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Cell Morphology and Mechanical Properties of Microcellular Mucell® Injection Molded Polyetherimide and Polyetherimide/Fillers Composite Foams

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ABSTRACT: Microcellular polyetherimide (PEI) foams were prepared by microcellular injection molding using supercritical nitrogen ($SC\text{-}N_2$) as foaming agent. The effects of four different processing parameters including shot size, injection speed, $SC\text{-}N_2$ content, and mold temperature on cell morphology and material properties were studied. Meanwhile, multiwalled carbon nanotube (MWCNT), nano-montmorillonoid (NMMT), and talcum powder (Talc) were introduced into PEI matrix as heterogeneous nucleation agents in order to further improve the cell morphology and mechanical properties of microcellular PEI foams. The results showed that the processing parameters had great influence on cell morphology. The lowest cell size can reach to 18.2 μm by optimizing the parameters of microcellular injection molding. Moreover, MWCNT can remarkably improve the cell morphology of microcellular PEI foams. It was worth mentioning that when the MWCNT content was 1 wt %, the microcellular PEI/MWCNT foams displayed optimum mechanical properties and the cell size decreased by 28.3% compared with microcellular PEI foams prepared by the same processing parameters. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 130: 4171–4181, 2013

KEYWORDS: foams; nanoparticles; nanowires and nanocrystals; mechanical properties; thermal properties

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INTRODUCTION

Microcellular polymers are materials characterized by a cell density in the range of 10^9-10^{15} cells/cm^3 and cell size in the range of 0.1–10 μm.[1–4] The microcellular foams have high strength-to-weight ratio, excellent heat and acoustic insulations, high energy or mass absorption, and materials saving[5] which result in the development of the microcellular foaming techniques such as batch foaming,[6,7] thermoforming, continuous filament and sheet extrusion,[8,9] and injection molding.[10] However, most of the foaming processes of polymer mentioned above cannot apply for industrial scale which means they have no commercial value. Moreover, the microcellular injection molding exhibits a number of superiorities such as outstanding dimensional accuracy, shortening the molding cycle, and reducing the warping and residual stress of products,[11–17] which has been widely used in many industries.[18] Therefore, numerous studies have been undertaken to investigate the microcellular foams by injection molding such as polyethylene, polypropylene, polystyrene, polyamide-6, and polylactide.[19–23] These literatures of microcellular foams mainly have been focused on the conventional thermoplastics. However, very little attention is paid to high-performance microcellular foams.

Polyetherimide (PEI), one of the most important high-performance engineering thermoplastics, is extensively used in commercial applications because of exhibiting excellent thermal stability, mechanical properties, electrical insulating property, wear-resisting property, dimensional stability, and flame resistance, etc. However, the current study on microcellular PEI foams mainly focused on the 150 μm–1.5 mm thick sheets of PEI by batch foaming.[24–27] There is a lack of articles on the research of microcellular PEI foams by microcellular injection molding.

In this article, a series of microcellular PEI foams were prepared by microcellular injection molding using supercritical nitrogen ($SC\text{-}N_2$) as foaming agent. The effects of processing parameters on cell morphology and material properties were studied. Drawing lessons from batch foaming,[28–30] the fillers were introduced into PEI matrix as heterogeneous agents in order to further improve the cell morphology and mechanical properties of microcellular PEI foams.

Table I. Processing Parameters of Microcellular Injection Molding

Trial for pure PEI	Shot size (mm)	Content of SC-N_2 (wt %)	Injection speed (mm/s)	Mold temperature (°C)
1	49	0	20	120
2	49	0.2	20	120
3	49	0.4	20	120
4	49	0.6	20	120
5	49	0.8	20	120
6	43	0.4	20	120
7	45	0.4	20	120
8	47	0.4	20	120
9	51	0.4	20	120
10	49	0.4	40	120
11	49	0.4	60	120
12	49	0.4	80	120
13	49	0.4	100	120
14	49	0.4	20	80
15	49	0.4	20	100
16	49	0.4	20	140
17	49	0.4	20	160
18	49	0.4	20	180

EXPERIMENTAL

Materials

PEI (Ultem 1100) was supplied by SABIC, Saudi Arabia. The density of amorphous PEI was 1.24 g/cm^3. Multiwalled carbon nanotube (MWCNT) (TNIM6, *L*/*D* was about 5000/3) was supplied by Chengdu Organic Chemicals Co., Ltd., China. Nanomontmorillonoid (NMMT) (DK 2, the diameter-thickness ratio was about 200) was supplied by Zhejiang Fenghong New Materials Co., Ltd., China. Talcum powder (Talc) powder (2500, the particle size was about 5 μm) was supplied by Guilin Guiguang Talc Developments Co., Ltd., China.

PEI Composites Preparation

PEI composites were processed in a PTW252 twin-screw extruder (HAAKE, Germany) to give samples. The rotational speed of extruder was 90 rpm, and the temperatures of its eight sections, from the charging hole to the ram head, were 340, 350, 355, 355, 360, 360, 340, and 350°C. All samples were dried at 140°C for 4 h to remove residual moisture.

Microcellular Foams Preparation

Microcellular PEI foams were prepared by an A VC 330H/80L injection molding machine (ENGEL, Austria) using SC-N_2 as foaming agent. The supercritical system was SII-TR-10 model (TREXEL, USA).

Characterization

Foam Density. The foam density was performed according to ISO 1183-1:2004, and each measurement was repeated at least five times. The density of samples was determined from eq. (1):

$$\rho = \frac{W_a \cdot \rho_w}{W_a - W_w} \quad (1)$$

where ρ_w is the density of samples in the water, W_a and W_w are the weight of samples in the air and water, respectively.

