

# Fabrication of cellular and microcellular ceramics with controllable open-cell content from polysiloxane-LDPE blends: I. Compounding and Foaming

Chunmin Wang · Jin Wang · Chul B. Park · Young-Wook Kim

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**Abstract** A novel processing route for fabricating cellular and microcellular ceramics with controllable open-cell content has been developed. The proposed strategy for producing cellular and microcellular ceramics involves: (i) development of desired foamable polysiloxane–polyolefin blends by using a compounding element, in which the polyolefin phase is uniformly dispersed in the polysiloxane matrix, (ii) foaming the obtained blends by implementing the thermodynamic instability principle to produce a cellular or microcellular ceramic precursor structure, and (iii) completing the organic–inorganic transition without sacrificing the obtained cellular or microcellular structure and inducing open-channels in the cell walls by burning out the sacrificial dispersed polyolefin phase at elevated temperatures. By controlling the viscosity of the dispersed polyolefin phase, the polyolefin concentration and compounding parameters, the polysiloxane–polyolefin blend morphology can be varied. Furthermore, plus a deliberate control of foaming and pyrolyzing parameters, the foam morphology and open-cell content of produced cellular and microcellular ceramics can be adjusted. In this paper, the technique to get a desired cellular and microcellular ceramic precursor structure is demonstrated. The deliberate pyrolysis technique to

complete the organic–inorganic transition and the mechanical properties of the obtained microcellular ceramics will be discussed in another paper.

## Introduction

Cellular and microcellular ceramics are special classes of porous materials comprised of cells with size ranging from a few microns to a few millimetres. The cells can be surrounded by ceramic walls (closed cell foam) or contain solid material at only cell edges (struts), thus creating an interconnected structure (open cell foam) [1, 2]. The ceramic foams can be synthesized by a number of different processing methods, and these processes, in turn, produce different characteristics in the materials, such as low density, low thermal conductivity, high porosity, high specific strength, high thermal shock resistance, high specific surface area and high resistance to environmental degradation. Due to these unique characteristics, cellular and microcellular ceramics have many applications. For example, high porosity open-celled porous ceramics can be used as fluid filters, catalyst supports, burners, gas diffusers, flame barriers, biomaterial for bone replacement and a carrier for enzymes and bacteria; and close-celled cellular and microcellular ceramics can also be used as energy absorbers and light-weight structure materials [1–16].

Since the cell morphology in cellular and microcellular ceramics, i.e., the cell density, cell size, and cell-size distribution, directly relate to their ability to perform desired functions in a particular application, it has been emphasized to fabricate a porous ceramic structure with controlled cell size distribution, cell

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C. Wang (✉) · J. Wang · C. B. Park  
Microcellular Plastics Manufacturing Laboratory,  
Department of Mechanical and Industrial Engineering,  
University of Toronto, M5S 3G8 Toronto, Ontario, Canada  
e-mail: chunmin@mie.utoronto.ca

Y.-W. Kim  
Department of Materials Science and Engineering,  
The University of Seoul, Seoul 130-743, Korea

density and porosity [3–16]. Furthermore, to fully exploit the industrial potential of cellular and microcellular ceramics, the need to develop a method of controlling the open-cell content of the ceramic foams will be strongly emphasized.

In order to get cellular and microcellular ceramics with controllable cell morphology and controllable open-cell content, numerous different methods have been developed over the past 40 years, including replication methods [3–5], direct foaming of a ceramic slurry [6–9] or of a sol–gel solution [10, 11], CVD deposition of various refractory materials on a foamed carbon skeleton [12], sintering of hollow spheres [13], siliciding of carbon preforms [14], pyrolyzing a preceramic polymer containing PMMA microbeads [15], and pyrolyzing a preceramic polymer foamed with expandable microspheres [16].

Recently, our feasibility study demonstrated that a low-density, fine-celled microcellular ceramic structure could be developed from various preceramic polymers with high pressure CO<sub>2</sub> by implementing the thermodynamic instability principle and then utilizing controlled pyrolysis [17–20]. With the well-established technique for polymer foaming, the cell density of obtained microcellular ceramics can be higher than 10<sup>9</sup> cells/cm<sup>3</sup> and average cell sizes can be smaller than 30 μm. However, only major closed-cell structures can be produced by this technique and because the obtained microcellular ceramics have a relative low porosity, it will limit the potential applications of the produced ceramic articles.

