Effects of physical foaming of PA66 + 30%GF thick-walled parts

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Abstract. The paper presents the effect of gas dosing parameters on the microporous structure of physically foamed polyamide 66 reinforced with 30 wt% of glass fibers (PA66 + 30% GF). The thick-walled molding was used as the research object. To realize the research aim, simulation software was applied. The variable parameter in a research program was a dose of supercritical fluid, in this case, nitrogen. The tested properties of a cellular structure were: cell size, cell density and local density of a material. All the parameters were considered based on 30 measurement points evenly distributed in the central zone of the sample's cross-sectional area. The largest size of pores was observed in the core region of molding for every amount of supercritical (SCF) fluid used. It was shown that regardless of the amount of gas, the pore size increases as a function of the distance from the mold cavity surface until it achieves a constant value. However, the most rapid increase was observed in the case of the smallest amount of gas dosed (0.25 wt%) which also resulted in the largest pore size in the core zone of a molding. The lowest value of cell density was noticed for 0.25 wt% of gas used. In the case of 0.5 wt%, 0.75 wt% and 1 wt% of gas dosed, results of cell size, as well as cell density, were comparable. However, dosing 1 wt% of gas resulted in obtaining the finest structure, characterized by the smallest pore size and highest cell density. As a parameter resulting from a cell size and cell density, the local density of material was analyzed. The lowest value of local density in the core area was noticed for 0.25 wt% of SCF dosed.

1 Introduction

The demand for plastics, growing from year to year, results in the continuous, dynamic development of the plastics processing industry. Due to the significant use of polymeric materials, solutions are being sought to reduce their consumption. A widely used method of weight reduction is the foaming of the polymeric melt. For this purpose, chemical blowing agents or supercritical inert gases are used. The mixing process of both components takes place in the plasticizing unit of the injection molding machine.

The production of lightweight structures by foaming generates several benefits in addition to the weight reduction of the product. These are, among others: reduction of density [1-2], reduction of deformation and processing shrinkage [3-4], reduction of melt's apparent

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viscosity [5], as well as reduction of material consumption. Moreover, the use of foaming agents in the injection process allows for even several times reducing the processing cycle. This is due to the fact that the expanding gas cells take over the role of the holding phase, which results in shrinkage reduction during the cooling of the molding [6]. Despite the many advantages of using foaming technologies, there are also disadvantages. The main disadvantage is the reduction of many mechanical properties, such as tensile strength [3, 7], bending strength [2, 3], modulus of elasticity [7], and in many cases, impact strength [2, 3]. In addition, in the case of physical foaming, a disadvantage is the high cost of the installation necessary to carry out the process. However, it is possible to significantly reduce the loss of mechanical properties by controlling the porous structure obtained by the process. Sykutera with the team showed that samples produced with inert gas might have higher impact strength than solid samples [4].

The parameters determining the quality of the porous structure are the size and number of cells in the foamed part. The requirement for improving the molding's mechanical properties while maintaining the desired density is to obtain a structure with the smallest possible gas pore size in the molding volume. A fine-pored and regular structure in the molding is possible to get in the microcellular injection molding (MIM). In this process, use a supercritical inert gas, i.e., nitrogen or carbon dioxide. This technology allows to achieve gas pores with a size often not exceeding 30 microns [5, 7, 8]. In the case of chemically foamed moldings, the obtained pores are characterized by larger dimensions and lower regularity [8]. It was shown that the physically foamed moldings have a better surface quality than the moldings obtained due to chemical foaming [6]. For this reason, wide application foamed moldings by MIM technology in the automotive and aviation industries was observed. This applies in particular to lightweight composites based on a polyamide matrix reinforced with glass fibers. The control of the microcellular structure is possible through several input variables such as injection process parameters [7, 9, 10], the geometry of moldings [11], type and quantity of fillers [7, 12], gas injection parameters [4, 5, 7]. Research shows that the finest cells structure can be obtained using micro and nanofillers, such as talc, glass, and carbon fibers [7, 12]. The use of these additives also reduces the processing shrinkage, which, in combination with the MIM technology, minimizes post-process changes in the geometry of injection molded parts [3]. Reducing the shrinkage of molded parts is particularly important in the case of semi-crystalline materials. The presence of the crystalline phase causes that changes in volume are more significant than in the case of amorphous materials [13].

In this paper, the influence of the process parameters on the structure of thick-walled foamed moldings PA66 GF30 was investigated. Tests were performed with the use of finite element methods. The input variable was the supercritical nitrogen amount that was dosed to the polyamide melt. The tested properties were the size and number of gas pores and the local density of the foamed parts. The study using Moldex3D® software (CoreTech System Co., Ltd., Taiwan) was realized.