SEM Observations. Samples were cryogenically fractured in liquid nitrogen and observed with an Apollo 300 scanning electron microscope (SEM). The cell density (N_f), the number of cells per cubic centimeter of solid polymer, was determined from eq. (2)[31,32]:

$$N_f = \left(\frac{nM^2}{A}\right)^{\frac{3}{2}} \left(\frac{1}{1-V_f}\right) \quad (2)$$

where n is the number of cells on the SEM micrograph, M is the magnification factor, A is the area of the micrograph (cm^2),

Table II. Effect of Shot Size on Cell Morphology of Microcellular PEI Foams

Trial for pure PEI	Shot size (mm)	Mass density (g/cm^3)	Weight reduction (%)	Cell density (cell/cm^3)	Cell size (μm)
6	43	1.13	8.6	4.6×10^8	36.1
7	45	1.14	8.2	9.7×10^8	28.0
8	47	1.17	6.0	2.3×10^9	23.5
3	49	1.18	5.0	2.9×10^9	23.3
9	51	1.19	4.1	6.3×10^9	19.2

Table III. Effect of Injection Speed on Cell Morphology of Microcellular PEI Foams

Trial for pure PEI	Injection speed (mm/s)	Mass density (g/cm^3)	Weight reduction (%)	Cell density (cell/cm^3)	Cell size (μm)
3	20	1.18	5.0	2.9×10^9	23.3
10	40	1.18	4.8	3.1×10^9	23.0
11	60	1.19	4.4	3.5×10^9	22.7
12	80	1.19	4.0	4.4×10^9	21.8
13	100	1.20	3.6	8.4×10^9	18.2

Table IV. Effect of SC-N_2 on Cell Morphology of Microcellular PEI Foams

Trial for pure PEI	Content of SC-N_2 (wt %)	Mass density (g/cm^3)	Weight reduction (%)	Cell density (cell/cm^3)	Cell size (μm)
1	0	1.24	-	-	-
2	0.2	1.19	4.0	6.7×10^9	19.1
3	0.4	1.18	5.0	2.9×10^9	23.3
4	0.6	1.17	6.0	3.7×10^8	43.0
5	0.8	1.16	6.1	2.9×10^8	46.8

Table V. Effect of Mold Temperature on Cell Morphology of Microcellular PEI Foams

Trial for pure PEI	Mould temperature (°C)	Mass density (g/cm^3)	Weight reduction (%)	Cell density (cell/cm^3)	Cell size (μm)
14	80	1.16	6.3	1.8×10^9	24.9
15	100	1.17	6.0	2.2×10^9	24.0
3	120	1.18	5.0	2.9×10^9	23.3
16	140	1.18	4.7	5.6×10^9	19.1
17	160	1.20	3.1	9.5×10^9	18.5
18	180	1.13	8.6	6.5×10^8	31.5

and V_f is the void fraction of the foamed sample, which can be estimated as

$$V_f = 1 - \frac{\rho_f}{\rho} \tag{3}$$

where ρ is the density of solid PEI and ρ_f is the density of microcellular PEI foams. From eqs. (2) and (3), the cell size, d, can be estimated as[33]

$$d = \sqrt[3]{\frac{6V_f}{\pi N_f (1 - V_f)}} \tag{4}$$

Mechanical Properties. The tensile and flexural properties of samples were carried out with a CMT 7015 material test instrument (SUNS, China) according to ISO 527-5:1997 and ISO 178:1993, relatively. The crosshead speed for both tensile and flexural measurements was 5 mm/min. The testing of notched

Table VI. Effect of Fillers on Cell Morphology of Microcellular PEI/Fillers Foams

Fillers	Mass density (g/cm^3)		Weight reduction (%)	Cell density (cell/cm^3)	Cell size (μm)
	Solid	Foam			
-	1.24	1.18	5.0	2.9×10^9	23.3
NMMT	1.26	1.21	4.0	1.7×10^9	29.7
Talc	1.25	1.19	4.9	3.9×10^9	21.2
MWCNT	1.26	1.20	4.9	9.7×10^9	16.7

