

## Rheology, Morphology, and Mechanical Properties of HMSPP/POE Blends and its Alternate Layered Foam

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**ABSTRACT:** In this article, the multilayered foaming sheet with alternate layered structure was successfully prepared through multilayer co-extrusion. The high melt strength polypropylene (HMSPP)/poly (ethylene-co-octene) (POE) blend and POE were designed as foaming layers and film layers, respectively. POE was added into HMSPP to reduce the crystalline degree and improve the processing performance. The rheological results indicated that the addition of POE had a little effect on relaxation process and the strain hardening behavior of HMSPP when the POE content was lower than 50%. The results of the foam morphology showed that the cell size and its distribution of the multilayered foaming sheet with alternate layers were better than that with single layer. In addition, the cell size reduced and the cell density increased with increasing the number of layers from 4 to 32. The mechanical properties of the multilayered foaming sheet with alternate layers also could be improved through assembling of foaming layers and film layers. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 41339.

**KEYWORDS:** foams; morphology; polyolefins; properties and characterization; porous materials

Received 29 January 2014; accepted 29 July 2014

DOI: 10.1002/app.41339

### INTRODUCTION

Polypropylene foams were considered as a substitute for other thermoplastic foams in industrial applications due to unique properties, such as excellent chemical resistance, high thermal stability, and good mechanical properties.<sup>1–3</sup> It is well known that polypropylene (PP) with linear molecular structure has poor foamability because of its high crystallinity and low melt strength.<sup>4</sup> To improve the foamability, some approaches have been used to improve the melt strength and the strain hardening behavior in the melt state include crosslinking,<sup>5–7</sup> blending,<sup>8–10</sup> and introduce long chain branches (LCB).<sup>11–13</sup> Also, the blending with other material is simple, economical, and commonly used method.<sup>14</sup> In addition, the high melt strength polypropylene (HMSPP) was prepared by radical reactions because of the introduction of long chain branches (LCB).<sup>15,16</sup> Reasons for their better performance in foaming are their enhanced extensional flow properties, that is, increased melt strength and increased extensibility of the melt.<sup>17</sup> However, due to its high cost and reduced processing performance, HMSPP mostly was blended with other polyolefins.

Rheological and mechanical properties of linear PP/HMSPP blend were systematically studied.<sup>18–22</sup> It was found that tensile stress, elongational viscosity, melt elasticity, and tensile modulus

of the blend increased with increasing the content of HMSPP. The shear thinning behavior of the blend became more obvious and the strain hardening behavior occurred while 10% HMSPP was added,<sup>17,23,24</sup> and other authors studied linear PP and HMSPP blends in an effort to obtain low-density polypropylene foams.<sup>25–28</sup> They studied the Linear PP foaming behavior through HMSPP modification, by adding 20–30% HMSPP. The linear PP foaming behavior have been greatly improved, benefiting viscosity and melt strength decrease sagging and coalescence of cells. However, high crystalline degree of such a blend was not beneficial to the foaming processing. Therefore, the amorphous polymer such as thermoplastic elastomer could be added into PP to reduce its crystallinity. Ying and co-workers found that the introduction of POE influenced the crystallization behavior of PP and then its crystallinity was reduced obviously.<sup>29</sup>

In addition to affecting the rheological properties of PP, the properties of the foams were determined by the foam morphology, such as cell type, cell size, and cell size distribution. Meanwhile, the foam morphology was strongly dependent on the preparation methods and techniques.<sup>30–33</sup> However, most of the foaming methods were difficult to precisely control the foam morphology.<sup>34,35</sup> Generally, most foams were polydisperse and

disordered, containing a wide range of cell sizes that were randomly packed together. As our understanding of foams and foamed materials advances, the foam morphology became one of increasingly important control parameters.<sup>36</sup> Excellent control was provided by using cell designing or ordered foams, which contained only equal-volume bubbles. These foams were special due to their tendency to self-order into periodic structures under gravity or confinement, resulting in excellent properties such as mechanical properties, thermal insulation, sound absorption, and so on. At present, ordered and periodic foams could be generated by the bubbling technique,<sup>36</sup> self-assembly methods<sup>37</sup> and supercritical carbon dioxide foaming,<sup>38,39</sup> which was limited to the foaming liquid or intermittent foams, and could not be applied in the extrusion foaming.