2 Research methods

The research was carried out with the use of polyamide PA66 GF30 Technyl AR130 (Rhodia, France), containing 30 wt% of short glass fibers. This material has a density of 1.37 g/cm^3. The recommended processing temperature is 270-290 ˚C and the mold temperature is 80 ˚C. For foaming process, supercritical nitrogen was used. To carry out the experiment the Moldex3D® software with the Foam Injection Molding module was used. The default mathematical model for gas pore growth was used, i.e. the Han and Yoo model. The parameters of the injection process used to perform the simulation are presented in Table 1.

Table 1. Injection molding parameters used in process simulations.

Parameter	Value
Mold temperature (°C)	90
Melt temperature (°C)	285
Flow rate (cm^3/s)	400
Switch point (%)	98
Holding time (s)	0.3
Holding pressure (MPa)	14
Cooling time (s)	50

The 3D model has been divided into triangular finite elements in the form of a Boundary Layer Mesh (BLM), the parameters of which are presented in Table 2. This type of mesh, unlike the standard mesh, allows the nodes to be densified in the cross-sectional area of the model, thanks to which the analyzed simulation results are characterized by higher accuracy.

Table 2. Mesh parameters.

Parameter	Value
Solid Mesh Elements	168572
Surface Mesh Elements	34100
Mesh size (mm)	2.2
Mesh boundary layers	3

The analyzed model is shown in Fig. 1. The molding consists of thick-walled samples designed for mechanical testing. The shape of the samples corresponds to the geometry of universal test samples compliant with the PN-EN ISO 527-2 standard (type 1B), scaled by factors equal to 1.5 for the smaller specimen and 2 for the larger one. As a result, the molding is built of two 6 mm thick and two 8.4 mm thick samples. The selected, further analysed test object is a sample with a thickness of 6 mm.

The input variable in the experiment was the amount of supercritical gas (SCF) dosed to the plasticizing unit of the injection molding machine. The variable values were set equal to: 0.25 wt%, 0.5 wt%, 0.75 wt%, 1 wt%.

Measurements of the average pore size, pore density and local material density were conducted using measurement points evenly distributed through the cross-section of the sample. The location of the points from which the data was read is shown in Fig. 2. A total of 30 measurement points were used. Each point was 0.1 mm away from the previous one. The location of the first measurement was precisely on the surface of the model, and the last measurement point was set in the center of the cross-section of the test specimen. This procedure was allowed to determine the distribution of the tested feature as a function of the distance from the sample's surface.
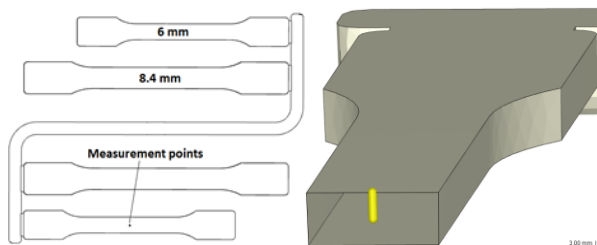
Fig. 1. The geometry of a tested molding with specimens thicknesses and measurement points described (left). Distribution of 30 measurement points on a cross-sectional view of a sample (right).

3 Results

The quality of a porous element is determined by, among others, the size of the pores in the part's structure. Fig. 2 shows the simulated gas pore size as a function of the distance from the molding surface. When analyzing the presented data, it can be seen that each of the curves shows an increasing tendency in the range of the distance from the surface equal to 0 to 2.4 mm. Above this range, all curves take a value close to the constant value. The recorded distributions show that regardless of the amount of gas used, the largest gas pores occur in the core zone of the molded part. The largest gas cell size was recorded for the gas dose equal to 0.25 wt% (black curve). In the area of the core of the sample, this value was approximately twice as high as for the other inert gas doses and was approximately equal to 114 μm. In the case of gas doses equal to 0.5 wt%, 0.75 wt%, and 1 wt%, these values were respectively: 60 μm, 64 μm, 54 μm.