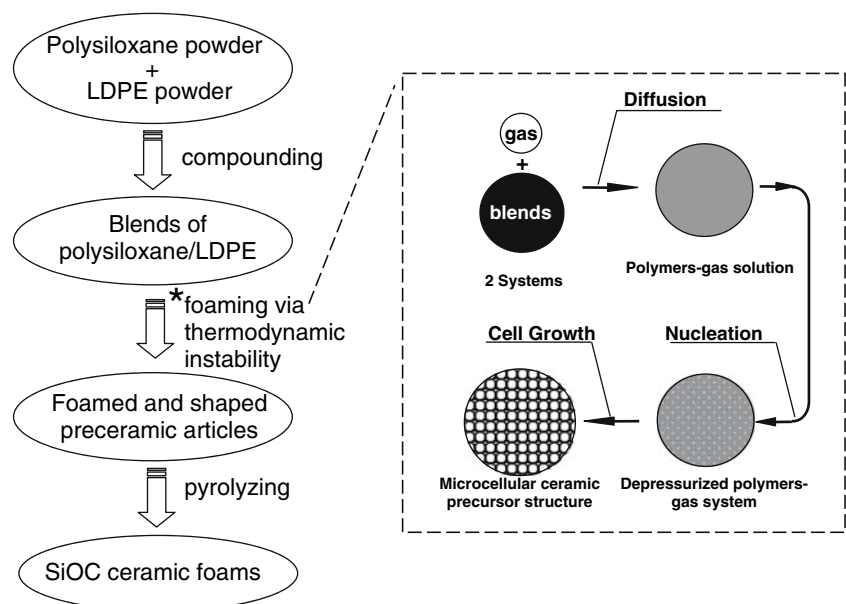
In the present work, a concept of generating cellular and microcellular ceramic precursors with controllable

open-cell content based on compounding and foaming polysiloxane-LDPE blends was developed. The cell morphology and the open-cell content of the produced preceramic foams were tailored by varying the viscosity and concentration of sacrificial LDPE phases and compounding temperatures. Furthermore, the feasibility of producing cellular and microcellular ceramic precursors from compounded polysiloxane-LDPE blends with high pressure CO<sub>2</sub> was illuminated with several examples.

**Scope of methodology**

Figure 1 shows a schematic of the technology proposed in this paper to produce cellular or microcellular ceramics with controllable open-cell content. Technologies are developed in 2 areas to meet the needs of various applications for ceramic foams: (1) open-cell control using a deliberate compounding technique and (ii) cell nucleation and growth control in foaming processes. A cellular or microcellular structure can be created by implementing the thermodynamic instability principle in a foaming system to produce the desired cell morphology such as, controllable cell size, cell density and cell distribution. Whereas, the open-cell content can be manipulated by controlling the polyolefin content and the compounding conditions in a compounder element such as an inner mixer. It can then be followed by inducing the desired open-channels in cell walls by burning out the sacrificial polyolefin phase during pyrolysis.

