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POLIMERY

Influence of chemical foaming on the structure and selected properties of glass fiber reinforced PA6

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Abstract: The effect of the chemical foaming on the structure (SEM) and selected properties of glass fiber (30, 60 wt%) reinforced polyamide 6 (PA 6) was investigated. Density, tensile properties and Charpy impact strength were determined. Hydrocerol ITP 825 was used as a blowing agent in the amount of 2 wt%. The size of the pores and the foaming degree depended on the distance from the injection point. The smallest pore size (64 μm) was observed for 60 wt% glass fiber reinforced PA6.

Keywords: highly filled polyamide 6, chemical foaming, injection molding, glass fiber, mechanical properties, morphology.

Wpływ spieniania chemicznego na strukturę i wybrane właściwości PA6 wzmocnionego włóknem szklanym

Streszczenie: Zbadano wpływ procesu spieniania chemicznego na strukturę (SEM) oraz wybrane właściwości poliamidu 6 wzmocnionego włóknem szklanym (30, 60 % mas.). Oznaczono gęstość, właściwości mechaniczne przy rozciąganiu i udarność Charpy'ego. Jako porofor stosowano Hydrocerol ITP 825 w ilości 2% mas. Wielkość porów i stopień spieniania zależały od odległości od punku wtryskiwania. Najmniejszą wielkość porów (64 μm) stwierdzono w przypadku poliamidu zawierającego 60% mas. włókna szklanego.

Słowa kluczowe: wysokonapełniony poliamid 6, spienianie chemiczne, wtryskiwanie, włókno szklane, właściwości mechaniczne, struktura.

Contemporary construction materials are faced with several specific requirements, including high mechanical properties, similar to standard construction materials while maintaining the lowest possible weight. An example of constantly developed materials are polymer composites based on polyamide matrix, filled with glass fibers. The reduction of these composites mass can be achieved by using both chemical and physical porosity, causing their structure to expand, *e.g.* in the injection molding process.

One of the research on composite materials directions is to learn about the influence of the matrix and reinforcement amount and type on the indicators characterizing their processing and functional properties. The role of the matrix is reduced to transferring the external stress to the reinforcement and giving the appearance, consistency and shape to the product. However, in the case of reinforcement, it is giving the mechanical properties to the composite [1-3]. As a matrix in composites, i.e. thermo-

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plastic polymers are used. The most common reinforcement in composite materials based on a polymer matrix is glass or carbon fiber. Composite materials based on a polymer matrix reinforced with fibers are used increasingly as replacements for light alloys [4]. These materials, most often processed by injection molding, are used in the automotive, aviation and machine industries (gears, slide-bearing shells) [1, 5].

As a result of polymeric materials foaming, it is possible to lower the products density, reduce deformation and processing shrinkage of moldings, and shorten the production cycle time [6, 7]. The porous structure can be obtained by adding a chemical blowing agent (CBA) (endothermic or exothermic) or a supercritical inert gas (carbon dioxide or nitrogen) in the case of the polymer physical blowing [7-10]. The cellular structure resulting from the blowing agent decomposition usually consists of a solid skin (outer layer) and a porous core (inner layer) [11]. The structure produced by chemical foaming is characterized by larger pore size and a lower pore density than in the cellular structure resulting from physical foaming [9]. An important advantage of using chemical blowing agents is that the process can be carried out with the conventional injection machines use, without the need for additional equipment, as in the case of physical foaming [9, 10].

As it results from the works [12, 13], porosity of polymers causes a reduction in tensile strength, impact strength and hardness [12]. The elongation at break in the case of a small dose of blowing agent decreases due to the action of the pores as a notch, while in the case of increasing the amount of blowing agent to 2%, an increase in elongation was observed [13], which was attributed to the homogeneous structure. The presence of blowing agents also influences the products color [11], which may result from the color of the active substance or the polymer carrier itself.

In the case of polyamide reinforced with glass fibers chemical foaming, the tests concerned relatively low contents of glass fibers (up to 30% by weight). As part of their research, Palutkiewicz and Bold [12] assessed the filler (fibers) and chemical blowing agent (Hydrocerol CF) effect on selected properties of thermoplastic moldings. The experiment was performed on polypropylene reinforced with carbon fibers and polyamide 6 filled with glass fibers. In this case, it was shown that the chemical blowing agent in the polyamide reinforced with 15% and with 30% glass fibers content reduced the mass of the products by 8%, lowered their hardness, and caused a slight decrease in tensile strength.