Hiltner and coworkers demonstrated for the first time that foam/film structures could be obtained through the microlayer coextrusion technology and found that these foams had unique mechanical properties.<sup>40</sup> The cell was orderly at a certain extent, but the cell remained polydisperse and disordered. However, coextrusion technology provided a new method for preparing ordered foams. Unfortunately, microlayer coextrusion technology did not attract more attention. So far, the correlative investigation was not reported.

A multilayer coextrusion system was designed in our laboratory. The system contained two extruders, feed block, multiplying elements, water chill block, and haulage drum. An assembly of  $n$  multiplying elements produced  $2^{(n+1)}$  layers.<sup>41</sup> In this article, multilayer coextrusion technology will be used to prepare the multilayered foaming sheet with ordered cell structure and alternate layered structure. The main objectives of this article are to study the rheological properties of HMSPP/POE blends with the amount of HMSPP and prepare the multilayered foaming sheet with alternate layers. The variation of cell size, cell density, cell structure, and cell size distribution with the number of layers and the effect of the number of layers on mechanical properties will be investigated.

## EXPERIMENTAL

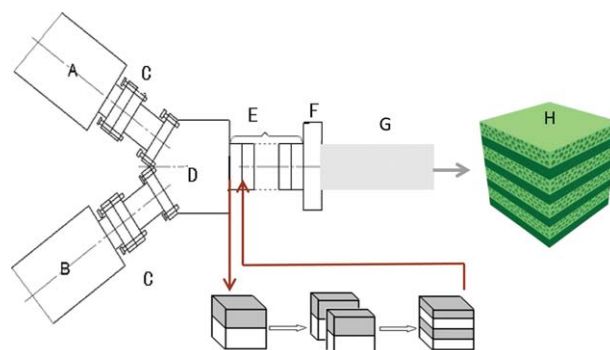
### Materials

HMSPP (Daploy™ WB260HMS) was supplied by Borealis A/S (Denmark). POE (ENGAGE™ 8150) was supplied by Dow Chemical Company. Talc was purchased from Guangxi Longguang Talc Development Co. (China). Azodicarbonamide (AC) was commercially available.

### Specimen Preparation

Standard samples of HMSPP, POE, and HMSPP/POE blends with composition of 100/0, 90/10, 80/20, 70/30, 50/50, 0/100 were prepared, respectively, through twin screw extrusion (diameter of screw was 20 mm and length/diameter was 40/1). The temperature profile from feed zone to die zone was set as 120, 180, 190, and 185°C while the rotation speed of the screw was fixed at 200 rpm. The extensional flow properties and the linear viscoelastic properties were investigated with the rheotest by Extensional rheometer and rotational rheometer.

A multilayer coextrusion system was used to coextrude (diameter was 25 mm and length/diameter was 30/1) the multilayered



**Figure 1.** Schematic of alternate multilayer co-extrusion system (A, B-extruder, C-connector, D-co-extrusion block, E-multiplying elements, F-die, G-multilayered foaming sheet, H-foam/film alternating multilayered structure). [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

