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Anfu Guo*, Hui Li, Jie Xu, Jianfeng Li and Fangyi Li

Effect of microstructure on the properties of polystyrene microporous foaming material

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Abstract: The performance of Polystyrene microporous foaming (PS-MCF) materials is influenced by their microstructures. Therefore, it is essential for industrializing them to investigate the relationship between their microstructure and material properties. In this study, the relationship between the microstructure, compressive property, and thermal conductivity of the PS-MCF materials was studied systematically. The results show that the ideal foaming pressure of PS-MCF materials, obtaining compression performance, is around 20 MPa. In addition, the increase of temperature causes the decrease of sample density. It effects that the compression modulus and strength increase with the decrease of foaming temperature. Because the expansion rate and cell diameter of the PS-MCF materials reduce the thickness of cell wall, they are also negatively correlated with their mechanical properties. Moreover, there is a negative linear correlation between the thermal conductivity and cell rate, whereas the cell diameter is positively correlated with the thermal conductivity.

Keywords: PS-MCF; process conditions; microstructure; compressive property; thermal conductivity

1 Introduction

Microporous foaming polymers are a type of polymer foam materials with a small cell size (1–100 μm) and high density ($\geq 10^8$ cells/ cm^3) (1,2). In comparison with conventional foaming polymers, microporous foaming

polymers have many advantages, such as high impact strength, low thermal conductivity, long fatigue life and good thermal stability (3–6).

Polystyrene (PS) is a polymer with styrene as the monomer. PS is considered ideal owing to its good light transmittance, excellent electrical insulation and good chemical stability. However, PS has significant brittleness, low impact strength, poor heat resistance and stress cracking tendency (7). Because conventional PS foam materials have advantages, such as low weight, low thermal conductivity, low water absorption, good electric properties, thermal insulation, shock absorption and sound insulation, they are widely used in various fields, such as construction, refrigeration, heat insulation and cushion packaging (8). However, they have some disadvantages, such as poor permeability resistance, low impact strength and low recovery speed from high impact; moreover, the cells rupture easily (9). A PS foaming material is generally prepared using a fatty hydrocarbon or halogenated hydrocarbon foaming agent, and the preparation process is characterized by certain toxicity, safety hazard, low boiling point and environmental pollution. The foaming agent used in the preparation of polystyrene microporous foaming (PS-MCF) materials is supercritical CO_2 . This method saves material, improves product performance, and is environment friendly. PS-MCF materials have been applied to computer science, aerospace, bio-medical and other cutting-edge fields (10).

In-depth studies have been conducted on microporous foaming materials. Kumar et al. (11) experimentally studied the tensile properties of a polycarbonate microporous foam and found that its tensile strength is directly proportional to the foam density. Xing et al. (12) prepared microporous crosslinked polyethylene foam using radiation and supercritical CO_2 . Sun and Mark (13) studied the effect of cell density on the mechanical properties of a polysulfone foam by conducting a tensile test and showed that its tensile strength is directly proportional to the cell density. Fu et al. (14) studied the processing parameters, such as mechanical properties, of a polymethylmethacrylate

* **Corresponding author: Anfu Guo**, School of Mechanical and Automobile Engineering, Liaocheng University, Liaocheng 252059, China, email: guoanfu@lcu.edu.cn

Hui Li, School of Mechanical and Automobile Engineering, Liaocheng University, Liaocheng 252059, China

Jie Xu, Jianfeng Li and Fangyi Li, School of Mechanical Engineering, Shandong University, Jinan 250061, China.

(PMMA) microcellular foam. By adjusting the process parameters and studying the material tensile performance indices under different process parameters, they found changes in the capability index with the change in the microstructure of the material. A new prediction model was established and the accuracy of the model in predicting mechanical properties of PMMA foam prepared with different process parameters was verified. Matuana et al. (15) studied the impact and tensile properties of polylactic acid (PLA) microporous foaming materials and found significant improvement in their mechanical properties owing to the existence of the microporous structure in the foam. In comparison with unfoamed PLA, the impact resistance and fracture strain of the PLA microporous foam (with an expansion ratio of 2) doubled and the toughness increased by four times. Williams et al. (16) tested the thermal conductivity of polyimide-series foam materials (TEEK) under different pressures. Takagi et al. (17) studied the thermal conductivity of a polyimide foam and found that its thermal conductivity decreases with the decrease in temperature and pressure and increases with the increase in density. Wang et al. (18) prepared a highly porous PMMA microporous foam using supercritical CO₂ and found that there exists an optimal unit size and the thermal conductivity of the material is minimum at a certain porosity. Currently, the preparation of foaming materials liking PS-MCF materials has been get significant progress. But for industrializing them, the mechanism of the effect of micromorphology on the properties of microporous foamed polystyrene needs to be further studied.

