The Use of Microcomputed Tomography to Evaluate Integral-Skin Cellular Polyolefin Composites

P. Karimipour-Fard[1], W. Y. Pao[1], G. Rizvi[1], R. Pop-Iliev[1]

[1]University of Ontario Institute of Technology, 2000 Simcoe Street N, Oshawa, ON, Canada L1K 7K4

Aims

Rapid Rotational Foam Molding (RRFM) is a patented processing method for producing integral-skin cellular polymeric composites. It produces composite articles by simultaneously employing rotational molding to produce the solid skin and extrusion foaming to produce the cellular core [1,2]. In this paper, the morphologies of experimentally obtained cellular structures in RRFM made of polyethylene (PE) and polypropylene (PP), respectively, were characterized using microcomputed tomography (Micro-CT). In RRFM processing, the properties of the resulting cellular structures can be generally affected by the implemented processing conditions as well as the relative location within the molding, such as for example the distance from the skin [1,3,4]. In order to assess the quality of the cellular structures of the produced samples both qualitatively and quantitatively in 3D, Micro-CT scanning and various post-processing techniques were implemented. The results are presented in terms of: 3D morphology, cell size distribution, cell density, and percent porosity.

Method

Rapid Rotational Foam Molding (RRFM) was used to produce polymeric composites with an outer solid skin and inner cellular structure [1,2]. During the manufacturing process, extruded foam is introduced on top of an alredy rotationally molded non-chilled skin. As the foam-filling step progresses the final properties of the cellular core differ at various distances from the skin. A combination of PE skin / PP core and PP skin / PE core were studied. Two grades of both PE and PP resins, including a homopolymer PP (hPP), copolymer PP (cPP), linear low density PE (LLDPE), and high density PE (sHDPE), were used to manufacture the foamed core. In this study, RRFM cellular structures were characterized in 3D and the quality was assessed at the core and near the skin of the produced samples.

X-ray Micro-CT scanner Skyscan 1172 from Bruker (Belgium) was used to characterize the cellular structures in 3D. Flat field correction was done to ensure quality of the captured images; 71 μA and 54 kV were set for all scans. Resolution of 2000 x 1000 pixels with size of 12 μm was set for the imaging process of the cellular structures. Rotational steps of 0.4°, frame averaging of 7, and 360° scans were performed to capture high quality imaging of the cellular structures. Alignment calibration of the sample stage was carried out before the scanning procedure. Image post-processing was performed with NRecon, CTAn, CTVox, Data Viewer, and CTVol softwares. At the first step, NRecon software was employed to reconstruct the 3D structure of the scanned images. Then, a cylindrical volume of interest (VOI) was chosen in CTAn software to assess morphological properties of the cellular structures in 3D. Thresholding and despeckeling procedures were done before the 3D analysis to achieve reliable morphological results (porosity analysis). 3D models of the inner cellular structure of the foamed samples were visualized by using CTVox. In addition, Data Viewer software was employed to visualize the cellular structures in xz, xy, and zy planes in space simultaneously. At the end, CTVol software was employed to show a 3D model with phase separations of cell walls, open cells, and closed cells.

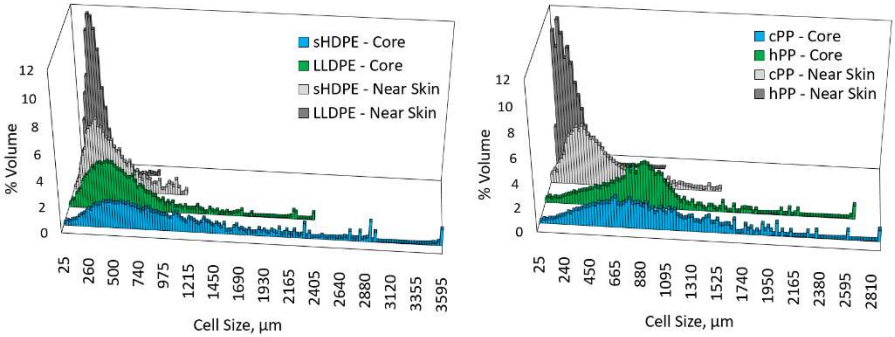
Results

The morphological results of cell size, cell density and percent porosity are presented in Table 1. PE foams from near the skin were observed to have smaller average cell size and distribution range than those from the core, as shown in Table 1 and Figure 1. This could be due to RRFM manufacturing procedure, where the foam was shaped in areas near the skin first and then it would expand towards the core. LLDPE near the skin had the lowest average cell size and highest cell density among the PE cellular structures, therefore, porosity is distributed more uniformly throughout the volume within the cellular structure. PE cellular structure at the core had higher percent porosity, which could be due to a wider cell size distribution and higher quantities of larger cells.