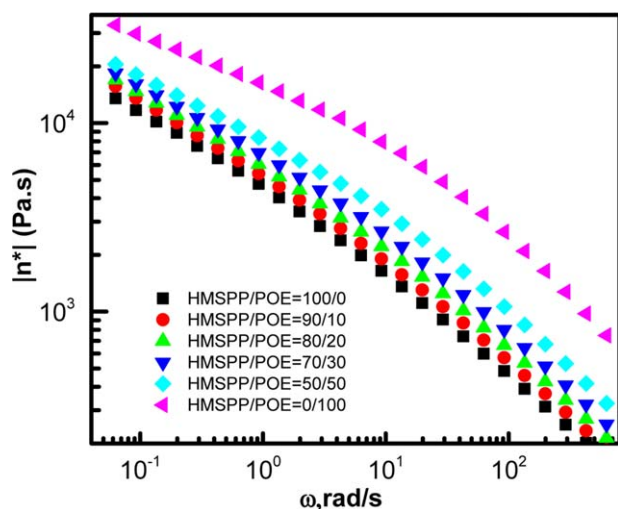
foaming sheets with alternate layered structure (as Figure 1). One extruder contained HMSPP/POE blends with chemical blowing agent and nucleating agent, and the other extruder contained the plain POE. After merging in the two-component feedblock, two kinds of the melts were formed into multiplying element by using the 1–4 multipliers. The 0.5% Azodicarbonamide (AC) and 1% talc were used as the chemical blowing agent and the nucleating agent in the foam layer, respectively. The rotation speed of the screw was 100 rpm, and the temperatures from the hopper to the exit of the extruder were set at 120, 190, 200, and 200°C, respectively. The temperature of LMEs was 200°C. A maximum processing temperature of the multiplying element at 200°C was chosen for optimum gas yield based on the decomposition kinetics of the blowing agent. A nip roll set-up was used as a sheet take-off.

### Measurements and Characterizations

**Rheological Testing.** The viscoelastic properties of the specimens were measured with an oscillatory rheometer (ARES 4400-94, TA Instruments) under a nitrogen atmosphere. The parallel plate with a diameter of 25 mm and a gap height of 2 mm was used for frequencies sweeps. The test specimen was cut from a sheet that has been prepared by compression molding at 200°C. The measure temperature was 180°C. The range of the frequency sweeps was from 0.0628 rad/s to 638.3 rad/s, and a strain of 2% was used. All tests were performed after a preheating step of about 3 min.

For elongational viscosity measurements, the samples were compression molded at 200°C into the sheet shape with 1 mm thickness. The test specimens were cut in a width of 10 mm and the length of 17 mm. The ARES rotational rheometer (TA Instrument) with Sentmanat Extensional Rheometer (SER) was used to measure the extensional viscosity of the samples. The measure temperature was 180°C. The elongational rate was  $0.1 \text{ s}^{-1}$  and  $1 \text{ s}^{-1}$ , respectively.

**Morphological Observation.** Scanning electron microscope (SEM) was used to examine the morphology of the multilayered foaming sheets. Samples were quenched in liquid nitrogen and fractured along transverse direction (TD) and flow direction



**Figure 2.** Complex viscosity versus angular frequency of the plain HMSPP, POE and HMSPP/POE blends at 180°C. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

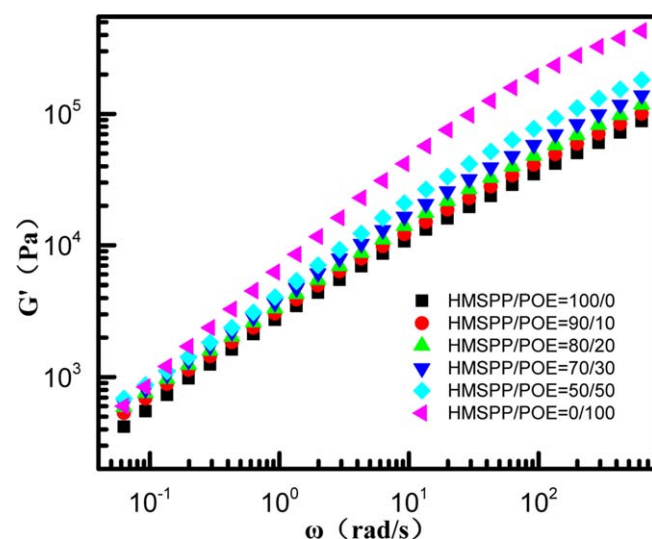
(FD). The measurements used a Hitachi S3400 + EDY SEM (Japan) at an accelerating voltage of 20 kV.