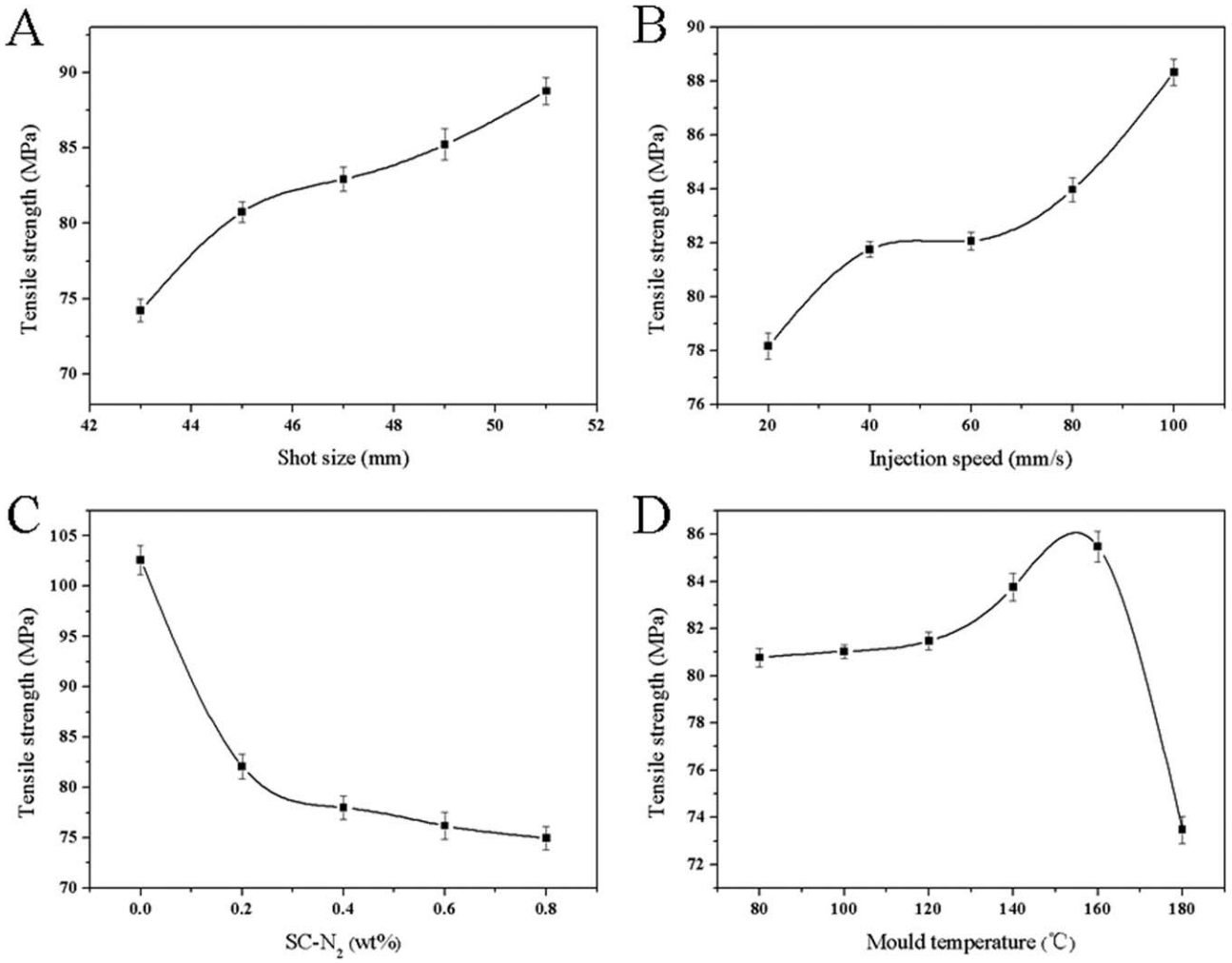

Figure 1. Effects of process parameters on tensile strength of microcellular PEI foams.

Charpy impact of samples was carried out with a PTM 1100 impact testing machine (SUNS, China) according to ISO 179-1:2000. All tests were carried out in air-conditioned room (25°C).

Electrical Properties. The dielectric constant of samples was carried out with a 4292A precision impedance analyzer (Agilent, USA), equipped with 16451B dielectric test fixture. B-type electrode was used for contacting electrode method. The frequency range was 50Hz–30 MHz and the environment temperature was 25°C.

Thermal Properties. The thermal conductivity of samples was carried out with an LFA447 laser thermal analyzer (Netzsch, Germany) following the ASTM E1461–2001 standard. The size of samples was Φ12.7 × 2 mm^2, and the environment temperature was 25°C.

RESULTS AND DISCUSSION

Influence of Processing Parameters on Cell Morphology and Material Properties

Cell Morphology. All the processing parameters of microcellular injection molding are shown in Table I. In Tables (II–V), all the processing parameters had major influence on cell morphology. As the shot size increased, the weight reduction and cell size decreased, but the cell density increased. It was probably because the content of PEI fusant into mold cavity increased with the growth of shot size, which led to enhancing the relative melt strength. So the frequency of cell coalescence decreased, resulting in the decrease of cell size.

The effect of injection speed exhibited the same tendency. The cell density and cell size of microcellular PEI foams increased from 2.9 × 10^9 cells/cm^3 to 8.4 × 10^9 cells/cm^3, and from 23.3 μm to 18.2 μm as the injection speed increased from 20 mm/s to 100 mm/s, respectively. This indicated that high injection speed can shorten the time of fusant into mold cavity, which was in favor of bubble fixation and reduced the probability of cell coalescence.

However, the influence of SC-N_2 content on cell morphology presented the opposite situation. When the SC-N_2 content dissolved in PEI fusant increased, the pressure of PEI/SC-N_2 homogeneous system raised. During the process of cell growth,

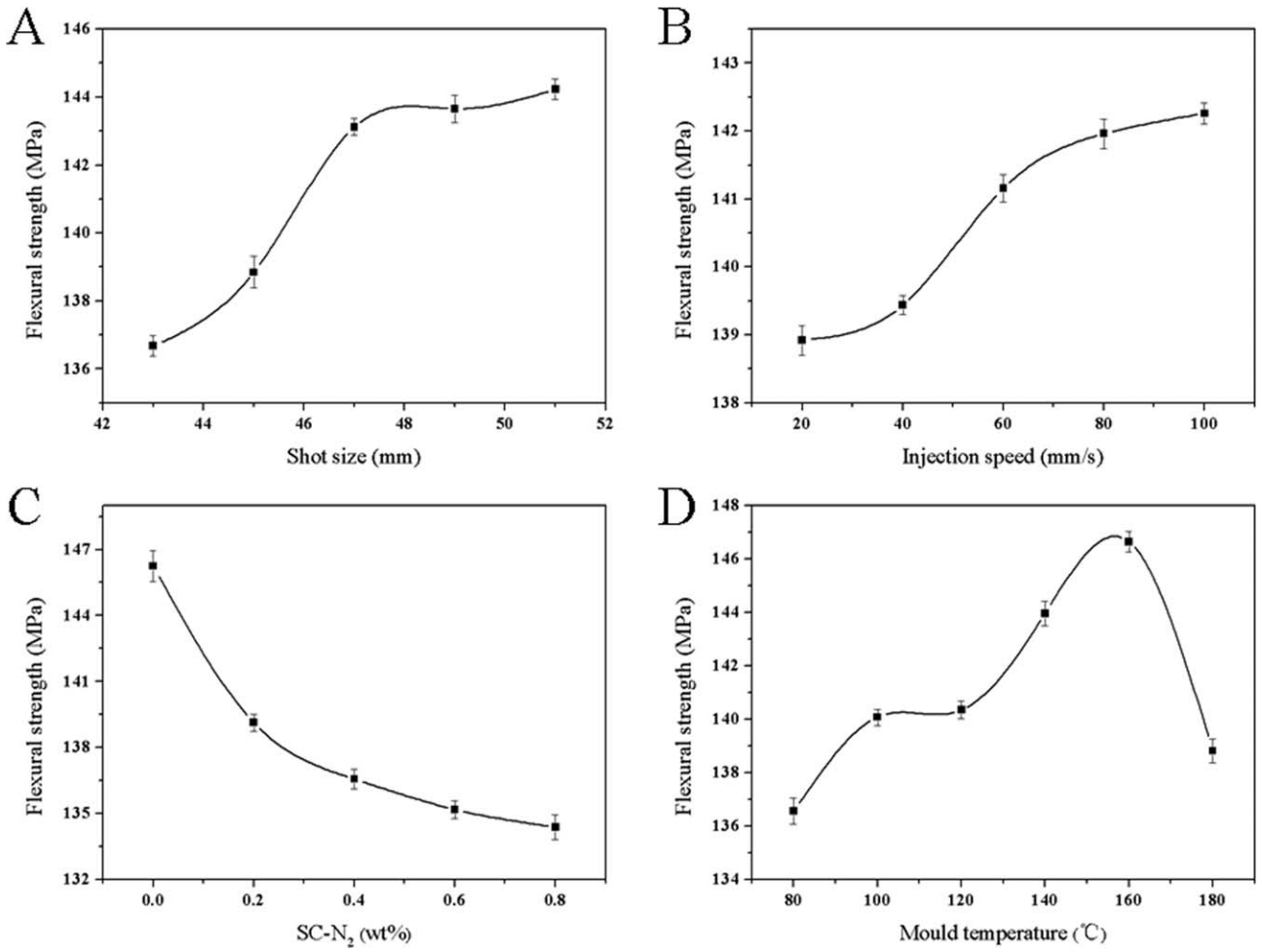

Figure 2. Effects of process parameters on flexural strength of microcellular PEI foams.