For PE skin / PP foam composites, morphological results from near the skin and at the core demonstrated the same trend that was observed in PP skin / PE foam composites, as shown in Table 1. hPP cellular structure from near the skin had lower average cell size and distribution compared to those of cPP; hPP also showed higher cell density and percent porosity. For cellular structures at the core, cell size distribution and cell density of hPP foam were better than those of cPP foam. It is noteworthy to mention that percent porosity was almost identical for hPP cellular structure from near the skin and at the core; the reason could be the balance in decrement of cell density and increment of cell size distribution from the skin to the core.

Table 1. Morphological results obtained by Micro-CT technique in 3D.

Skin	Foam	Location	Avg. Cell Size (μm)	Max. Cell Size (μm)	Cell Density (cells/cm^3)	% Porosity
PP	LLDPE	Near Skin	190	810	9.06E+05	77.11
PP	sHDPE	Near Skin	260	1120	6.30E+05	74.92
PP	LLDPE	Core	405	2310	4.22E+05	82.45
PP	sHDPE	Core	570	3595	5.40E+04	84.86
PE	cPP	Near Skin	310	1500	1.14E+05	56.66
PE	hPP	Near Skin	95	595	4.60E+06	70.57
PE	cPP	Core	665	2880	1.56E+05	74.91
PE	hPP	Core	880	2595	7.35E+05	70.24



(a) PP Skin/PE Foams (b) PE Skin/PP Foams

Figure 1. Cell size distributions.

Detailed 3D models of hPP and sHDPE foams are shown in Figure 2 to visualize the quality of the internal cellular structures in 3D to confirm the quantitative results; these two samples were chosen because they had the best and the worst quality based on the quantitative results obtained by Micro-CT technique, respectively. Uniform hPP cellular structure and non-uniform sHDPE cellular structure are evident in Figure 2. Also, the 3D models confirmed the result of equal percent porosity throughout hPP cellular structure by keeping the balance of cell size and cell density. Three slices of hPP and sHDPE cellular structures from near the skin are shown in Figure 3. It was confirmed that hPP foam has uniform cell size distribution with relatively smaller cells throughout the entire cellular structure, and sHDPE foam has non-uniform cell size distribution and large pores. Figure 4 presents 3D models of hPP and sHDPE cellular structures with phase separations to illustrate the open cells, closed cells and cell wall structures in 3D with high resolution. The uniform cell size distribution, small average cell size and high cell density of hPP cellular structure is evident in Figure 4 and was observed to be the opposite for sHDPE cellular structure in all of those terms.

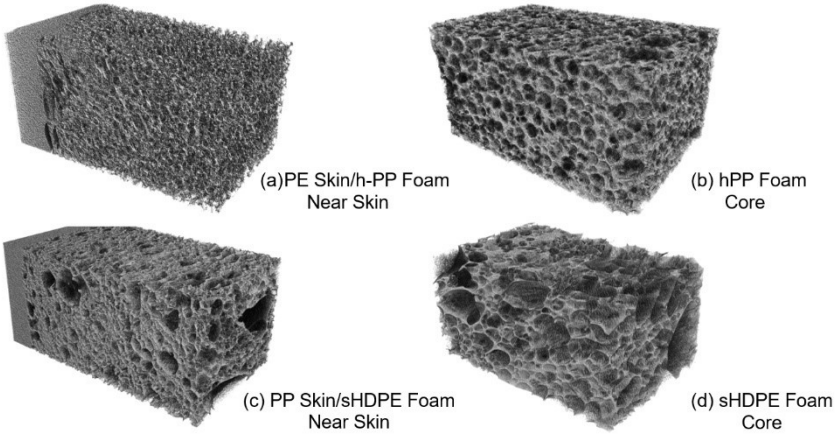
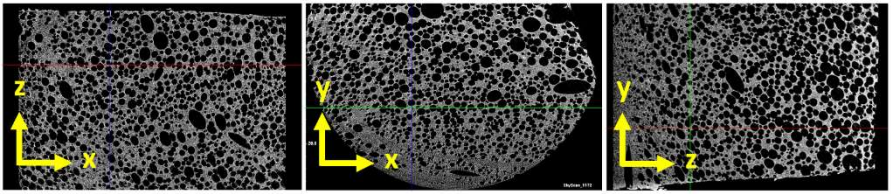
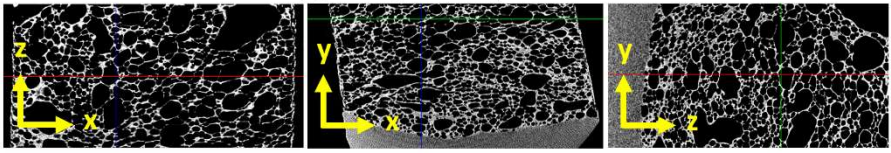