**Fig. 1** Overall schematic of the proposed technology to produce microcellular ceramics



**Fig. 2** Schematic of the proposed technology to control open-cell content in the produced microcellular ceramics

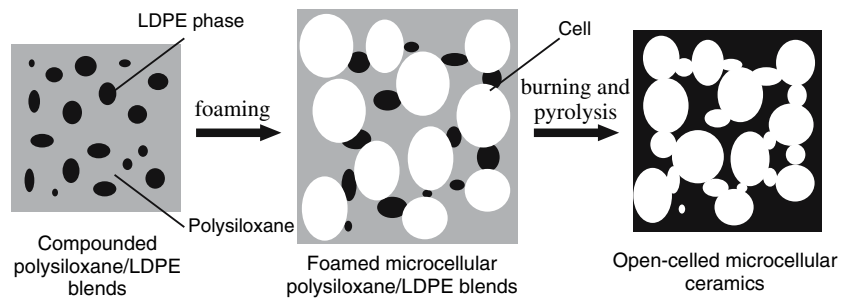


Figure 2 shows in detail a schematic of the proposed technology to control the open-cell content in the cellular or microcellular ceramics processing. By varying the polyolefin content and distribution in the foamed polysiloxane matrix, controlled open-cell contents can be obtained after burning out the dispersed polyolefin. Herein, the amount and type of the polyolefin will be important for ensuring proper dispersion of the polyolefin during compounding and foaming, and a proper dispersion of the polyolefin in the polysiloxane matrix will be further critical for controlling the open-cell content of the final microcellular ceramic articles. Because the polyolefin will act as a sacrificial phase to induce open channels in the cell walls after burning out at an elevated temperature, numerous separately isolated polyolefin domains should be induced in the foam structure. These domains are preferred to be located in the cell walls rather than in the corners (cell struts). In order to get the above mentioned uniform dispersion, a high shear field should be promoted to the compounding procedure.

After the polysiloxane–polyolefin mixture is formed, a foam structure will be created by introducing a thermodynamic instability into the blends/gas system to promote a large cell density in the polysiloxane–polyolefin blends matrix. The following steps will be involved in the proposed processing by using a batch foaming system: (i) saturating the polysiloxane–polyolefin blends using high pressure gases, liquids or supercritical fluids, (ii) nucleating and growing a large number of microcells using a thermodynamic instability via a rapid pressure drop or optional rapid heating, (iii) optional stabilizing.

Typically, the first step is accomplished by fully saturating the compounded polysiloxane–polyolefin blends using high pressure gases, such as  $\text{CO}_2$  or  $\text{N}_2$  in a sealed chamber. The gas type and saturation pressure will affect the foaming behavior of the blends. For example, gaseous or supercritical  $\text{CO}_2$  dissolved into polysiloxane has a strong plasticizing effect and lowers the softening temperature below room temperature

(R.T.), resulting in foaming when rapidly depressurized [17, 18]. In contrast, according to our preliminary experimental results, even higher pressure  $\text{N}_2$  cannot vary the softening temperature of polysiloxane; therefore, subsequent heating is required for inducing thermodynamic instability and foaming when using  $\text{N}_2$ . The next step of the foaming process involves subjecting the saturated blends to a thermodynamic instability to nucleate microvoids, which is achieved by rapidly lowering the solubility of gas in the polysiloxane–polyolefin blends via a rapid pressure drop and/or temperature control. Cell nucleation can occur homogeneously throughout the material or heterogeneously at high-energy regions such as phase boundaries in the blends. In fact, cell nucleation is critical in cellular and microcellular ceramic production since it affects the cell morphology, and to a large extent, the final properties of the cellular and microcellular ceramics. Once the cell nuclei are formed, they tend to grow and reduce the total density as the gas in the blends continues to diffuse into those cells. The growth of cells depends on the diffusion rate of the gas, the melt strength of the blends/gas solution, the available gas inside the solution, and the time allowed for cells to grow [21, 22].

Once a microcellular structure is formed from the polysiloxane–polyolefin blends in the batch processing equipments, the foamed articles will be fully cross-linked and then pyrolyzed at elevated temperatures to produce cellular and microcellular ceramics. During pyrolysis the polyolefin will burn out and the organic–inorganic transition will be completed without sacrificing the obtained foam morphology. This paper will focus on the compounding and foaming behaviors of polysiloxane–LDPE blends, and the pyrolysis behaviors of the obtained ceramic precursor will be discussed in a following paper.