the frequency of cell coalescence increased, which resulted in reducing the cell density and augmenting the cell size.

The effect of mold temperature on cell morphology was obviously different from other parameters. With the increasing of mold temperature, cell size decreased to the minimum value 18.5 μm, and then increased to the maximum value 31.5 μm. It was probably because with the increasing of mold temperature, the time of fusant into mold cavity was shorter, so the probability of cell coalescence reduced and the cell size decreased. When the mold temperature reached 180°C, the temperature difference between fusant and mold further declined, and part of molecular chain of PEI started to move, which led to reducing the melt strength of PEI matrix.[34] So the cell coalescence occurred and cell size increased.

Mechanical Properties. The effects of processing parameters on mechanical properties are shown in Figures 1–3, respectively. As shown in these figures, the mechanical properties of microcellular PEI foams were strongly dependent on the processing parameters. First, the mechanical properties of microcellular PEI foams increased with the growth of shot size and injection speed, respectively. Second, with the SC-N_2 content increasing, the mechanical properties of microcellular PEI foams decreased when the other parameters were the same. Finally, the mechanical properties of microcellular PEI foams achieved maximum value when the mold temperature was 160°C. As well known, the structure of materials depended on the properties of materials. According to the above investigation, the processing parameters determined the structure of cells, so the effects of processing parameters on cell size and mechanical properties had the same trend. Meanwhile, the smaller the cell size was, the better the mechanical properties were.[21] So any processing parameters that can decrease the cell size will redound to improving the mechanical properties of microcellular PEI foams.

Figure 4 displays the SEM micrographs of cell morphology of microcellular PEI foams. As shown in Figure 4, the outer layer of microcellular PEI foams was solid structure, but the inner layer of microcellular PEI foams was foam structure. Moreover, the effect of mold temperature on the thickness of unfoamed surface was further investigated because the mold temperature had great influence on melt strength. From Figure 5, one can see that with the growth of mold temperature, the thickness of unfoamed surface decreased, but it was not beyond 600 μm.

Then, both sides of the samples were removed for 1 mm to sufficiently clear away the unfoamed surface in order to study the

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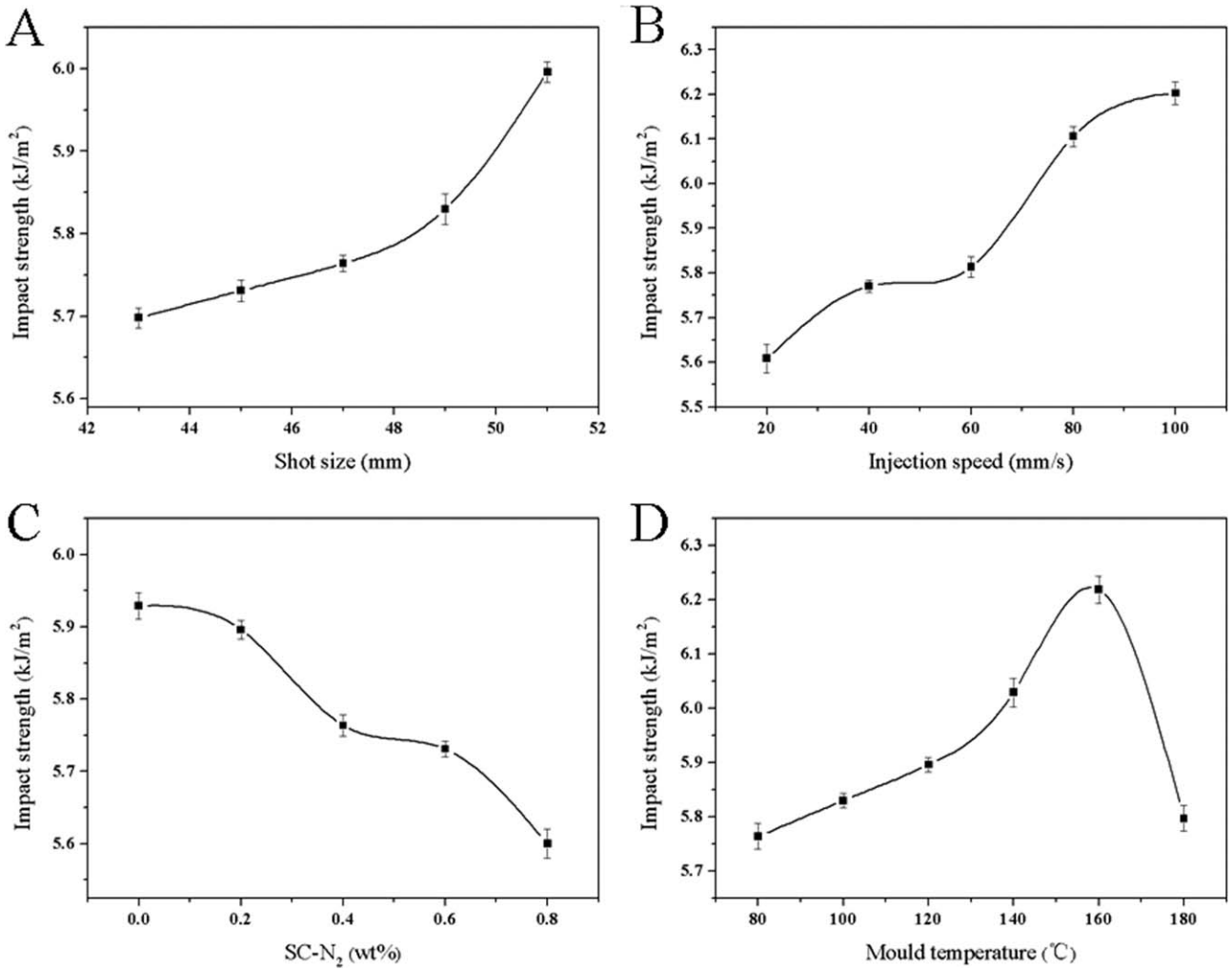

Figure 3. Effects of process parameters on impact strength of microcellular PEI foams.