In subsequent studies, Palutkiewicz and Garbacz [11] analyzed the effect of the blowing agent, glass fibers and mold temperature on selected properties of chemically foamed polyamide 6 reinforced with glass fibers, also containing 15 and 30 wt%. They showed that the use of 1 wt% of the blowing agent contributed to the shortening of the injection molding process cycle times, due to the shortening of the holding phase, while maintaining the molded parts dimensional accuracy. The authors' research confirmed that the presence of glass fibers had a greater impact on the mass of products and their tensile strength than the presence of the porous structure. Porous moldings were characterized by a lower weight than solid moldings, and their tensile strength decreased by 6%. The elongation at maximum stress and the hardness depend on both the dose of the blowing agent and the filler. With an increase in the blowing agent and glass fiber content, the elongation at break decreases. In contrast, the hardness decreases with an increasing amount of blowing agent and increases with the addition of glass fibers. The study did not confirm a significant influence of the filler in the form of glass fiber on the moldings porous structure. In the structural studies, the authors showed a significant influence of mold temperature on the pores size. The higher temperature of the mold made it possible to obtain a small number of large pores in the molded part structure.

Increased fiber content at the level of 50% by mass. in PA6 matrix was analyzed by Roch and his team [14]. In this work, selected performance indicators of a material reinforced with 50% of long and short glass fibers were presented. The material was foamed with the endothermic chemical blowing agent Hydrocerol ITP 825 by Clariant, as well as with supercritical nitrogen (Mucell® technology). In addition, the breathing mold technology was applied. The authors obtained a clear reduction in density by 30% for both blowing agents. Along with the increase in density reduction, an almost linear decrease of the specific modulus of longitudinal elasticity was noted for all tested materials. The specific bending strength for the chemically foamed material was constant, while in the case of physical foaming, the value decreased by about 17%. The authors also observed a thicker skin layer for the chemically foamed material than in the case of physical foaming. In the case of polyamide not filled with glass fibers, the structure was characterized by a distinct non-foamed skin zone and a porous core. Different characteristics were observed for the material containing glass fibers. The skin zone, the zone with small pores and the zone with large pores in the core area were observed. However, the authors did not show any significant influence of the glass fibers length on the specific modulus of elasticity, energy absorption and structure.

Selected functional properties improvement of composite materials consists in particular in increasing the mechanical properties or obtaining an acceptable value of these properties, while reducing the density and maintaining the repeatability of the structural elements geometric features. This makes it necessary to undertake further research aimed at determining the chemical foaming effect of PA6 with an even higher glass fibers content on the structure and selected properties of parts obtained in the injection molding process, taking into account the pores constitution process in the mold cavity.

EXPERIMENTAL PART

Materials

The tests were carried out using polyamide 6 containing 60 wt% of glass fibers (PA6 GF60) with the trade name Restramid B27 GF60 WH-IK 1B0000EX (Polimarky Sp. z o.o. Sp. K, Poland) and polyamide 6 (PA6) with the trade name Nyorbits 28 (BG Polychem Sp. z o.o., Poland). The matrix of the Restramid B27 GF60 material was PA6 with the trade name Nyorbits 28. PA6 GF60 had a density of 1690 kg/m^3, and in the case of PA6 it was 1120 kg/m^3. The study also covered a composite containing 30 wt% of glass fibers (PA6 GF30). This material was obtained by mixing PA6 GF60 with pure PA6 in a 1:1 ratio. PA6 GF30 had a density of 1370 kg/m^3.

As part of the own research, the available blowing agents for the processing of polyamides at high temperatures were assessed. The endothermic chemical blowing agent Hydrocerol ITP 825 (Clariant, Switzerland), which was characterized by the decomposition temperature closest to the melting point of PA6 GF60, was selected. This blowing agent was dosed in the amount of 2 wt% in relation to the polymer matrix. The active substance content in the blowing agent used was 40%. In order to verify the effectiveness of the blowing process, moldings with and without blowing agent were produced.

Sample preparation

Before processing, the raw material was dried in a KMF 115 dryer (Binder, Germany) at a temperature of 80°C, until the moisture content was below 0.1%, according to the recommendations of the material manufacturer. The test samples were produced by injection molding, using an e-victory 110 hybrid injection molding machine (Engel, Austria) with a maximum clamping force of 1100 kN and a screw diameter of 35 mm. Dog-bone samples with standardized dimensions specified in the ISO 3167:2014 type A standard (Fig. 1) were produced in a four-cavity injection mold. The injection process parameters are presented in table 1.