**Apparent Density and Cell Density.** Automatic density analyzer (AccuPyc 1340 II, Micromeritics) was used to measure the overall density of the multilayered foaming sheets at 23°C. The test specimens were cut in a width of 15 mm and the length of 15 mm. At least five specimens for each sample were tested and the average value was calculated.

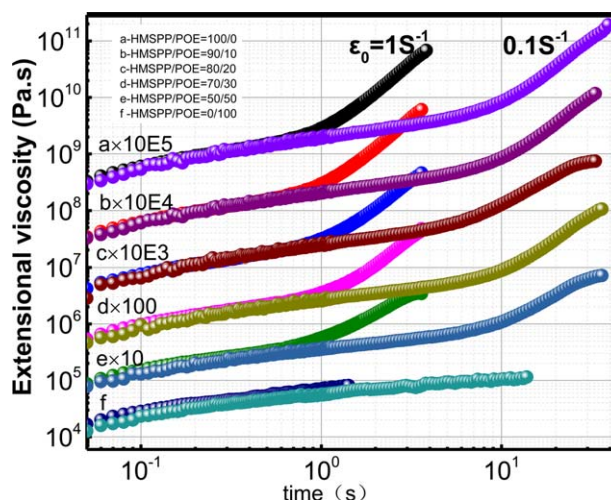
The cell density was calculated by the following equation:

$$N_f = \frac{n_{TD} M^2}{A n_{FD}} \quad (1)$$

where  $N_f$  is the cell density of the foam,  $n_{TD}$  is the number of cells in the transverse direction,  $M$  is the magnifications,  $A$  is



**Figure 3.** Storage modulus versus angular frequency of the plain HMSPP, POE and HMSPP/POE blends at 180°C. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 4.** Transient extensional viscosity ( $\eta_e(t, \epsilon_0)$ ) of HMSPP/POE blends as a function of time  $t$  with different Hencky strain rates  $\epsilon_0$  and POE content ( $T = 180^\circ\text{C}$ ). [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

the area of transverse direction,  $n_{FD}$  is the number of cells in the flow direction.

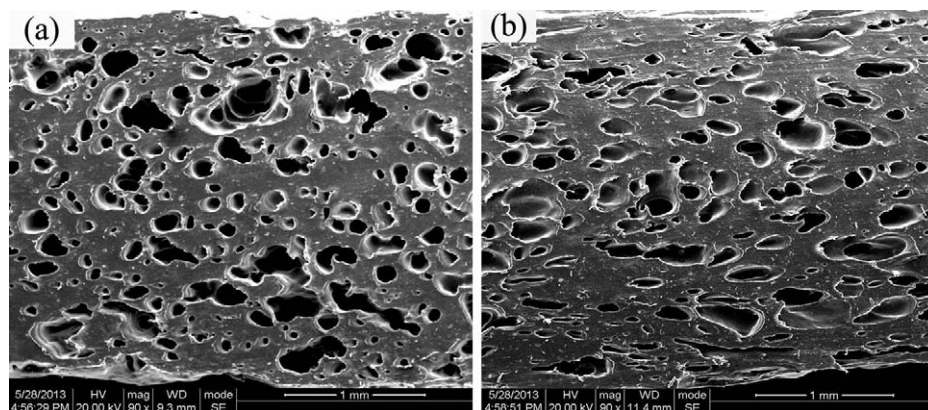
**Tensile and Compression Testing.** According to ASTM D 638, the tensile testing used an Instron 5567 tension machine (Instron Corporation) at 23°C, and the rate was 20 mm/min. According to ASTM D3574, the compression testing was used an Instron 5567 tension machine (Instron Corporation) at 23°C, and the rate was 2 mm/min. At least of five specimens for each sample were tested and the average value was calculated.