Fig. 3 shows the cell density, i.e., number of pores per unit volume, in this case, cm3. The vertical axis was presented using a logarithmic scale due to the significant differences in the presented values. For 0.5 wt%, 0.75 wt%, and 1 wt%, the cell density value decreases with increasing distance from the surface until reaching a constant value in areas distant from the part's surface by more than 1.6 mm. In the case of the gas dose equal to 0.25 wt%, a different tendency was noticed. The function increases until a maximum is reached for 1.7 mm from the surface, then the values decrease until a constant is obtained for the distance from the surface greater than 2.4 mm. The specimen containing 1 wt% of nitrogen has the highest number of cells, for which the presence of approximately 1350000 pores per cm^3 was recorded in the core of the molding. For 0.5 wt% and 0.75 wt%, these values were very close and were approximately equal to 800000 per cm^3. For a gas dose of 0.25 wt%, a much lower value of 150000 pores per cm^3 was recorded. This means that using a 4-fold lower amount of gas resulted in forming a 9-fold smaller number of pores in the core zone.
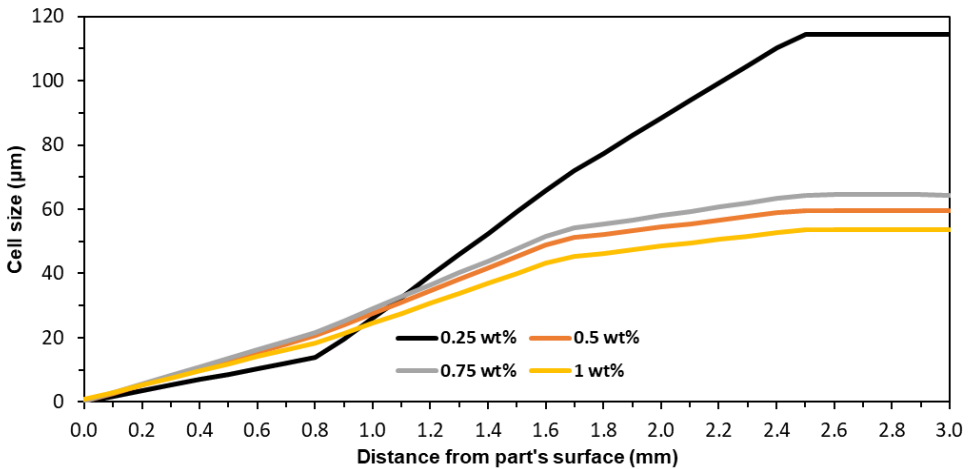
Fig. 2. Distribution of cell diameter as a function of distance from the molded part surface.
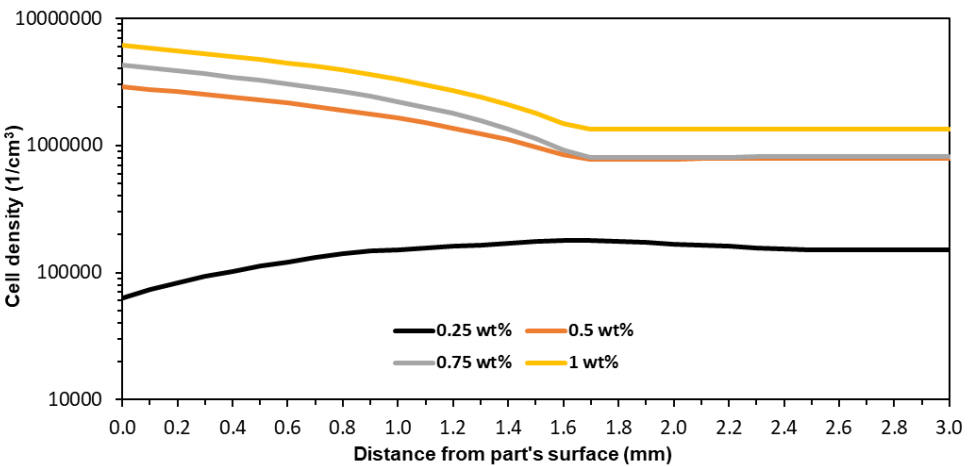

Fig. 3. Distribution of cell density as a function of distance from the molded part surface.