For all compositions, the same process parameters were used, such as: temperature of the plasticizing system zones, mold temperature, dosing volume and clamping force. The solid samples were produced with a holding pressure of 30 MPa, operated for 40 seconds

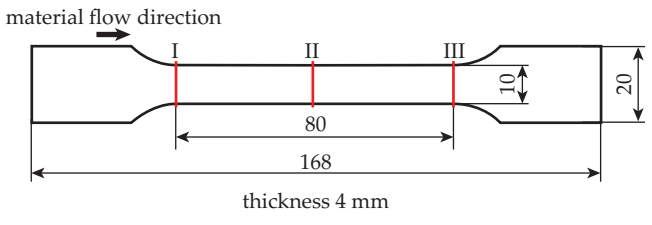

Fig. 1 Geometry of the test specimen - geometrical dimensions with the division of the molding into measurement zones: I – beginning, II – middle, III – end of the measuring section

T a b l e 1. Injection molding process parameters (*parameters used in foaming process)

Parameter	PA6 / PA6 + CBA	PA6 GF30 / PA6 GF30 + CBA	PA6 GF60 / PA6 GF60 + CBA
Temperature of the plasticizing system zones (T_n,Tz_3,Tz_2,Tz_1,T_t), °C	270, 270, 250, 230, 40		
Mold temperature, °C	60		
Injection rate, cm^3/s	50		100
Dosing volume, cm^3	80		
Counter-pressure, MPa	5		10
Cooling time, s	40 / 80*		60 / 80*
Holding pressure, MPa	30 / 0*		50 / 0*
Packing time, s	40 / 0*		20 / 0*
Switch point, cm^3	13 / 13,5*	15 / 12*	10 / 12*
Clamping force, kN	1000		

for PA6 and PA6 GF30. The pressure and the holding time were selected in such a way that the holding pressure was equal to 50% of the injection pressure, and the holding time lasted until the injection gate was solidified. In the case of samples containing 60% of glass fiber, the injection speed and back pressure were also increased due to the difficulties in the material dosing and filling the mold cavity. When producing porous samples, the switch point was also modified to achieve a mass reduction of 10%. The assumed level of mass reduction was dictated by the maximum change in the material volume as a result of its cooling, which was estimated based on PVT diagrams of materials of identical composition and density [15-17]. After each part was removed from the mold, it was weighted together with the cold runner.

Methods

Density

The density was determined by the immersion method in accordance with the PN-EN ISO 1183-1 standard, using the AD50 scales (Axis, Poland) with a measurement accuracy of 0.001 g. Before testing, the samples were dried in a Binder KMF 115 dryer until the relative humidity was below 0, 2%. The mass of the samples was weighed in air and in methyl alcohol, and then their density was determined. The ambient temperature was 23°C. The measurement was carried out on 10 samples. From each area, samples with dimensions of 10x10x4 mm were cut from the beginning (zone I), middle (zone II) and end (zone III) of the 80 mm long test specimen.

Static tensile properties

The static tensile test was carried out using an universal testing machine Z030 (Zwick / Roell, Germany), in

accordance with the PN-EN ISO 527-1 standard. The tensile strength and elongation at break were determined. The elongation speed when measuring the modulus of elasticity was 1 mm / min and then was increased to 50 mm / min. The tests were carried out on 10 previously dried samples from each measurement series, at an ambient temperature of 23°C.

Charpy's impact strength

The impact strength was determined by the Charpy's method in accordance with the PN-EN ISO 179-2 standard (type I samples). 80 mm long rectangular samples were obtained by cutting out from universal test specimens. The test was carried out with the Zwick/Roell HIT 50P impact tester, with the use of the 15 J pendulum, and the support spacing was 62 mm. 10 dried unnotched samples from each measurement series were tested at an ambient temperature of 23°C.

Morphology

The structural analysis of porous samples was performed by scanning electron microscopy. A 5600 microscope (JEOL Ltd., Japan) was used for the research. The electron accelerating voltage was the same for all analyses and was equal to 1 kV. The analyzed fractures were obtained by impact fracture of samples previously immersed in ethanol at a temperature of 70°C. The fractures prepared in this way were sprayed with platinum in a JEOL JFC-1300 vacuum sputtering machine for 30 seconds. The average pore size in the molded part core was estimated on the basis of SEM photos from the beginning, middle and end of the measuring section (Fig. 1) using the ImageJ ver. 1.52a (Rasband W. ImageJ, U.S. National Institutes of Health, USA).

RESULTS AND DISCUSION

Density

Analyzing the density of the produced composites, presented in Fig. 2, a clear decrease in the average density for the materials containing the porous structure was observed.

