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COMPOSITES THEORY AND PRACTICE

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EXAMINATIONS OF PROPERTIES AND STRUCTURE OF POLYMER COMPOSITES WITH QUARTZ FILLER

The results of examinations of polyamide 6 with quartz sand and glass fiber composites are presented. A composite was made using an extrusion machine collaborating with a granulator. The samples for the examinations were made using a Krauss Maffei KM65-160C1 screw injection moulding machine. Samples from polyamide 6 were also made to compare the properties of the composites and unfilled polymer. Investigations of the mechanical properties: tensile strength and hardness were carried out, and also the structure was determined by means of scanning electron microscopy. The aim of the investigations is to determine the influence of the filler on the composite properties and to receive a new, cheaper constructional material. The properties of the composite were determined from samples produced by injection moulding. As a result of such a method of sample preparation, the properties were conditioned by the processing parameters. The investigations were performed in order to estimate the processing capability and usability of PA6 composites with quartz sand and glass fiber addition. The lowest value of tensile strength of the polyamide 6/quartz sand composite was obtained. An increase in the hardness value of PA6 filled with quartz sand and glass fiber was obtained. The character of the changes of tensile strength and hardness values for the examined materials was evaluated, which proves the essential influence of the filler addition on the mechanical properties change. The tensile strength of the composite increases, while the hardness increases as the content of quartz sand and glass fiber increases.

Keywords: polyamide, glass fiber, quartz sand, composites

BADANIA WŁAŚCIWOŚCI I STRUKTURY KOMPOZYTU POLIMEROWEGO Z NAPEŁNIACZEM CERAMICZNYM

W artykule omówiono wyniki badań właściwości mechanicznych oraz badań struktury kompozytu poliamidu 6 z piaskiem kwarcowym i włóknem szklanym. Celem badań materiałoznawczych było określenie wpływu rodzaju i zawartości zastosowanych napełniaczy ceramicznych na strukturę i właściwości wytworzonego trójskładnikowego kompozytu polimerowego. W tym celu przeprowadzono badania skaningowej mikroskopii elektronowej, wytrzymałości na rozciąganie i twardości kompozytu. Badania przeprowadzono na próbkach kompozytów wytworzonych metodą wtryskiwania przy parametrach przetwórstwa, dla których uzyskano najkorzystniejsze właściwości. Badania wykazały wzrost wartości wytrzymałości na rozciąganie w próbkach z dodatkiem włókna szklanego 15 i 30% z 42 MPa dla poliamidu 6 do odpowiednio 76 i 118 MPa. Zastąpienie 7,5 i 15% włókna szklanego piaskiem kwarcowym powoduje spadek wartości wytrzymałości na rozciąganie do odpowiednio 56 i 63 MPa. Stwierdzono słabą przydatność pomiaru twardości metodą Shore'a w określaniu charakterystyki badanych kompozytów. Wszystkie badane próbki kompozytów charakteryzowały się twardością na zbliżonym poziomie, wynoszącą 79÷80 stopni Shore'a. Zarejestrowano wzrost wartości twardości badanych kompozytów, zmierzoną metodą wciskania kulki w porównaniu do poliamidu 6. Dodatek włókna szklanego 15 i 30% powoduje wzrost twardości ze 102 MPa dla poliamidu 6 do odpowiednio 150 i 162 MPa. Zastąpienie 7,5 i 15% włókna szklanego piaskiem kwarcowym powoduje nieznaczny spadek twardości HB do odpowiednio 146 i 159 MPa. W badaniach mikroskopowych zarejestrowano zmiany strukturalne spowodowane zastosowaniem różnego typu napełniaczy. Stwierdzono pewne wady strukturalne związane z adhezją pomiędzy osnową a użytymi napełniaczami.

Słowa kluczowe: poliamid, włókno szklane, piasek kwarcowy, kompozyty

INTRODUCTION

Polymer composites are used in different industry sectors all over the world and are mainly obtained using extrusion and injection technologies. The use of polymers as a matrix has contributed to the development of specific properties, such as corrosion resistance, low weight, good formability, efficient vibration damping and good thermal and electrical insulation properties. The above characteristics have translated into a variety of applications for these composites [1-6]. The structural components of polymer composites include [1, 3]:

- polymers (thermoplastics, thermosets and elastomers),
- reinforcement fibres (glass fibres, carbon fibres etc.),
- powder fillers.

Polymer composites are multi-component materials that are composed of a polymer that represents the matrix and the second or third component. This study presents the results of examinations of a three-component composite.

Polymers that have been used as a matrix in composite materials help transfer the load to fibres, have an effect on the manufacturing method used and determine the shape of the product and its thermal and chemical properties [3-10].