## RESULTS AND DISCUSSION

### Linear Viscoelastic Properties

The complex viscosity ( $\eta^*$ ) versus angular frequency ( $\omega$ ) of HMSPP, POE, and HMSPP/POE blends was shown in Figure 2. As shown in the whole range of explored frequency, HMSPP, POE, and HMSPP/POE blends exhibited a decrease in the viscosity value with increasing frequency, that is, they were pseudoplastic melts. It was also observed that POE presented a higher viscosity and HMSPP and HMSPP/POE blends presented more noticeable shear thinning behavior than POE at all frequencies and the tendency becomes stronger with the decreasing of POE content. This behavior should be related to the effect of the broad MWD (molecular weight distribution) and LCB of HMSPP.<sup>42–46</sup> In addition, HMSPP and POE had good interfacial interaction, it was also affecting the shear thinning behavior.<sup>47–49</sup> Meanwhile, as shown in the whole range of explored frequency, the viscosities of the HMSPP/POE blends were between those of the virgin components, and these values increased as the POE concentration increased.

Besides the complex viscosity, the storage modulus ( $G'$ ) of the blends was even more sensitive to POE.<sup>50,51</sup> In the terminal zone, where only the longest relaxation times contribute to the viscoelastic behavior,  $G'$  of linear polymers follow the well-known frequency dependence, that is,  $G' \propto \omega^2$ .<sup>52</sup> The  $G'$  of all samples plotted as a function of frequency were shown in Figure 3. It was observed that POE presented higher  $G'$  than



**Figure 5.** The cell morphology of single layer foaming sheet in the vertical direction (TD) and in the flow direction (FD). (a) TD, (b) FD.

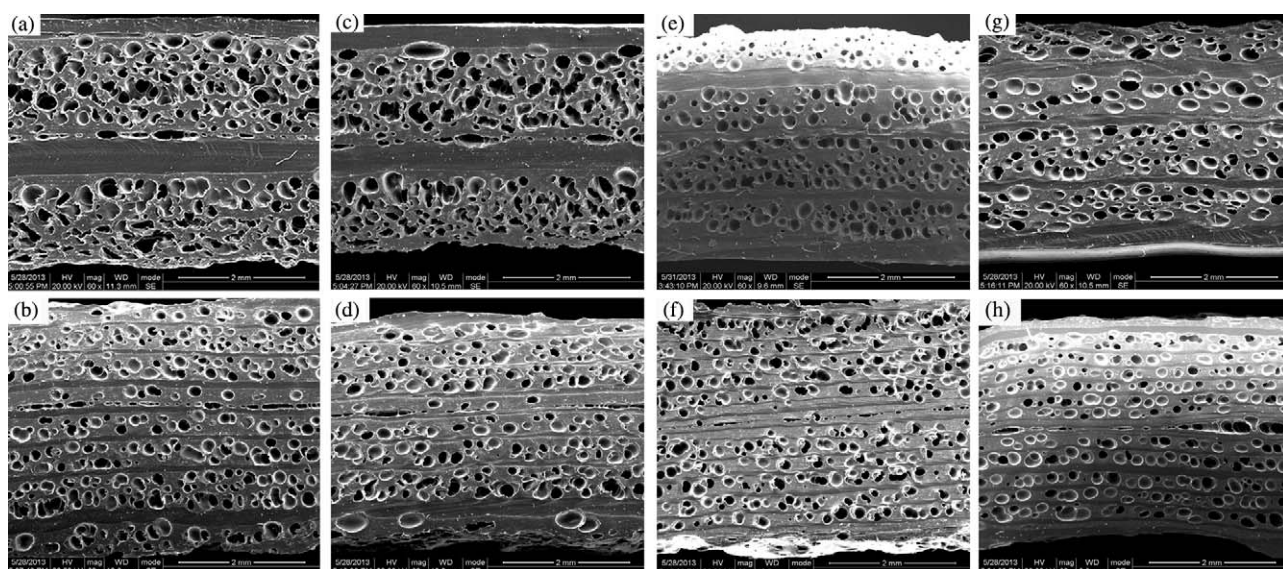
HMSPP within the frequency range studied, and the  $G'$  of HMSPP/POE blends for every composition were between the virgin components and increased when the POE concentration increased because of the good compatibility between HMSPP and POE. The enhanced elasticity of the blends when compared with the matrix phase at low frequencies has already been reported by many researchers for different polymer blends.<sup>53,54</sup> This behavior could be attributed to a form relaxation process of the dispersed phase droplets when slightly sheared. The similar situation for HMSPP/POE blends also appeared at low frequencies, suggesting a longer relaxation mechanism because of the interfacial interaction the HMSPP and POE.