The main objective of this study is to investigate the relationship between the microstructure of PS-MCF materials and their properties. PS-MCF samples with different microstructures were prepared by adjusting the process conditions, such that the influencing mechanisms of the material microstructure on the compressive property and thermal conductivity of the samples could be investigated systematically.

2 Experimental details

2.1 Material

General purpose, standard grade polystyrene was purchased from Chi Mei Corporation (Taiwan, China). Industrial grade CO₂ with a purity greater than 99.9% was purchased from Jiangsu Lianhai Biological Science limited company (Jiangsu, China).

2.2 Preparation of polystyrene samples

The PS-MCF samples with various microforms were prepared by the CO₂ pressure-lowering pressure method, in an autoclave with automated temperature control, according to individual test requirements. The materials, loaded into a foaming mold, were placed in the autoclave and the autoclave chamber was evacuated for 2 min using a vacuum pump. The autoclave was heated to the required foaming temperature and held at temperature for 10 min after reaching the set pressure. CO₂ was injected into the foaming mold and the samples were fully saturated for a certain period to form a PS/CO₂ homogeneous phase system. The pressure was then rapidly lowered to atmospheric pressure, and the samples were taken out cool and set.

2.3 Characterization of material microstructure

The samples were treated with liquid nitrogen embrittlement and then sprayed with gold. Finally, they were observed under a Quanta FEG 250 scanning electron microscope.

2.3.1 Calculation of cell diameter

The SEM images of the PS-MCF samples were analyzed using the software ImageJ. To ensure the accuracy of the results, the total number of cells in the image should exceed 100. The cell size is usually characterized in terms of the average cell diameter $\bar{D}$, which can be obtained as follows:

$$\bar{D} = \frac{\sum_{i=1}^n D_i}{n} \quad (1)$$

where n is the number of cells in an SEM image; D_i is the diameter of the cell (calculated to scale) in an SEM image, in μm ; $\bar{D}$ is the average cell diameter, in μm .

2.3.2 Calculation of expansion ratio

The apparent density refers to the density of the sample. The apparent density of samples was tested by using the drainage method supported by and electronic balance in GB/T1033-86. The expansion ratio is also called the volume expansion ratio and is used to calculate the volume ratio

of foamed and pre-foamed samples. Equation 2 is used to obtain the expansion ratio.

$$\varphi = \frac{V_f}{V_0} = \frac{\frac{1}{\rho_f}}{\frac{1}{\rho_0}} = \frac{\rho_0}{\rho_f} \quad (2)$$

where V_f is the volume of the sample after foaming, in cm^3 ; V_0 is the volume of the sample before foaming, in cm^3 ; ρ_0 is the density of the sample before foaming, in g/cm^3 ; ρ_f is the density of the sample after foaming, in g/cm^3 .

2.4 Characterization of material property

2.4.1 Compression property test

The PS-MCF samples were tested for compression property in accordance with the GB/T 8813-1988 standard “compression test method of hard foam plastics” using the HDW-2000 universal electronic tester. Parts of the foamed samples were rectangular in shape, with a length, width, and height of 10, 10, and 4 mm, respectively. A compression speed of 1 mm/min and compression amount of 2.5 mm were applied to the samples. There were eight groups of samples. Five samples were tested in each group, the average value was considered.

2.4.2 Thermal conductivity property test

The thermal conductivity of the materials was tested using the TPS 2500 thermal constant analyzer, the measurement range, temperature range and accuracy of which are 0.005-500 W/mK, 10-1000 K, and $\pm 3\%$, respectively. The transient plane source (TPS) method was used to measure the thermal conductivity of the Styrofoam. A disc temperature-dependent resistor was selected as the probe. The thickness of the probe was only 0.025 mm and it was placed at the center of the sample. The probe resistance decreases with the increase in the temperature of probe. It can be measured by the voltage and current of the probe that the thermal conductivity property of the sample.

The thermal conductivity measurement was conducted at room temperature (23°C) for 10 s. The output power was set to 0.005 W. Parts of the foamed samples were cut into two cylindrical shapes of diameter 13 mm and height 7 mm, while ensuring that the cutting surface was smooth. The probe was then placed between the gap created by the two shapes, and a clamp was used to press them down. Among the 180 data points displayed by the

measurement software, 150 data were selected for the thermal conductivity measurement. In order to ensure the accuracy of data and avoid excessive workload, the influence of contact resistance between the sensor and sample was reduced by removing the first 30 data points. The sample was placed under standard conditions for more than one month to ensure that air was the only gas in the Styrofoam, before measuring the thermal conductivity.

3 Results and discussion

3.1 Compressive performance analysis

3.1.1 Effects of foaming temperature and foaming pressure on compression performance

To explore the effect of the foaming temperature on the compressive performance of the PS-MCF materials, microporous foaming samples 1, 2, 3 and 4, as listed in Table 1, were selected for testing. The micromorphology parameters of the samples under different foaming conditions were listed in Table 1.