Moreover, this analysis shows the difference in the average density for the individual zones of the molding along the flow path in the molding cavity. In zone I (closest to the injection point), the density of PA6 containing the porous structure decreased by 5.6% compared to the solid sample. In the case of materials containing glass fibers, the reduction in average density was achieved at a similar level, i.e. 6.7% for PA6 GF30 and 5.5% for PA6 GF60. In the middle of the measuring section (zone II) there was a decrease in the average density value by 8% for PA6 and by 8.7% for PA6 GF30 in relation to solid samples. On the other hand, for high-filled polyamide, the average density in this zone decreased by 7.1%. In zone III, the highest decrease in the average density for moldings containing a porous structure was recorded. The PA6 density in this area decreased by 14.3%, PA6 GF30 by 11.3%, and PA6 GF60 by 11.2%. Differences in the average density value in particular zones of the molded part may result from the fact that the plastic pressure decreases with the increase of the distance from the injection point, which was characterized by E. Bociąga [18]. As it results from the experiment conducted there, it may have a decisive influence on the conditions of the porous structure growth, and thus a bigger reduction in the density. Among the materials tested, the lowest reduction in average density in each test zone was recorded for the polyamide containing 60 wt% of glass fiber. The material with a high filler content (glass fiber) is characterized by smaller changes in the volume of the material due to temperature changes, which is confirmed by PVT charts provided by plastic manufacturers [15–17]. The expanding gas pores thus have a space limited by the glass fibers to expand during the cooling of the melt. The high content of glass fibers in the composition may also be a factor that hinders the homogenization of the polymer with the blowing agent and may have a negative effect on obtaining a homogeneous structure.

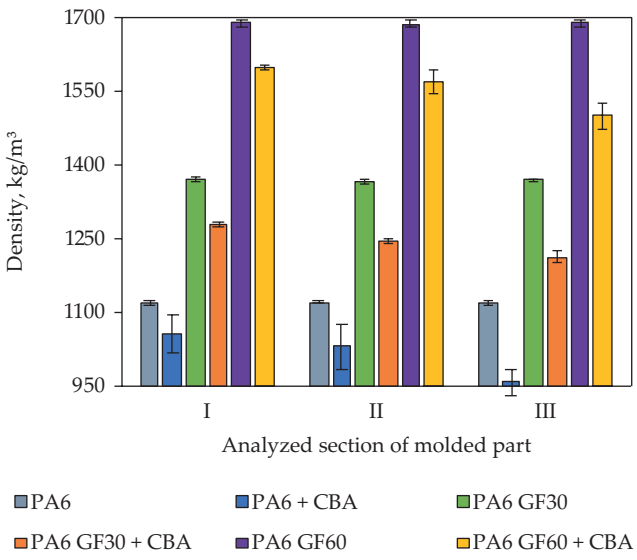

Fig.2. The average density of the tested polyamide composites along with the standard deviation in individual zones along the flow path

Tensile properties

Figure 3 shows the tensile strength of the tested composites. For all series containing a porous structure, a decrease in the average tensile strength was noted in relation to the solid material. In the case of porous PA6 GF60, an average tensile strength of 119.8 MPa was obtained. This value was lower by 22.4% in relation to the solid PA6 GF60 sample. This was the greatest decrease in this material feature among the tested composites. The porous polyamide containing 30% of glass fibers achieved an average tensile strength of 84.8 MPa,

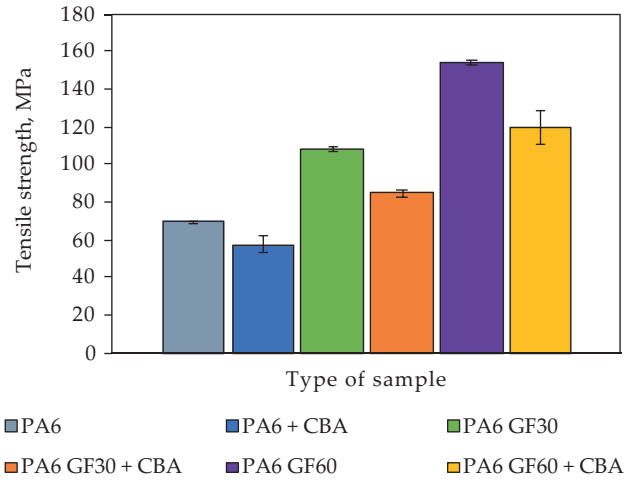

Fig. 3. Average tensile strength of the series tested, together with the standard deviation