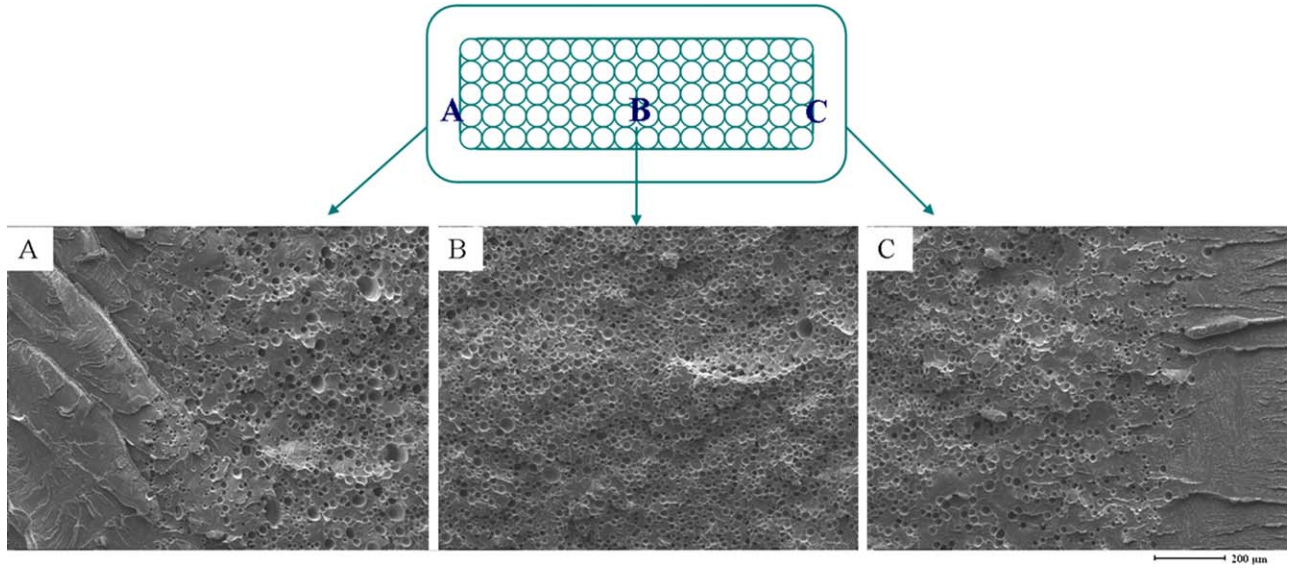

Figure 4. SEM micrographs of cryofracture surface morphology of microcellular PEI foams: (A) and (C) represent the surface layer of sample, (B) represent the middle part of sample. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

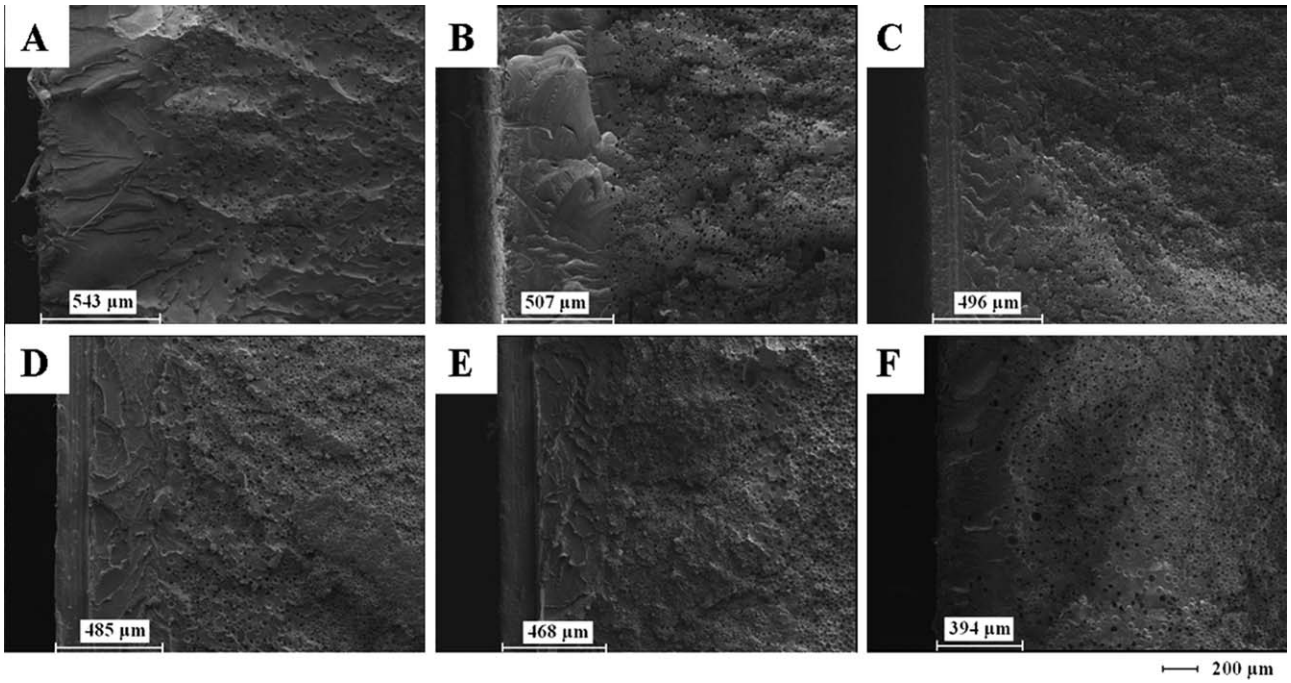

Figure 5. Effects of mold temperature on the unfoamed surface thickness of microcellular PEI foams: (A) 80°C, (B) 100°C, (C) 120°C, (D) 140°C, (E) 160°C, and (F) 180°C.