Figure 1a illustrates the compression stress-strain curves of the samples at different foaming temperatures. As shown in Figure 1a, the compression deformation process of the samples proceeds in three stages. Accordingly, the curve can be divided into three stages: a linearly elastic phase, uniform phase and compaction phase (19-22). Under the same strain, the corresponding stress decreases with the increase in foaming temperature.

Figure 1b illustrates the effects of the foaming temperature on the compression strength and modulus of the PS-MCF materials. As shown in Figure 1b, the compression strength and modulus increase as the

Table 1: Micromorphology parameters of the samples under different foaming conditions.

Sample	Conditions (temperature ($^\circ\text{C}$) – pressure (MPa))	Expansion ratio	Cell diameter (μm)
1	120–15	23.85	15.96
2	125–15	38.90	21.31
3	130–15	40.91	26.10
4	135–15	52.12	23.96
5	130–10	38.35	58.17
6	130–20	46.23	18.34
7	130–25	52.12	15.69

Figure 1: Effect of the microstructure on the properties of polystyrene microporous foaming material: (a) compression stress-strain curves of the samples at different foaming temperatures; (b) effects of the foaming temperature on the compression strength and modulus of the PS-MCF materials; (c) compression stress-strain curves of the samples under different foaming pressures; (d) effects of the foaming pressure on the compression strength and compression modulus of the PS-MCF materials; (e) compression stress-strain curves of the samples 1 and 7; (f) compression stress-strain curves of the samples 2 and 4; (g) compression stress-strain curves of the samples 3 and 6; (h) compression stress-strain curves of the samples 4 and 7.

foaming temperature gradually decreases from 135°C to 120°C, though the stiffnesses of the materials reduce. In the Gibson and Ashby theoretical model, the compressive elastic modulus and strength can be used for the analysis as a function of the relative density; here, these factors increase with the increase in the relative density (23-25). Considering that the sample density is significantly affected by the foaming temperature and decreases with the increase in the foaming temperature (26,27), the data are consistent with the above theoretical model.

To explore the effect of the foaming pressure on the compression performance of the PS-MCF materials, samples 3, 5, 6 and 7, as listed in Table 1, were selected for the compression performance analysis. Figure 1c shows the compression stress-strain curves of the samples under different foaming pressures. As shown in Figure 1c, the stress-strain curve changes irregularly with the foaming pressure. In addition, the sample density changes with the increase in the foaming pressure (28). However, as shown in Figure 1d, the compressive elastic modulus and compressive strength of the PS-MCF materials do not decrease with the decrease in the density. This is because the foaming pressure not only affects the relative density, but also the diameter of the cells. The decrease of cell diameter leads to the decrease of compression modulus and compression strength.

The analysis of the effect of pressure on the compressive properties of the samples shows that the effect of material microstructure on the mechanical properties is significant. In the Gibson-Ashby model, the analysis is performed only in terms of density, whereas the microstructure of the foaming material is not considered. To better understand the factors influencing the mechanical properties of foamed materials, it is necessary to study the influence of material microstructure on the properties.

3.1.2 Effects of expansion ratio on compressive performance

To explore the effect of the foaming ratio on the compressive performance, samples with a similar cell diameter but with different expansion ratios, i.e., microporous foaming samples 1, 7, 2 and 4, as listed in Table 1, were selected.

As shown in Figure 1e, in the compression stress-strain curves of samples 1 and 8, the stress value of the sample with a lower expansion ratio is greater when the strain is constant. The same conclusion can be drawn from Figure 1f. The stress values of samples 2 and 4 with a lower foaming ratio are greater. The results show that the compression strength of the sample with a lower expansion

ratio is greater when the cell diameter is constant. This is because the samples with a lower expansion ratio have thicker cell walls and foam skeletons.

3.1.3 Effect of cell diameter on compression performance

To explore the effect of the cell diameter on the compression performance, samples with a similar expansion ratio but with different cell diameters, i.e., microporous foaming samples 3, 6, 4 and 7, as listed in Table 1, were selected.

Figure 1g shows the compression stress-strain curves of samples 3 and 7. As shown in Figure 1g, when the strain is constant, the stress value of the sample with a smaller cell diameter is greater. The same conclusion can be drawn from Figure 1h. The stress values of samples 4 and 8, which have smaller cell diameters, are greater. Moreover, the compression strength of the sample with a smaller cell diameter but with a similar expansion ratio is greater.

3.2 Thermal conductivity analysis

3.2.1 Effects of expansion ratio on thermal conductivity

To explore the effects of expansion ratio on the thermal conductivity of PS-MCF materials, twelve groups of samples with different expansion ratio were prepared by adjusting the process conditions using the CO₂ pressure-lowering method. Each group of sample data is the average of ten material sample data. The thermal conductivity of them was tested, as listed in Table 2.