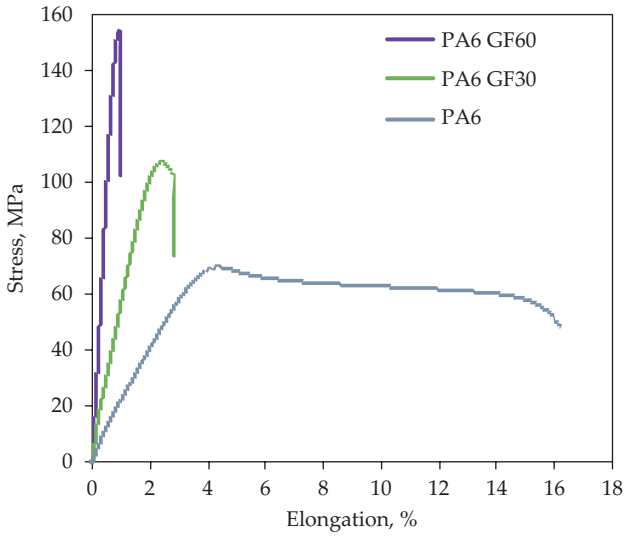

Fig. 4. The relationship between the stress and the elongation of samples

i.e. it decreased by 21.3% compared to the solid sample. The lowest decrease in the average tensile strength was observed for PA6 + CBA, i.e. 17.5%.

The analysis of the tensile curves showing the stress-elongation relation (Fig. 4 and Fig. 5) indicates that the tensile strength of a polyamide containing 60 wt% of glass fibers is equal to the stress at break and strongly depends on the elongation at break, since the curve derivative is positive over its entire course. As a result of the blowing agent addition, the elongation at break was reduced by 15.8% compared to the solid sample. For PA6 GF30 + CBA, the elongation decreased by 15.8%, while for the unfilled polyamide, the elongation decreased by as much as 52%. In the case of all porous samples filled with glass fibers, the brittle fracture occurred in the extreme part of the measuring section (third zone), furthest from the injection point. On the other hand, in the case of composites with a solid structure, the complete fracture occurred in the central area of the measuring section. It was also observed that some solid PA6 samples did not break completely. The decrease in the strength in the end area of the molded specimen may result from the fact that at the end of the material flow path, significant pressure drops occur inside the injection mold, which in turn increases the foaming process intensity [18]. The bigger number of pores and their larger size may reduce the mechanical properties through the action of the notch and reduce the actual surface area affected by the tensile forces.

The specific strength (km), understood as the ratio of the tensile strength (N/m^2) to the specific weight (N/m^3) was calculated. The calculations took into account the density from the place where the sample was broken (zone III of the molded part). The obtained results are shown in Figure 6.

The presence of the porous structure in PA6 did not contribute to the decrease in the average specific tensile strength. In the case of materials containing glass fibers, the average specific strength decreased by 11.3% for PA6 GF30 and by 12.7% for PA6 GF60. Moreover, it was observed that the average specific strength of the chemically foamed PA6