It was clear from all these linear viscoelastic plots that a longer relaxation process appeared in the HMSPP/POE blends. It was believed that this longer relaxation process was related to the interfacial interaction between HMSPP and POE. It could be found that the POE contents were slight effect longer relaxation process. It could improve the foaming processing and reduce

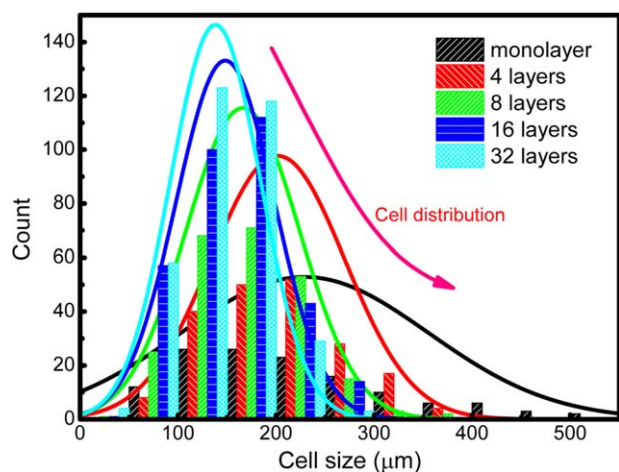
costs by increasing the amount of POE. However, all these plots only proved that the long relaxation process could be found from HMSPP/POE blends. The strain hardening behavior of HMSPP/POE blends was not obtained directly. In other words, the influence of POE content in foaming was not certified.

#### Extensional Rheology

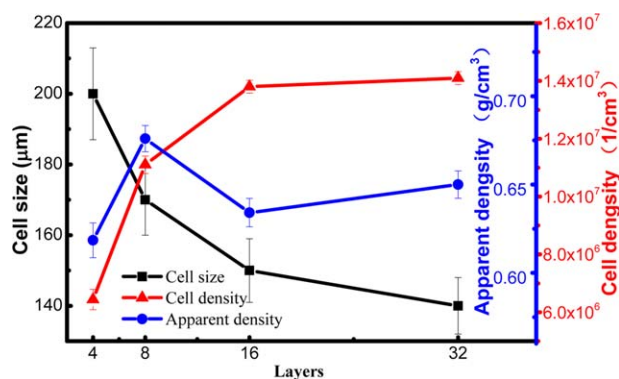
The  $\eta_e(t, \epsilon_0)$  of HMSPP/POE blends as a function of time  $t$  was shown in Figure 4. The  $\eta_e(t, \epsilon_0)$  of POE overlapped and did not deviate upward at all strain rates conducted. The  $\eta_e(t, \epsilon_0)$  of HMSPP and HMSPP/POE blends showed the expected behavior, the so-called strain hardening, for all strain rates. Meanwhile, the  $\eta_e(t, \epsilon_0)$  of HMSPP/POE blends only slight lower than of HMSPP. The results indicated the strain hardening behavior of HMSPP/POE blends was similar HMSPP. These phenomenon was similar to the previous  $G'$ . It indicated that HMSPP and POE were not fully compatible, but both had good interfacial interaction and maintained the strain hardening of HMSPP in the melt. This phenomenon was beneficial to the