Figure 2 depicts the relationship between the thermal conductivity and expansion ratio of the PS-MCF samples.

Table 2: Relationship between the thermal conductivity and expansion ratio of the of PS-MCF materials.

sample	1	2	3	4	5	6
Expansion ratio	38.35	41.16	44.26	46.23	43.13	23.85
Thermal conductivity (mW/m·K)	28.1	26.8	24.2	24.6	23.7	35
sample	7	8	9	10	11	12
Expansion ratio	38.9	47.37	52.12	29.87	27.38	52.12
Thermal conductivity (mW/m·K)	25.3	23.2	22.3	31.8	30.7	22.7

Figure 2: Relationship between the thermal conductivity and expansion ratio of the PS-MCF materials.

Table 3: Thermal conductivity of the PS-MCF samples in the comparison groups.

Sample	Expansion ratio	Cell diameter (μm)	Heat conductivity coefficient ($\text{mW/m}\cdot\text{K}$)
1	a	46.23	18.34
	b	47.37	26.10
2	c	52.12	15.69
	d	52.12	23.96
3	e	38.35	58.17
	f	38.90	21.31
4	g	29.87	41.08
	h	27.38	13.79

As shown in Figure 2, the values were fitted and were found that the thermal conductivity of the material decreases linearly with the increase in the expansion ratio of the material. The thermal conductivity of the material is significantly affected by its expansion ratio. In the stage of low expansion ratio, the thermal conductivity of the composite is mainly affected by the solid polymer. However, as the expansion ratio and CO_2 content of the PS-MCF material increase, CO_2 affects its thermal conductivity. In addition, the thermal conductivity of CO_2 is lower than that of PS. Therefore, the thermal conductivity of the PS-MCF material decreases with the increase in the foaming ratio.

3.2.2 Effect of cell diameter on thermal conductivity

To explore the effect of cell size on the thermal conductivity of PS-MCF materials, samples with a similar expansion ratio but with different cell diameters were selected, as listed in Table 3. In addition, new samples with a similar

expansion ratio but with different cell diameters also were prepared for the analysis. Figure 3 illustrates the SEM images and process parameters of a section of the PS-MCF samples.

In Table 3, the samples in the comparison groups 1, 2, 3 and 4 exhibit a similar expansion ratio. But they have different cell diameters, as show in Figure 3. The analysis of the comparison group 1, listed in Table 2, shows that the expansion ratios of samples (a) and (b) are similar, cell diameter of sample (a) is lower than that of sample (b), and thermal conductivity of sample (a) is lower than that of sample (b). The same trends can be found in the other

Figure 3: SEM images of PS-MCF samples: (a) 130°C, 20 MPa, 1 h; (b) 130°C, 15 MPa, 1 h; (c) 130°C, 25 MPa, 1 h; (d) 135°C, 15 MPa, 1 h; (e) 130°C, 10 MPa, 1 h; (f) 125°C, 15 MPa, 1 h; (g) 130°C, 15 MPa, 2 h; (h) 125°C, 25 MPa, 1 h..

groups. The thermal conductivities of the samples with a higher cell diameter are greater. In the comparison group 3, the difference in the cell diameters of samples (e) and (f) is significant, the same applies to the corresponding thermal conductivity. The greater the difference in the cell diameter, the greater is the difference in the thermal conductivity. In the comparison group 4, the expansion ratio of sample (g) is slightly greater than that of sample (h), whereas the cell diameter of sample (g) is significantly greater than that of sample (h). This is because the cell diameter of the material varies significantly, and the influence of the cell diameter on the thermal conductivity is greater than that of the expansion ratio. These factors reduce the effect of the expansion ratio. This is evident in the fact that the thermal conductivity of sample (g) is greater than that of sample (h).

For materials with the same expansion ratio, the smaller the cell diameter, the greater is the number of cells and the greater is the amount of heat absorbed and scattered by the cell wall during the heat transfer process. The cell diameter of the material affects the thickness of the foam skeleton and amount of cell wall. In the process of heat conduction between solid and gas, the cell wall and foam skeleton absorb or scatter some energy and consequently affect the thermal conductivity of the foam material. This is the reason for the low thermal conductivity of the material.

4 Conclusion

In this study, PS-MCF samples with different microforms were prepared by adjusting the process conditions. The relationship between the microform and material properties was studied. The results show that the thickness of cell wall and foam skeleton is import factor determining the compressive strength of PS-MCF materials. As a result, the increase of expansion ratio or cell diameter both reduced the compression strength of PS-MCF materials. A negative linear correlation was observed between the material expansion ratio and thermal conductivity. As the cell diameter of PS-MCF materials with the same expansion ratio increases, the number of cells increases and thermal conductivity decreases.

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