decreased when the cell size increased. Equations derived from the HT and GA models fit the experimental modulus data quite well. The results of this work led to the conclusion that

the quantity of the solid material is the main factor determining the tensile strength.

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