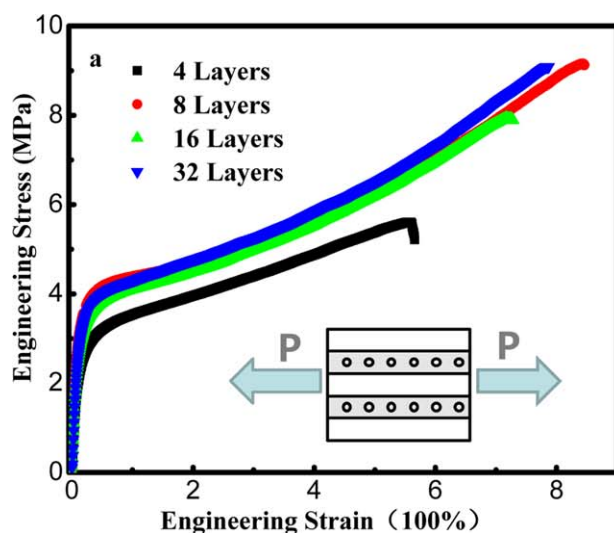
**Figure 6.** The cell morphology of multilayered foaming sheets with alternate layers foam/film (70/30) structure by different number of layers. (a) 4 layers, TD, (b) 4 layers, FD, (c) 8 layers, TD, (d) 8 layers, FD, (e) 16 layers, TD, (f) 16 layers, FD, (g) 32 layers, TD, (h) 32 layers, FD.



**Figure 7.** The Cell distribution of the multilayered foaming sheets with various layers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 8.** The cell size, apparent density and cell density of the multilayered foaming sheets with various layers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



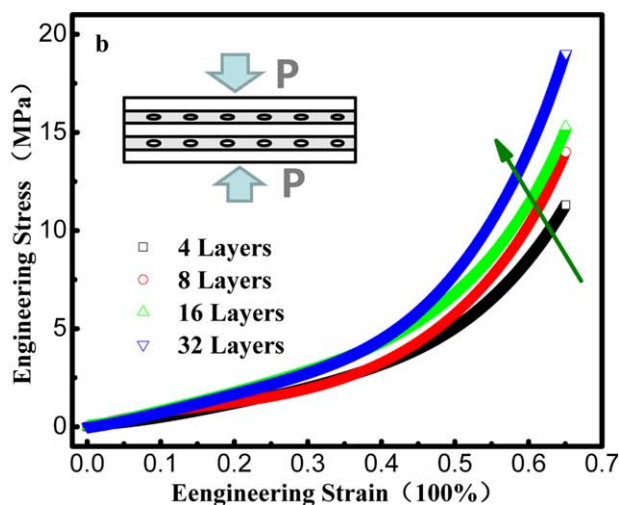
foaming processing. Therefore, the HMSPP/POE blend (50/50) was selected for coextrusion foaming.

### Foaming by Coextrusion

The final foam material was influenced by many reasons in the extrusion foaming process. Khemani<sup>55</sup> summarized the main factors in the polymer foaming process, these factors have significant effects on the extrusion foaming such as material properties, blowing agents, nucleating agents, extruder head, and screw design. To prepare the desired foam material, suitable conditions and die design were the most important. We needed to focus different influencing factor in the different devices, especially. The foaming process of multilayered coextrusion could be divided into three stages; nucleation, bubble growth, and stabilization stages. As the chemical blowing agent decomposes in the extruder, the gas was released. If the amount of gas released was significantly below the estimated solubility limit, it could be safely assumed that no nucleation takes place in the extruder or in the multiplying element.<sup>56</sup> The alternating multilayered sheet experienced a pressure drop when it exited the “exit” die. Therefore, the dissolved gas started to nucleate in the form of bubbles. The bubbles grew as more gas diffuses into the bubbles. The bubbles growth was stabilized as the sheet cools down. The right choice of film material and an optimum concentration of blowing agent ensured maximum foaming without layer break-up.