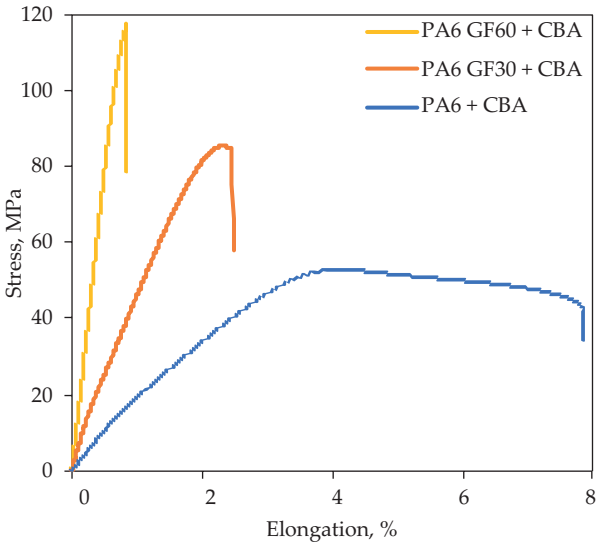

Fig. 5. The relationship between the stress and the elongation of samples

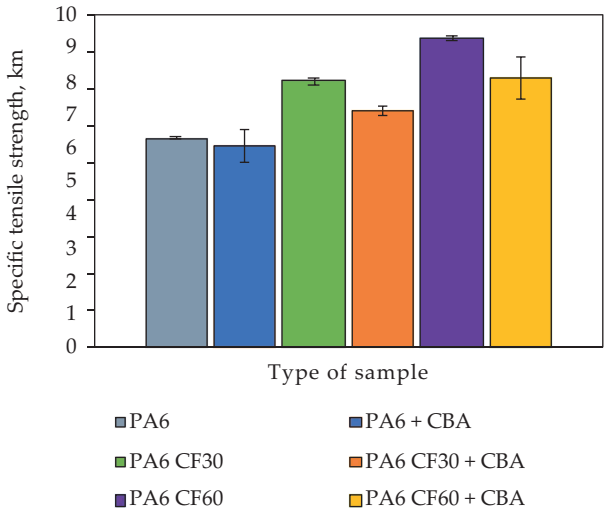

Fig. 6. Specific tensile strength of the tested series of polyamide 6

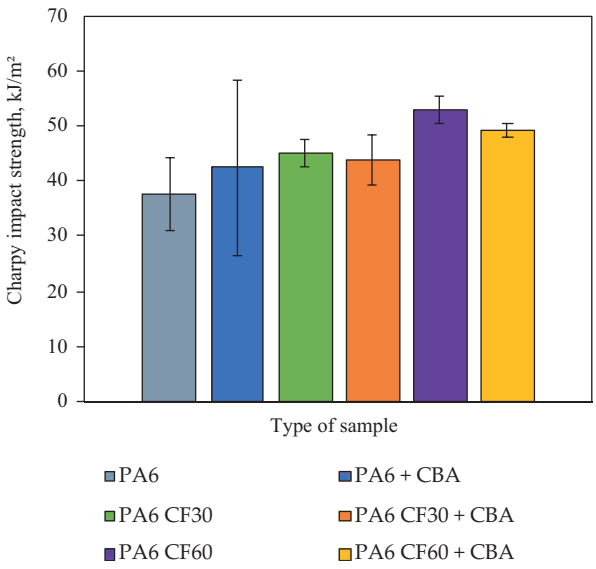

Fig. 7. Average impact strength of the tested PA6 composites with the standard deviation

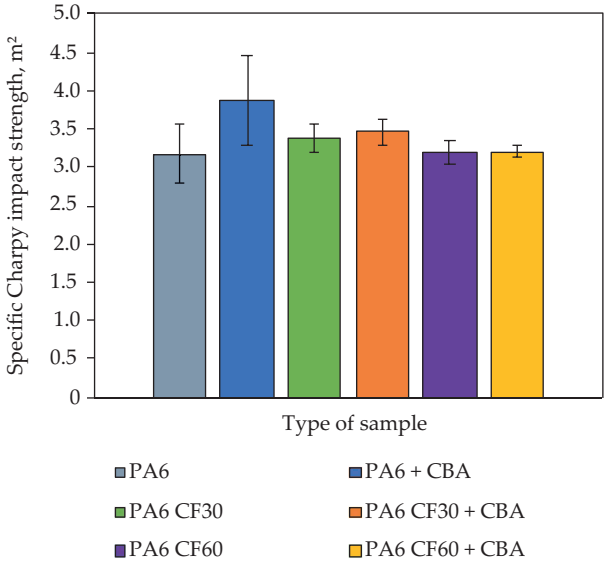

Fig 8. Average specific impact strength of the tested PA6 composites with the standard deviation

GF60 was similar to the specific strength of the non-porous GF30 PA6. The specific strength of unfilled polyamide 6 did not decrease significantly as a result of the foaming process. On the other hand, porous compositions containing glass fibers showed lower specific strength compared to solid materials. This phenomenon may result from the reduction of the contact surface between the fibers and the polymer matrix due to the presence of gas pores in the structure, especially pores, which were constituted directly on the single glass fiber.

Impact strength

The impact strength of the tested PA6 composites is shown in Figure 7. As a result of adding the blowing agent, both to PA6 GF60 and PA6 GF30, the average impact strength of the samples decreased compared to the solid samples. For PA6 GF60 + CBA, the average impact strength was 49.2 kJ/m^2, i.e. it decreased by 7.1% compared to the solid material. For a porous material containing 30% of glass fibers, the average impact strength reduction of 3% was obtained. In the case of unfilled polyamide, it was noticed that not all samples were broken. The blowing agent addition to PA6 increased the average impact strength by 13%. The analysis of the obtained fractures shows that the fracture propagation of the samples begins in the skin layer. According to the SEM images, this zone has little or no pores. Moreover, the pores present in the core of the specimen make it difficult to transfer the stresses from the impact point of the pendulum to the opposite surface where the sample fracture is initiated. As a consequence, it is possible to increase the impact strength of a sample by a blowing agent addition. The increase in impact strength of porous samples has also been described in the literature [19].

The specific impact strength (m^2) understood as the ratio of the impact strength (kJ/m^2) to the specific weight (N/m^3) was calculated. The calculations took into account the local density at the point of the pendulum impact during the impact test (zone II of the sample). The obtained results are shown in Figure 8. The presence of the porous structure in PA6 increased the average specific impact toughness by 32%, and for PA6 GF30 by 6,2%. As a result of the chemical blowing process, the specific impact strength of the polyamide containing 60% glass fiber did not change significantly.