In the analyzed process, regardless of the value of the gas dose, the amount of material supplied to the injection mold was constant. As a result of the assumptions made regarding the processing conditions, a strong correlation between the size of the pores and their number was achieved. The use of a higher dose of gas resulted in the formation of more gas nuclei, which then expanded due to the pressure drop in the injection mold. The expanding gas cells inhibited each other's growth, causing the pore diameter to decrease as a function of their number. Different phenomena were observed for the gas concentration at the level of 0.25 wt%. In the range from 1 to 2.4 mm of distance from the surface, an increase in the size of the gas cells can be observed, while the cell density decreases only for the distance in the range of 1.7 mm to 2.4 mm. In order to understand the observed relationships, the local density of the material was analyzed (Fig. 4).
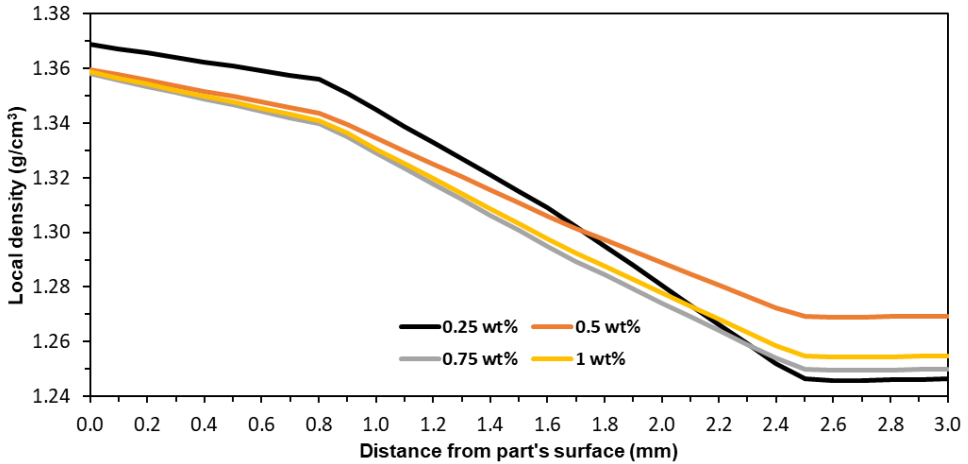
Fig. 4. Distribution of local density as a function of distance from the molded part surface.

All the analyzed curves of density distribution reach the minimum in the core of the molded part, i.e., in the range of 2.5 mm to 3 mm from the part's surface. It can be seen that the local density is strongly correlated with the size of the gas cells. In every considered case, the presence of larger pores results in a reduced density. The test carried out with 0.25 wt% nitrogen, despite the presence of pores approximately twice as large as in the other series, shows the local density in the core zone at a level comparable to the tests conducted with the use of 0.75 wt% and 1 wt% of gas. This was due to the much lower number of gas pores recorded for this series. At the same time, the application of the gas dose at the level of 0.25 wt% resulted in obtaining the most irregular density distribution in all the tests performed.

Comparison of all the analyzed values allows concluding that in the case of the tested thick-walled samples (6 mm thick), the use of supercritical gas concentrations of 0.25 wt% was inappropriate. The presented results prove that in the case of this gas concentration, the material structure is non-homogeneous, and large dimensions characterize the obtained pores. Such a result is undesirable because it has a negative impact on the mechanical properties of molded parts, which has been scientifically proven [2, 14]. The fine-pored structure was obtained with a gas concentration of 1 wt%. The smallest diameter characterizes the pores obtained, and their number per unit volume was the largest among all the series. As a result, the most optimal structure in terms of mechanical properties was obtained while maintaining the density reduction.

4 Conclusions

As a part of the research, the microcellular structure of PA66 GF30 composites, foamed with the use of nitrogen in a supercritical state, was analyzed. It was found that Moldex3D software allows for computer simulation of the microcellular injection molding was applied for thick-walled moldings. In order to achieve the aim of the research, the following values were analyzed: the size of the gas pores and their number, as well as the local density of the material. The values were read at 30 measurement points, the first of which was located on the surface of the molded part, and each subsequent one was 0.1 mm away from the previous one. As a result, the distributions of the studied values on the sample cross-section were obtained.

The conducted research confirmed that increasing the dose of the gas applied significantly improves the quality of the microcellular structure. It is essential that the gas dose used should

not be lower than the limit value, as it may result in a diametrically larger size of the gas pores. It was proved in the example of gas concentration equal to 0.25 wt%. For higher concentrations of inert gas, similar results were obtained. However, it can be concluded that the most optimal structure was obtained for the highest dose of gas tested.

However, an interesting phenomenon is observed in the distance from the surface in the range of 0 - 0.9 mm. For the lowest gas concentration (0.25 wt%), the smallest pores of the molding were recorded. The presence of smaller pores in the near-surface zone has a positive effect on some mechanical properties. However, it should be considered that the obtained values result from specific mathematical models and may differ from reality. A particularly doubtful fact is the presence of pores on the surface of the molded part and in very close proximity to it. In the scientific literature can be observed that the presence of gas cells is not noticed in the skin layer [5, 7].

The conducted test should be extended with the laboratory experiment. It is also reasonable to perform additional measurements ranging from 0.25 wt% to 0.5 wt% of the gas used to find the minimum gas concentration allowing for a homogeneous fine-pored structure.

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