### Experimental procedure

A commercially available polysiloxane (YR3370, GE Toshiba Silicones Co., Ltd, Tokyo, Japan) was used as

a preceramic polymer. Its pyrolysis in inert atmospheres yielded a  $\text{Si}_x\text{O}_y\text{C}_z$  ceramic, with a weight loss of about 19%. Four grades of LDPE (LC0218, Nova Chemical, Canada, the Melt Flow Index according to ASTM standard D1238 (designated as MFI) is 1.5 g/10 min); LDPE722, Dow Chemical, Canada, MFI = 8.0; LDPE993I, Dow Chemical, Canada, MFI = 25; and LD509, Exxonmobil Chemical Co., USA, MFI = 70) were compared by investigating their viscosities. The rheological behaviors of the four grades LDPE and YR3370 were measured by using a rheometer (RS-200, Rheometrics Inc., Piscataway, USA) with a parallel plate geometry (plate diameter of 25 mm and a gap of 1.0 mm). The materials were rheologically characterized by recording the dynamic frequency sweeps at 120 °C; and finally LD509 (Exxonmobil Chemical Co., Houston, USA) was selected as the sacrificial polymeric material. Eight batches of mixtures were prepared and the contents of LDPE in the mixtures were varied from 2 wt% to 50 wt%, respectively.

The mixtures were directly compounded using an inner mixer (3-piece Mixer, C.W. Brabender Instruments, Inc., Hackensack, USA). The compounding temperature was set at 120 °C. The driver speed was 50 rpm and the compounding time was 15 min for each batch.

In order to investigate the effect of compounding temperature on the blend morphology, a particular study was carried out in the inner mixer under fixed material parameters. For case, LD509 (Exxonmobil Chemical Co., Houston, USA) was used and the concentration of LDPE was fixed to 10 wt%. The compounding temperatures were varied from 110 °C to 140 °C, respectively.

After compounding, parts of each obtained blends were placed in a pressure chamber where the samples were saturated for 24 h with high pressure  $\text{CO}_2$ . The saturation pressure was maintained at 800 psi and saturation temperature was room temperature. Next, a thermodynamic instability was introduced by rapidly dropping the pressure at room temperature, and then a foamed preceramic structure was produced after cell nucleation and growth.

The microstructures of the foamed preceramic specimens were observed by scanning electron microscopy (SEM, S-4300, Hitachi High Technologies Co., Japan). The void fraction of the obtained porous ceramic precursors was measured by using an image analyzer (Image-Pro Plus, Media Cybernetics, USA). The cell density of the porous ceramic precursors was measured by counting the number of the cells in a

two-dimensional image on the microstructure and by converting it to 3-dimensions [17].

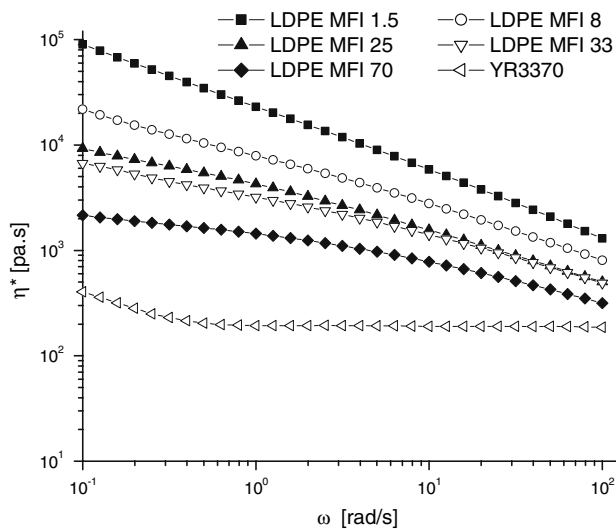
## Results and discussion

In order to control the open-cell content of the desired ceramic articles, an important element of this research is concerned with the dispersion and mixing of polysiloxane/LDPE blends in melt-compounding processing equipment such as an inner mixer. With the ultimate objective of controlling factors to produce desirable foamable blends and consequently control the open-cell content in the ceramic articles, the amount and type of LDPE, the size and shape of dispersed LDPE phase as functions of compounding parameters, should be investigated.

Generally, the minor LDPE phase in the immiscible polysiloxane/LDPE blends is deformable, as opposed to composite materials, which contain a rigid minor phase such as solid particles or microspheres. A wide range of sizes and shapes can thus be obtained for this dispersed phase during processing. With careful manipulation, the dispersed LDPE domains can range in size from submicron to hundreds of microns. Furthermore, spherical, ellipsoidal, fiber-like, ribbon-like or co-continuous morphologies can be produced under various conditions. The final morphology obtained is a balance between the deformation-disintegration phenomena and coalescence [23]. In the present work, factors affecting the manipulation of shape and size of dispersed LDPE were demonstrated, respectively.