Most frequently, the matrix in polymeric composites is made of thermosets (thermosetting and chemically setting materials), thermoplastics and, less often, of elastomers. Nearly all the plastics from the group of thermosets and thermoplastics can be reinforced with fibres. The use of thermoplastics as a composite matrix is becoming more and more popular due to uncomplicated processing, the opportunity of longer granulate storage, easy and fast recycling and higher impact resistance and resistance to cracking [1, 3]. The physical modification of thermoplastic polymers with powder or a fibrous filler leads to obtaining a new material with specific properties and structure, which can be dedicated to concrete applications, with particular focus on components used in transport, the arms industry, construction and automotive sectors. Furthermore, polymer composites with ceramic fillers are used for manufacturing sports equipment, medical equipment etc. A number of industrial companies utilize advanced materials such as polymeric composites for manufacturing components for planes and the aerospace industry, with unchanged properties compared to previously used materials. The automotive industry also uses polymer composites since they exhibit good thermal, mechanical, physical and functional properties or resistance to atmospheric conditions [9].

The aim of the material examinations presented in this paper was to determine the type and content of ceramic fillers on the structure and properties of a three-component polymer composite obtained in the study. The following examinations were carried out in the study: scanning electron microscopy, tensile strength testing and composite hardness testing.

MATERIAL AND METHODS

Specimens of Tarnamid T-27 (PA6) with different contents of ceramic fillers in the form of quartz sand with particle sizes of 0.10/0.16/0.2 containing SiO_2 (99.24%), Fe_2O_3 (0.1%) and carbonates (0.07%) with cut glass fibre with the E symbol made of boron-aluminium-silicon glass with the content of alkaline oxides below 1% were used in the study. Both the quartz sand and glass fibres were covered with a silane preparation. The moulded pieces were obtained using a Krauss Maffei KM65-160C1 injection moulding machine with a feed screw of a diameter of 30 mm and L/D ratio of 23, with three zones, constant pitch over the whole length and clamping force of 650 kN. The basic conditions of specimen injection are compared in Table 1.

TABLE 1. **Injection moulding parameters of composite specimens**
TABELA 1. **Warunki wtryskiwania próbek kompozytów**

Injection moulding parameter	Plastic processed
Nozzle temperature [°C]	265
Mould temperature [°C]	80
Injection pressure [MPa]	100
Clamping pressure [MPa]	45
Clamping time [s]	20
Cooling time [s]	20

The symbols of individual specimens and content percentage of ceramic fillers are presented in Table 2.

TABLE 2. **Symbols of specimens used in study**
TABELA 2. **Oznaczenie próbek do badań**

Specimen No.	Content of quartz sand content [%]	Content of glass fibre content [%]
1	-	-
2	-	15
3	-	30
4	7.5	7.5
5	15	15
6	15	-
7	30	-

The static tensile test was carried out using a tensile strength machine Zwick/Roell Z100 according to current standards. All the specimens were stretched at room temperature with a similar rate of 4.2 mm/min. The Shore method and ball indentation method with a total load of 358 N were employed in the study. A Joel JSM-6610LV scanning electron microscope was also used to examine the microstructure of the specimens. Observations were carried out on specimen cross-sections. In order to obtain a conducting layer, the specimens were sprayed with gold.

RESULTS AND DISCUSSION

Table 3 presents the results of the tensile strength testing. Figure 1 illustrates example diagrams of tensile strength for the specimens made of polyamide and composites with different contents of ceramic fillers.

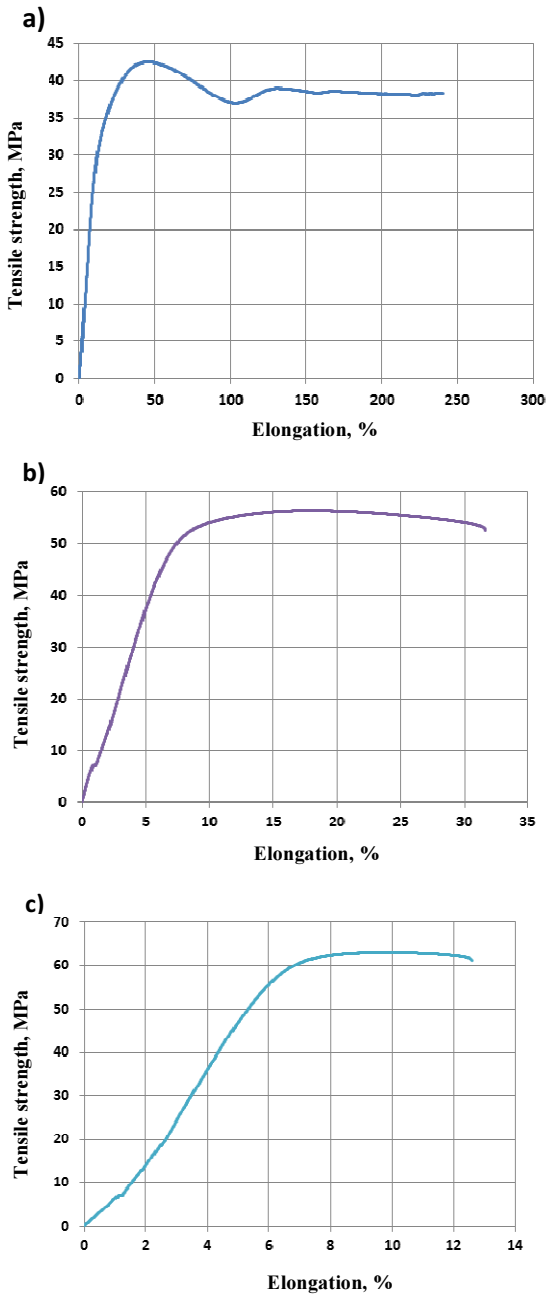

Fig. 1. Tensile strength diagrams: a) PA, b) PA6 + 7.5% PK + 7.5% WS, (c) PA6 + 15% PK + 15% WS