Morphology

The average pore size divided into zones of the sample is shown in Figure 9. The structure in zones I and II for PA6, PA6 GF30 and PA6 GF60 was characterized by pores in a shape similar to spherical. The largest pore sizes in each zone of the sample were observed for the PA6 struc-

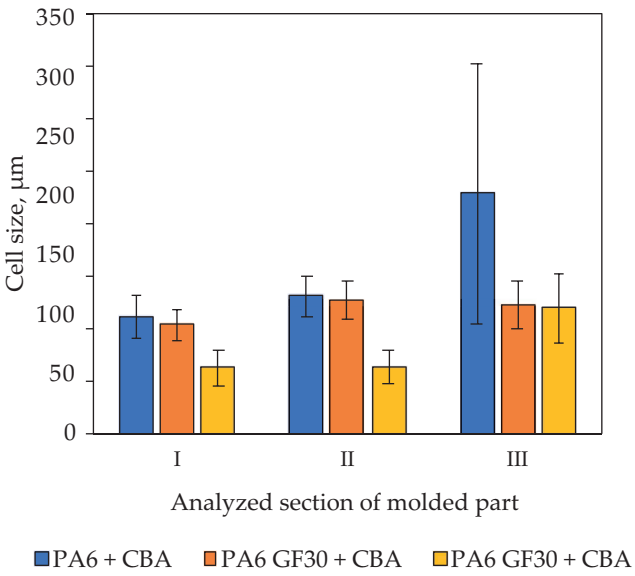

Fig. 9. The average size of the pores in the individual zones core of the tested moldings

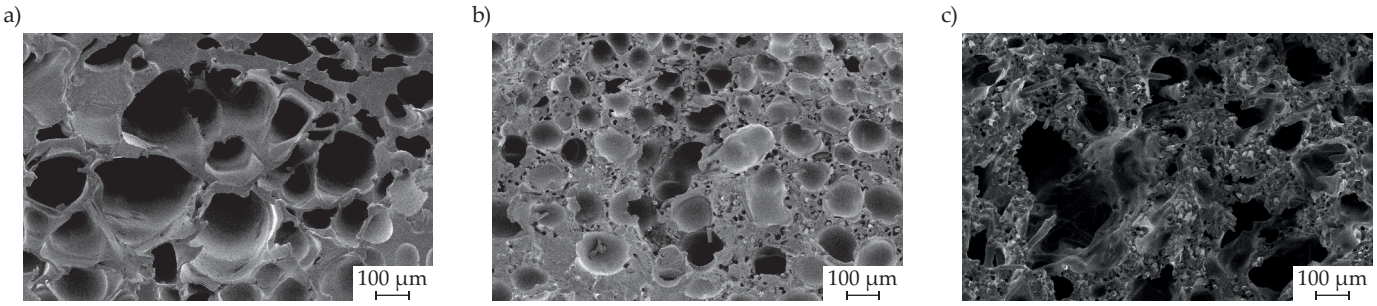

Fig. 10. The core structure of the molded part in zone III: a) PA6 + CBA, b) PA6 GF30 + CBA, c) PA6 GF60 + CBA