### Viscosity ratio

The viscosity ratio, that is the viscosity of the dispersed phase/the viscosity of the matrix, has been shown to be one of the most important variables for controlling blend morphology. Generally, it is believed that the viscosity ratio should be an approximate unit when designing new polymer blends. If the minor component has a lower viscosity than the major one, the minor component will be finely and uniformly dispersed; conversely, the minor component will normally be coarsely dispersed if its viscosity is higher than that of the major component [23]. In our research, because the viscosity of melt YR3370 is quite lower than most commercial LDPE resins (seen in Fig. 3), the viscosity ratio will be much higher than 1; consequently, the melt drops of LDPE can be easily deformed into fiber-like and even ribbon-like consistency under the influence of a shear flow, and yet do not break forming a

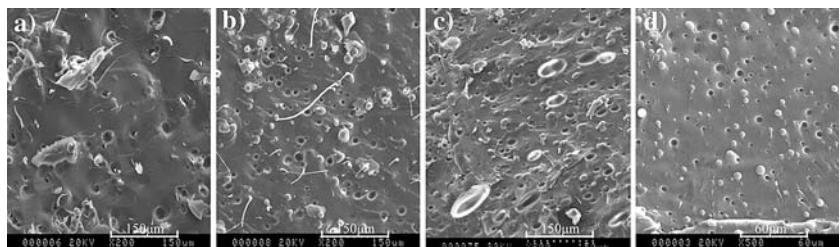


**Fig. 3** Complex viscosity ( $\eta^*$ ) as a function of shear rate for both LDPE and YR3370

spherical dispersed phase. Herein, we changed the viscosity of LDPE by utilizing different grades of commercial LDPE resin and had various MFI to investigate the relationships between viscosity ratio and dispersed phase morphology.

YR3370 was used as the major component and four grades of LDPE with various MFI (1.5, 8, 25, and 70) were used as dispersed polymeric materials, respectively. Four batches of powder blends were prepared for the melt-compounding experiments and the LDPE content was fixed to 10 wt% in all the batches. From Fig. 4, it can be clearly seen that the distribution and morphology of LDPE phase in the blends were correspondingly controlled by the viscosity ratio of investigated polysiloxane and LDPE polymers. When a high viscous LDPE (MFI = 1.5) was used in the compounding, there was a remarkable difference in viscosity and the LDPE phase was coarsely dispersed and deformed into ribbon-like and cylinder-like morphology. Therefore, few spherical dispersed phases could be observed in the compounded blends. As the viscosity of LDPE decreased to MFI equal to 8.0, most of the dispersed LDPE domains were deformed into cylinder-like phase and small amount of fiber-like

**Fig. 4** The effect of LDPE viscosity on the blends morphology, compounded under condition 120 °C/ 50 rpm/15 min, LDPE concentration is fixed to 10 wt% (a) MFI 1.5, (b) MFI 8.0, (c) MFI 25, and (d) MFI 70



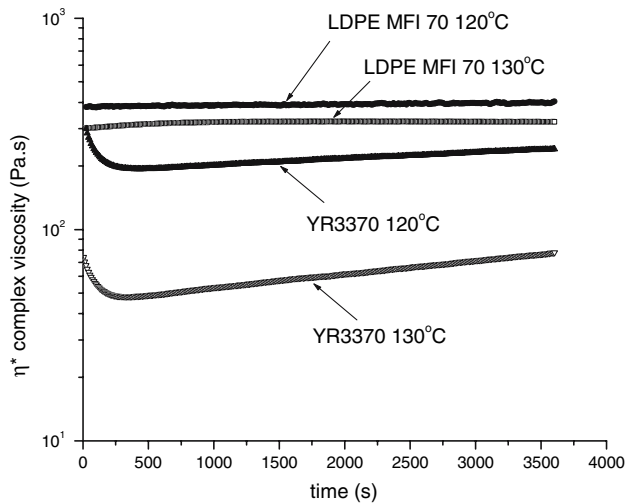
or spherical LDPE phase could be found in the blends. When the minor components with lower viscous LDPE (MFI = 25) were replaced, most of the dispersed phases were ellipsoidal or spherical and the dispersion was more uniform. Thus it can be concluded that the disintegration of elongated fiber-like LDPE droplets mostly occurred around this viscosity ratio and the droplet breakup could only occur more easily when the viscosity ratio had decreased further. As an example, when the LDPE resin with MFI equal to 70 was selected, a more uniformly dispersed, tiny spherical LDPE phase was produced after compounding processing. The average particle size of the dispersed LDPE was smaller than 6 microns.