The morphology of single layer foaming sheet was shown in Figure 5. The cell is too large, nonuniform dispersion and plenty perforation. The average cell size is 230  $\mu\text{m}$ , which was analyzed by the software. This was due to the strongly extrusion shear and a lot of bubble coalescence.

By increasing the number of layers, the cell size was reduced considerably, without adversely affecting the density. The morphology of the multilayered foaming sheet with different layers was shown in Figure 6. The isotropic cell morphology was observed. Meanwhile, the cell morphology was similar in the



**Figure 9.** The Stress–strain curves (a) and compressive stress–strain curves (b) of the multilayered foaming sheets with various layers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

transverse direction and in the flow direction. The cell distribution changed from disorder and wide to order and narrow with the number of layers increasing (as Figures 7 and 8). In other words, the cell distribution spread from the many layers foam to the single layer foam with the number of layers increasing. Therefore, the reduction of the cell size was attributed to enhanced nucleation and/or suppressed cell coalescence under confinement. Also, the cells transformed from the part closed cell and part open cell to all closed cell. It has been observed that the cells with small size were obtained when the foaming took place under geometrical confinement between two impermeable plates. Meanwhile, the foaming region was cut to smaller area with increasing the number of layers, the smaller area meant the shorter gas for foaming in the foaming layer. Therefore, the cell size was reduced with increasing the number of layers.

The apparent density was governed by the total amount of gas released in the system. Because the total amount of gas was fixed, the apparent density did not change. This explained why the density did not change significantly even though the cell size was reduced considerably (as shown in Figure 8). The cell size was reduced from 200  $\mu\text{m}$  to 140  $\mu\text{m}$  when the number of layers was increased from 4 to 32. Meanwhile, the cell density was increased with layers improving. This also proved that the film layer could increase the nucleation density and prevent cell coalescence.

#### Mechanical Properties

Figure 9(a) showed the tensile stress–strain curves of the multilayered foaming sheets with various layers in the flow direction. It is well known that the monodisperse or ordered cell morphology will bring a good performance. The elongation at break and tensile yield strength of the 8, 16, 32 layers foam was better than the 4 layers. The result indicated that the mechanical properties could be improved through the design of the cell structure. Similarly, Figure 9(b) showed the compressive response of the foaming sheets with various layers in the axial direction. In the initial low-strain region, cell bending and cell face stretching took place. The cells started to collapse with strain increasing. The cell collapse was seen as a long plateau in the stress–strain curve. After the cells had collapsed, densification started to occur and the stress started to increase rapidly with strain. The curve of compressive stress–strain showed that the foams with more layers have better resistance compression property. The structure of the layers foam could be recovered almost completely after compression.

#### CONCLUSIONS

The linear viscoelastic properties in HMSPP/POE blends indicated that the POE contents were slightly affect at longer relaxation process of HMSPP/POE blends. It could improve the foaming processing by increasing the amount of POE. Meanwhile, HMSPP and POE had good interfacial interaction and maintained the strain hardening of HMSPP in the melt. This phenomenon was beneficial foaming process.

The foaming sheet with alternate layered structure was successfully prepared through multilayer coextrusion. This method first provided the new idea to obtain the orderly foam morphology

under geometrical confinement between two impermeable plates. The foam morphology with alternate layers was better than with the single layer, such as the cell size, cell distribution. Meanwhile, the orderliness of cell size was improved when the number of layers was increased from 4 to 32. In addition, the film layer could increase the nucleation density and prevent cell coalescence. The mechanical properties of layers foams could be improved by foam/film structure. Meanwhile, the structure of the layers foam could be recovered almost completely after compression.

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