effect of complete foam structure on the mechanical properties. With the increasing of mold temperature, the mechanical properties of complete PEI foams increased to the maximum value, and then decreased to the minimum value as seen in Figure 6. The mechanical properties of complete PEI foams exhibited similar trend compared with that of PEI foams containing unformed surface. However, the tensile strength and flexural strength of complete PEI foams were obviously smaller than that of PEI foams with unfoamed surface, which proved that the existence of unfoamed surface could keep the microcellular PEI foams stiff. Meanwhile, the impact strength of complete PEI foams performed at a level higher than that of PEI foams with unfoamed surface, indicating that the microcellular structure can not only absorb a large amount of the fracture energy but also prevent the expansion of the crack during the impact test.

Electrical and Thermal Properties. Both the dielectric constant and thermal conductivity of microcellular PEI foams had direct relationships with the morphology and accumulation mode of cells.[35,36] As shown in Figure 7, the dielectric constant and thermal conductivity of samples declined as the mass density of microcellular PEI foams decreased. It was probably because the introduction of cell increased the free volume of internal microstructure and reduced the number of polarization groups within the unit volume, which led to the reduction of dielectric constant.[37–39] At the same time, it is well known that the thermal conduction of materials is caused by the colliding and transferring of microscopic particles in materials.[40–43] The thermal behavior of polymeric composites can be changed by addition of thermal conductive fillers. The microcellular PEI foams can be deemed to polymeric composites. Nevertheless, the thermal conductivity of N_2 was far lower than that

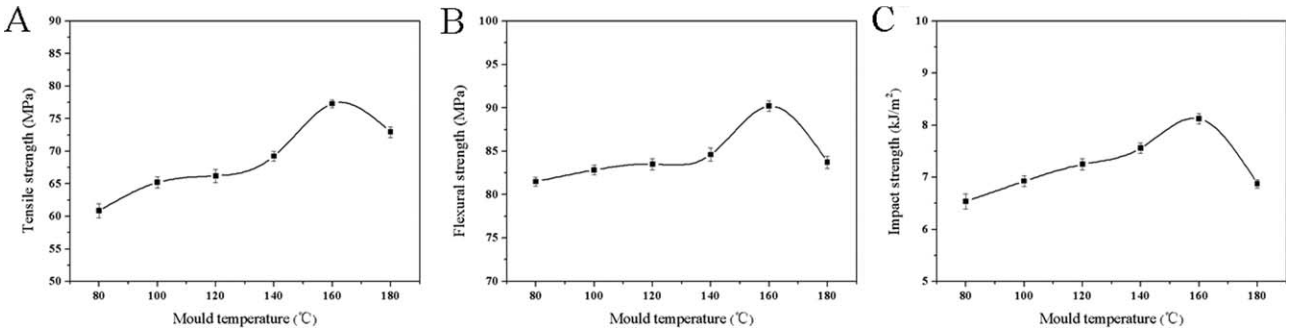

Figure 6. Effects of mold temperature on the mechanical properties of complete PEI foams.

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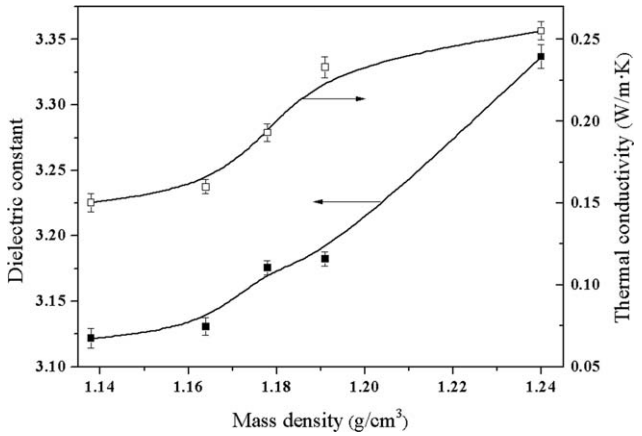

Figure 7. Effect of mass density on dielectric constant and thermal conductivity of microcellular PEI foams.

of PEI matrix, so the presence of N_2 cells can dramatically decrease the thermal conductivity of microcellular PEI foams. When the mass density of microcellular PEI foams reached 1.13 g/cm^3, the thermal conductivity reduces by 41.54%.

Influence of Fillers on Cell Morphology and Mechanical Properties

The MWCNT, NMMT, and Talc were selected as heterogeneous nucleation agents in order to further improve the cell morphology and the mechanical properties of microcellular PEI foams. It is well known that during the procedure of microcellular foams, the fillers that have small size and large surface area can provide a larger number of nucleating points, and induce heterogeneous nucleation.[44] As expected, there was an improvement in cell morphology of microcellular PEI foams after the addition of MWCNT and Talc, as seen in Figure 8 and Table VI. Furthermore, the MWCNT had the most excellent nucleating effect on improving the cell morphology. The minimum cell size of microcellular PEI/MWCNT foams can reach 16.7 μm under the same processing parameters, which decreased by 28.3% compared with pure microcellular PEI foams. This was because MWCNT and Talc can uniformly disperse in PEI matrix. The better dispersibility of particles can play an important role in heterogeneous nucleation and decrease the cell size of microcellular PEI/fillers foams as seen in Figure 8(C,D). However, the NMMT displayed the minus effect that the presence of NMMT increased the cell size of microcellular PEI/NMMT foams at the same situation. As shown in Figure 8(B), the NMMT particles

Table VII. Effect of MWCNT Content on Cell Morphology of Microcellular PEI/MWCNT Foams

	Mass density (g/cm^3)			
MWCNT content (wt %)	Solid	Foam	Cell density (cell/cm^3)	Cell size (μm)
0	1.24	1.18	2.9×10^9	23.3
0.2	1.25	1.18	3.0×10^9	22.1
0.5	1.25	1.18	5.3×10^9	18.3
1	1.26	1.20	9.7×10^9	16.7
2	1.26	1.17	2.8×10^9	21.3
3	1.27	1.17	1.9×10^9	23.8