### Compounding temperature

A particular study has been carried out in the inner mixer under fixed material parameters, such as the grade of the LDPE and the composition of the blend. Consequently, the influence of compounding parameters on particle size can be investigated.

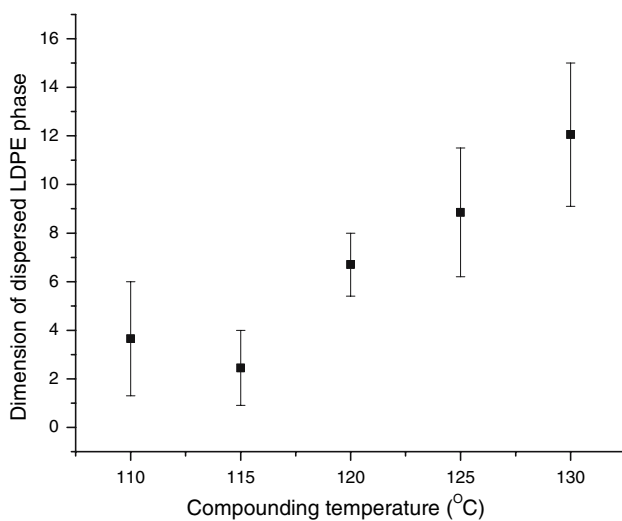
As mentioned before, the final morphology obtained is a result of competition between the deformation and disintegration phenomena on the one hand and coalescence on the other. By increasing the compounding temperature, the viscosity of LDPE and YR3370 will be decreased, respectively. However, in the temperature that ranges between 120 °C and 140 °C (lower than cross-linking temperature of YR3370), the viscosity change of YR3370 is much more sensitive to temperature alteration than that of LDPE (see Fig. 5). As a result, increasing the compounding temperature from 120 °C to 140 °C, the viscosity ratio between LDPE and YR3370 will increase remarkably. Moreover, due to the dramatic decrease of the viscosity of the YR3370 matrix, the force used to induce deformation and disintegration of LDPE phases will decrease correspondingly and the resistance to prevent coalescence between dispersed LDPE domains will decrease too. Therefore, the number-average particles size will increase when the compounding temperature increases.





**Fig. 5** Complex viscosity ( $\eta^*$ ) as a function of heating temperature for both LDPE and YR3370

However, when the compounding temperature decreases around the melt temperature of the LDPE, the number-average particle size will also increase. This phenomenon can be ascribed to the dramatically increase of viscosity of LDPE around its  $T_m$  point (peak melting temperature 104 °C, from manufacturer). Therefore, an optimum compounding temperature should exist in the range of the melt temperature of LDPE and 120 °C. The results from compounding experiments carried out between 110 °C and 130 °C demonstrated the above conclusion (see Fig. 6).



**Fig. 6** The effect of compounding temperature on the dimension of dispersed LDPE phase, compounded under condition 50 rpm/15 min, LDPE concentration is fixed to 10 wt%

## Composition

When the types of LDPE and YR3370 and the compounding conditions are fixed, the concentration of LDPE in the blends will mostly affect the blends morphology.