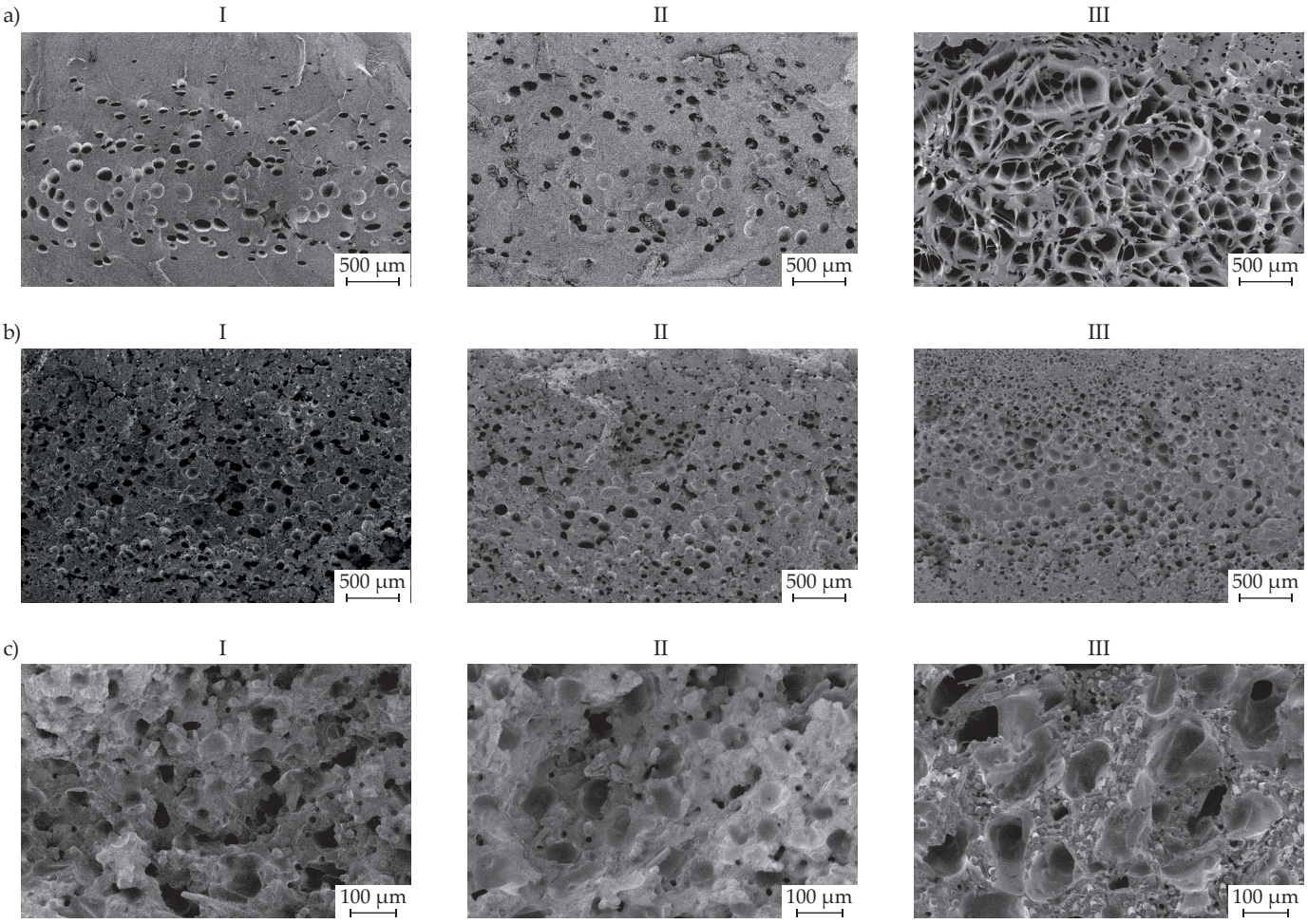

Fig. 11. The core structure of the tested moldings in individual zones: a) PA6 + CBA, b) PA6 GF30 + CBA, c) PA6 GF60 + CBA; I – beginning, II – middle, III – end of the measuring section

ture unfilled with glass fiber. Increasing the amount of this filler may hinder the conditions necessary for pore growth, and also reduce the average pore size at selected locations of the sample core. For the composition containing 30% of glass fibers in zone I, the pores had an average size of 104.2 μm, while in the next zone their average size was 127.7 μm. In the case of PA6 GF60 in zone I, the pores had an average size of 63.3 μm, and in the next zone their average size was 64.0 μm. At the end of the measuring section of the sample (zone III), an increase in the pores size and their irregular shape in the structure of PA6 and PA6 GF60 were observed. Only for PA6 GF30 a slightly smaller average pore size was obtained. The structure of PA6 GF30 is characterized by the highest homogeneity, which is confirmed by the similar average pore size in each zone of the sample. Moreover, it was also observed in the third zone that the pores tend to merge, creating spaces of large size (Fig. 10). This is most evident for a structure with high glass fibers content and free of glass fibers.

Analyzing SEM photos from individual zones of the moldings core (Fig. 11), the dependence of the porous

structure can be noticed, both on the degree of filling with glass fibers and the sampling place for image analysis. As the distance from the injection point (zone III) increased, a more intense foaming effect was observed, characterized by an increase in the number and size of pores. It is influenced by the decreasing pressure of the molten material, resulting from the flow conditions and filling of the mold cavity. It can be assumed that the lower pressure value in the cavity is the main cause of the more intense pore growth.

CONCLUSIONS

The research showed that the foaming process of PA6 moldings containing a high content of glass fibers caused a decrease in their specific tensile strength and did not contribute to a significant change in the specific impact strength. Smaller pores have been observed in the material containing higher glass fiber content. Variability of the formation conditions of the porous structure and its heterogeneity may adversely affect the products functional properties, especially in the case of more complex shapes of the molding cavities. Chemical foaming of PA6 containing a high glass fiber content contributed to the change of mechanical properties and density reduction. Materials with such properties can be used in the automotive, machine and aviation industries.

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