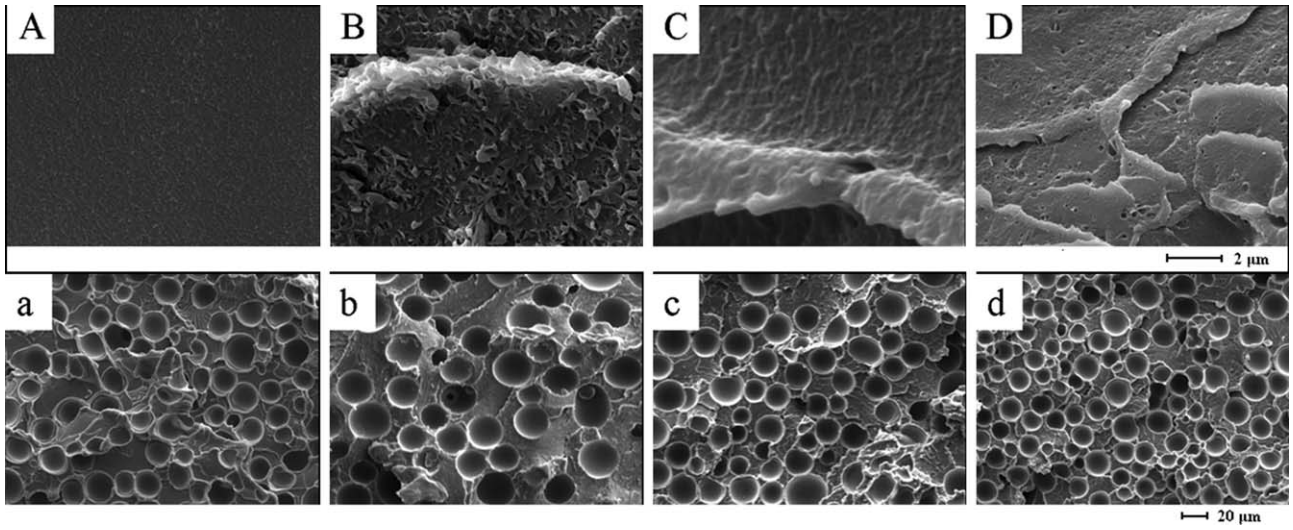

Figure 8. SEM micrographs of cryofracture surface morphology of samples with the different fillers: (A)/(a), (B)/(b), (C)/(c), (D)/(d) represent PEI, PEI/NMMT, PEI/Talc, PEI/MWCNT composites, respectively.

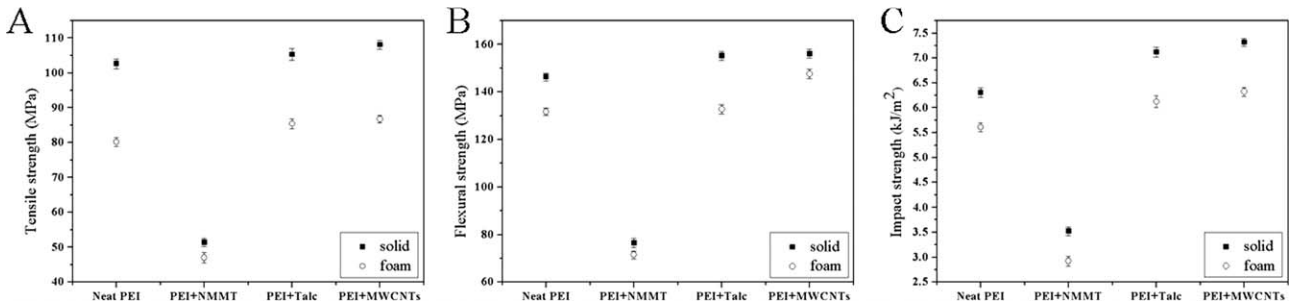

Figure 9. The relationships of mechanical properties with the type of fillers (The content of fillers was 1 wt %, respectively).

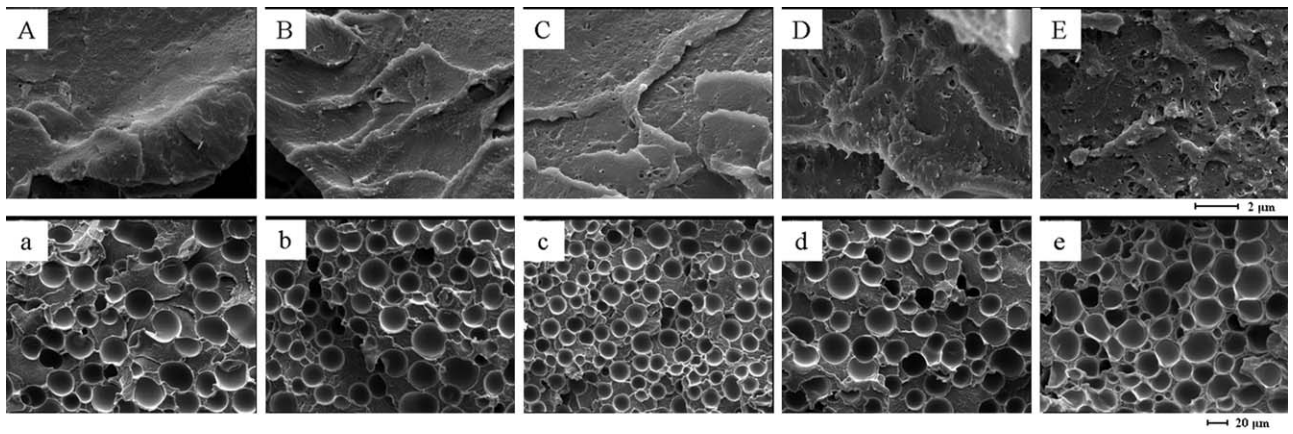

Figure 10. SEM micrographs of cryofracture surface morphology of PEI/MWCNT composites and microcellular PEI/MWCNT foams: (A)/(a), (B)/(b), (C)/(c), (D)/(d), (E)/(e) represent 0.2 wt %, 0.5 wt %, 1 wt %, 2 wt %, 3 wt % respectively.

easily aggregated together, leading to increasing size of NMMT particles. It was difficult that the large size of particles acted as the effective heterogeneous nucleation agent.