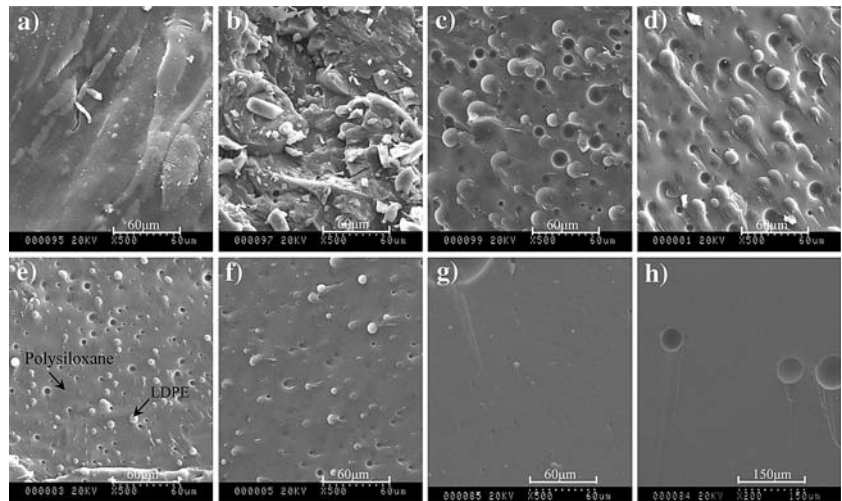
The present research indicates that at about 50 wt% LDPE, there was an intermediate region in which both LDPE and YR3370 were continuous, and as the content of LDPE decreased to 40%, some cylinder-like LDPE phases were observed in the compounded blend. At lower LDPE concentrations, there was a gradual decrease in the dispersed phase dimension with decreasing LDPE concentration. By further decreasing the fraction of the dispersed LDPE phase, it resulted in a decrease in the particle size due to the mitigated coalescence. When the LDPE concentration was lower than 10%, the dimension of the minor phase became less sensitive to concentration, which was perhaps caused by the significantly decreased particle-particle interactions and lowered coalescence (see Fig. 7). In order to further decrease the dispersed particles size, either a more powerful shear field or an optimum viscosity ratio should be induced to the compounding system.

Once the controlled well-dispersed LDPE/YR3370 blends were developed, cellular or microcellular structures were created by introducing a thermodynamic instability into the blends/gas system to promote large cell density. In the present research, we used a batch foaming system to identify the possibility of utilizing the thermodynamic instability to produce microcellular ceramic precursors.

The first step of the processing involved dissolving carbon dioxide under high pressure into the LDPE/YR3370 polymer blends to saturate them up to the equilibrium gas concentration, i.e., the solubility. Once saturated blends were formed after 24 h of saturation, a number of microvoids were rapidly nucleated by introducing a thermodynamic instability via a rapid pressure drop in the LDPE/YR3370 blends.

Since the glass transition temperature of LDPE was much higher than room temperature even saturated by using a high pressure  $\text{CO}_2$ , the dispersed LDPE phase in the blends could not be foamed when rapidly depressurized at room temperature. Consequently, cell nucleation could only occur homogeneously throughout the YR3370 matrix or heterogeneously at high-energy regions such as with the boundaries between the solid LDPE and melt YR3370 phases. Well dispersed LDPE particles could not only control the open-cell content by inducing open channels in the cell walls after being burnt out, but also acts as a nucleating

**Fig. 7** The effect of LDPE concentration on the blends morphology, compounded under condition 120 °C / 50 rpm/15 min, LDPE MFI is equal to 70 g/10 min. (a) 50%, (b) 40%, (c) 30%, (d) 20%, (e) 10%, (f) 5%, (g) 2%, and (h) 0%



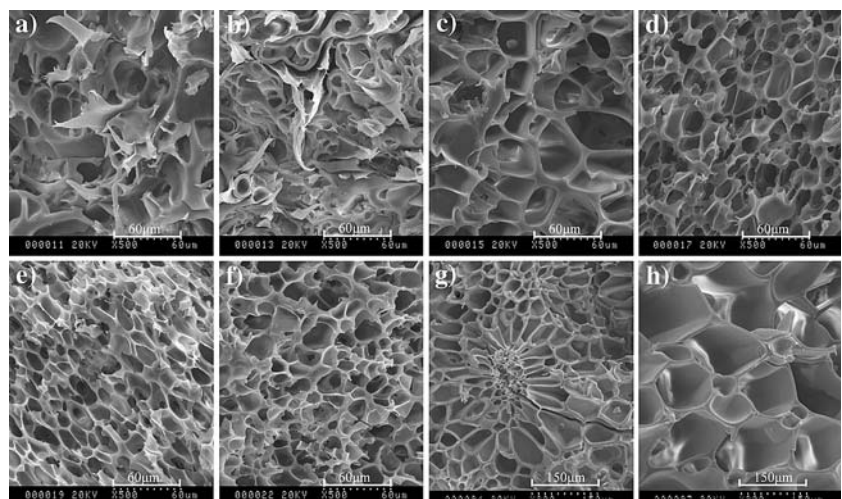
agent in foam processing by inducing large amounts of domains with a high-energy interfacial region. It was demonstrated in Fig. 8(g) that a large number of much more small cells nucleated around the dispersed LDPE spherical phase compared to the cells formed in the YR3370 matrix.