Figure 2. 3D models of internal cellular structures.

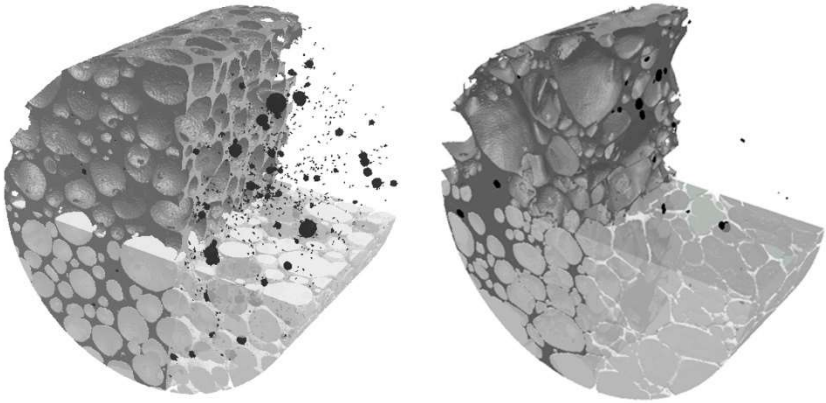


(a) PE skin/hPP Foam, Near Skin



(b) PP skin/sHDPE Foam, Near Skin

Figure 3. Cellular structure slices in xz, xy, and zy planes.



(a) PE Skin/hPP Foam – Core (b) PP Skin/sHDPE Foam – Core

Figure 4. 3D Model of cellular structures in 3 phases: cell walls (dark grey), open cells (light grey), and closed cells (black).

Conclusion

In this paper, microcomputed tomography (Micro-CT) technique was used to analyze morphology of integral cellular polyolefin composites, both qualitatively and quantitatively in 3D. Combinations of PE skin / PP foam and PP skin / PE foam composites were manufactured by using RRFM technique. The X-ray Micro-CT scanner Skyscan 1172 from Bruker and various post-processing softwares were used to analyze morphology of cellular structures with high precision. The obtained results in this study could be summarized as follow:

- By using Micro-CT technique, internal cellular structures of the foamed samples were visualized and the morphological properties of those samples were achieved with high precision in 3D.
- LLDPE cellular structure near the skin had the best quality among all of the PP skin / PE foam composites based on the morphological analysis in 3D.
- PE skin / hPP foam composite cellular structure had the most uniform percent porosity throughout the skin to the core.
- In terms of highest cell density, smallest average cell size and smallest cell size distribution, PE skin / hPP foam composite had the best cellular structure among all of the assessed samples in this study.

References:

1. R. Pop-Iliev, “Processing of Integral Skin Cellular Polymeric Composites in Rapid Rotational Foam Molding” *Acta Physica Polonica A*, **120**, 2, 292-297, 2011.
2. R. Pop-Iliev, et al., “Rapid Rotational Foam Molding Process” US patent: US 8628704 B2, 2014.
3. P. Shahi, et al., “An Experimental Study on Foaming of Linear Low-density Polyethylene/High-density Polyethylene Blends” *Journal of Cellular Plastics*, **53**, 83-105, 2017.
4. P. Shahi, et al., “Morphological Analysis of Foamed HDPE/LLDPE Blends by X-ray Micro-Tomography: Effect of Blending, Mixing Intensity and Foaming Temperature” *Cellular Polymers*, **36**, 221-250, 2017.