Figure 9 shows the relationships between the mechanical properties and the type of fillers. As shown in Figure 9, the mechanical properties of PEI/NMMT composites were obviously smaller than that of PEI matrix, which was mainly attributed to the dispersion of NMMT. It is well known that the better dispersibility of fillers can improve the mechanical properties of composites. One can see that the MWCNT and Talc had significant reinforcing effect on PEI matrix, which is confirmed in previous studies.[28,45] Meanwhile, whether in foaming system, or

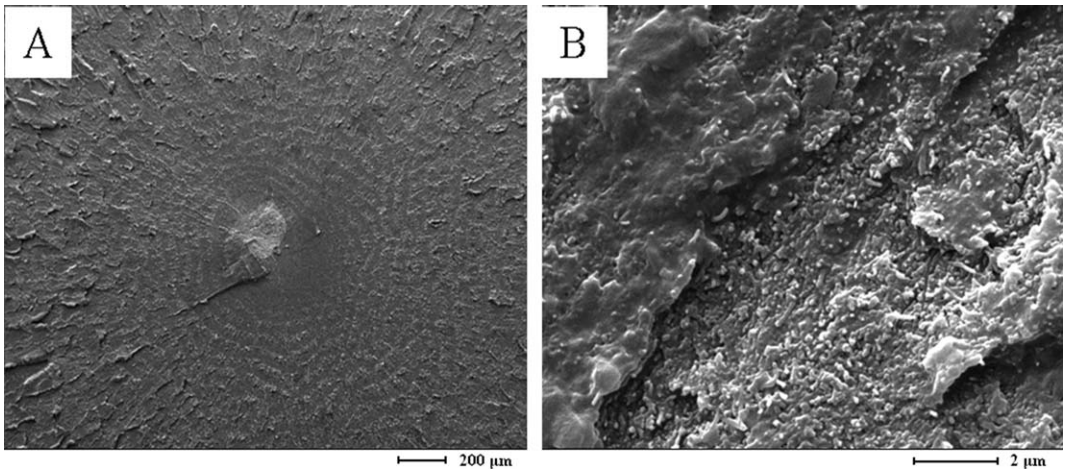

Figure 11. SEM micrographs of cryofracture surface morphology of PEI/MWCNT composites. Magnification: (A) 100×; (B) 10,000×.

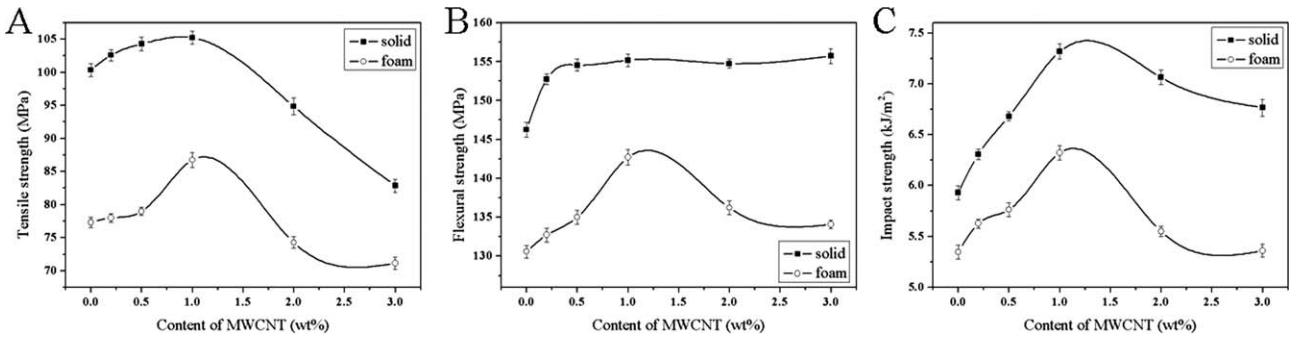

Figure 12. Effects of MWCNT content on mechanical properties of PEI/MWCNT composites and microcellular PEI/MWCNT foams

not foaming system, the mechanical properties of PEI/fillers composites exhibited the same tendency.

Influence of MWCNT on Cell Morphology and Mechanical Properties

The above research found that the MWCNT had a great influence on cell morphology and mechanical properties of microcellular PEI/MWCNT foams. So the influence of MWCNT content on cell morphology and mechanical properties was investigated. As shown in Figure 10 and Table VII, the cell size of microcellular PEI/MWCNT foams decreased to the minimum value 16.7 μm when the MWCNT content was 1 wt %, and then the cell size of microcellular PEI/MWCNT foams increased to 23.8 μm when the MWCNT content reached to 3 wt %. The direct reason that the cell size of microcellular PEI/MWCNT foams declined was also the better dispersibility of MWCNT, whereas the agglomerated MWCNT particles were the cause of increment of cell size, as shown in Figure 11.

Figure 12 shows the curves of the mechanical properties versus the MWCNT content for the PEI/MWCNT composites and microcellular PEI/MWCNT foams. As shown in Figure 12, when the MWCNT content was 1 wt %, the PEI/MWCNT composites and microcellular PEI/MWCNT foams obtained the best mechanical properties.

CONCLUSIONS

Microcellular PEI foams were prepared by microcellular injection molding using SC-N_2 as foaming agent. The effects of processing parameters on cell morphology and material properties were studied. The smallest cell size of microcellular PEI foams can reach 18.2 μm by optimizing the processing parameters. Meanwhile, the presence of cell can obviously decrease the dielectric constant and thermal conductivity of microcellular PEI foams. The MWCNT, NMMT, and Talc were selected as heterogeneous nucleation agents in order to further improve the cell morphology and the mechanical properties of microcellular PEI foams. The MWCNT showed the most excellent nucleating effect on improving the cell morphology. The minimum cell size of microcellular PEI/MWCNT foams can reach upto 16.7μm, which decreased by 28.3% compared with pure microcellular PEI foams. Moreover, the MWCNT played an important role in improving the mechanical properties of the PEI/MWCNT composites and microcellular PEI/MWCNT foams.

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