The experimental results suggested that gaseous CO<sub>2</sub>, which was dissolved into polysiloxane, had a plasticizing effect and lowered the softening temperature down below the room temperature (R.T.). This resulted in foaming to occur when depressurized at room temperature. The cell size and cell density were strongly dependent on their processing variables and blend compositions. The cell densities of YR20%LDPE, YR10%LDPE and YR5%LDPE were  $3.5 \times 10^8$ ,  $4.0 \times 10^8$ , and  $2.9 \times 10^8$  cells/cm<sup>3</sup>, respectively. Correspondingly, the average cell sizes were smaller than 30 μm. However, as the concentration of LDPE became lower than 2%, cell density decreased

dramatically from  $1.2 \times 10^8$  to  $5.7 \times 10^6$  cells/cm<sup>3</sup>, and the average cell size increased to above 100 μm. This occurred perhaps because the cell nucleation mechanism changed from heterogeneous to homogeneous nucleation. The microstructures clearly showed that an improved uniformity in the size and shape of the cell spaces was obtained by introducing an optimum concentration of dispersed LDPE phase.

As mentioned above, a proper dispersion of the LDPE in the foamed YR3370 matrix will be critical to control the open-cell content of the final microcellular ceramic articles. Because the LDPE will act as a sacrificial phase after burning out at an elevated temperature, the content, size and distribution of the LDPE in the foamed blends play a critical role in determining the final open-cell content of microcellular ceramics. In the present research, various LDPE/YR3370 blends with different LDPE concentration were compounded and foamed using a batch system, it

**Fig. 8** The effect of LDPE concentration on the foam morphologies, compounded under condition 120 °C/ 50 rpm/15 min and foamed after saturation for 24 h at R.T under 800 psi CO<sub>2</sub>. (a) 50%, (b) 40%, (c) 30%, (d) 20%, (e) 10%, (f) 5%, (g) 2%, and (h) 0%



showed that the LDPE phase continued after foaming at the LDPE concentration above 40 wt%; In contrast, at a lower concentration, the LDPE were dispersed spherically after compounding and foaming, and the size of the dispersed phase decreased with the decreasing concentration of LDPE. However, when the concentration of LDPE was around 30%, the size of the dispersed LDPE was too big and the LDPE phases were entrapped into the wall corners rather than being well dispersed in the cell walls (see Fig. 8). In turn, the open-cell channels could not be induced into the final ceramic foam morphology after burning out the LDPE. Conversely, if the LDPE concentration was lower than 10 wt% and the size of dispersed LDPE was small enough, the spherical phases could be uniformly dispersed in both the cell walls and corners after foaming. This would be more preferred for open-cell controlling in the final ceramic morphology.

### Summary

In this paper, the processing concept based on inducing thermodynamic instability for achieving cellular or microcellular ceramics with desired cell morphologies and controllable open-cell content has been elucidated. A preliminary investigation on the relationships between the viscosity ratio of LDPE/YR3370, composition of blends, processing parameters in compounding and dispersed phase behaviours and morphologies are presented and discussed. It can be concluded that manipulating the dispersed LDPE phase morphology in an immiscible LDPE/YR3370 polymer blend using melt-compounding techniques, is a complex operation, which depends on a variety of materials and processing parameters. Finally, the feasibility of producing microcellular ceramic precursors with compounded LDPE/YR3370 blends by utilizing the thermodynamic instability is identified.

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