Rys. 1. Wykresy wytrzymałości na rozciąganie: a) PA, b) PA6 + 7,5% PK + 7,5% WS, c) PA6 + 15% PK + 15% WS

TABLE 3. **Results of tensile strength testing**
TABELA 3. **Zestawienie wyników badania wytrzymałości na rozciąganie**

Specimen No.	F_{max} [N]	R_m [MPa]
1 / (Tarnamid T-27)	1750.73	42
2 / (PA6 + 15% WS)	3194.42	76
3 / (PA6 + 30% WS)	4859.57	118
4 / (PA6 + 7.5% PK + 7.5%WS)	2342.93	56
5 / (PA6 + 15% PK + 15%WS)	2598.47	63
6 / (PA6 + 15% PK)	1842.72	44
7 / (PA6 + 30% PK)	1448.21	35

The static tensile strength testing found the lowest value of tensile strength R_m of 35 MPa for the specimen with the addition of 30% quartz sand. This value was lower even than the tensile strength recorded for Tarnamid T-27 (42 MPa). In other cases, the addition of both cut glass fibre and cut glass fibre with quartz sand caused an increase in tensile strength. The highest values were found for the specimens with additions of 15 and 30% cut glass fibre (76 and 118 MPa, respectively). Replacing part of the cut glass fibre with 7.5 and 15% quartz sand caused a decline in the R_m values to 56 and 63 MPa, respectively. This fact should be attributed to the high hardness of the silica particles.

Measurements using the Shore durometer method and ball indentation method used typically for composite and polymer materials were employed to provide a more comprehensive characterization of the mechanical properties of the composites obtained in the study. The results are presented in Figures 2 and 3. The data contained in Figure 2 show that the addition of cut glass fibre and quartz sand causes an increase in hardness in all the specimens to a similar level of 79 to 80 degrees on the D scale of the Shore durometer. Ball indentation turned out to be a more reliable method of hardness measurement.

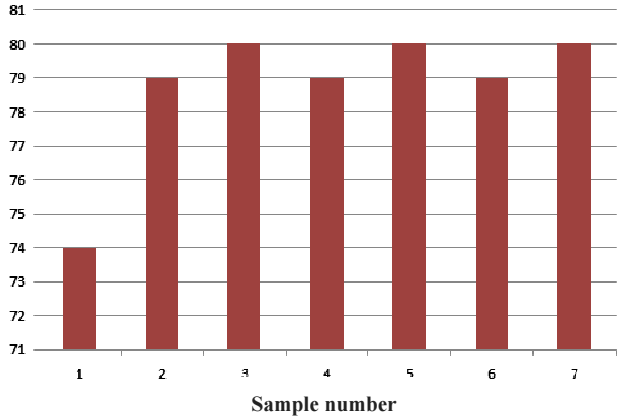

Fig. 2. Hardness measured by means of Shore method (D scale)

Rys. 2. Wyniki pomiaru twardości metodą Shore'a typu D

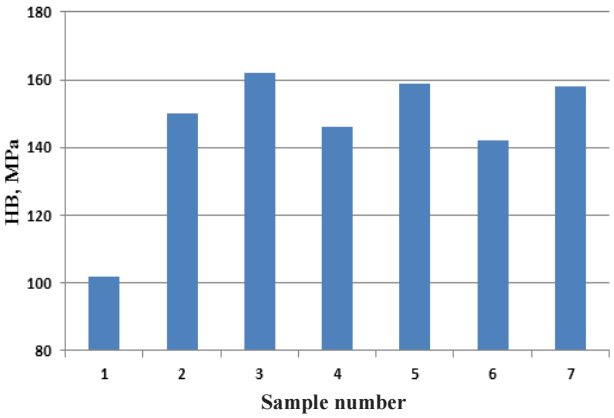

Fig. 3. Hardness measured by means of ball indentation method

Rys. 3. Wyniki pomiaru twardości metodą wciskania kulki

The data presented in Figure 3 show that the mean hardness of Tarnamid T-27 was 102 MPa. The additions of 15 and 30% of glass fibre caused an increase in hardness to 150 and 162 MPa. Replacing a part of the cut glass fibre with 7.5 and 15% of quartz sand caused an insignificant decline in hardness to 146 and 159 MPa, respectively. Slightly different values of hardness were obtained for the specimens with 15 and 30% of quartz sand and were 142 and 148 MPa, respectively.

The analysis of specimen cross-sections using a scanning electron microscope (SEM) revealed that part of the cut glass fibres was not only fractured during tensile tests but they were also removed from the matrix, which was reflected by numerous pores (spaces left after fibres, see Fig. 4).

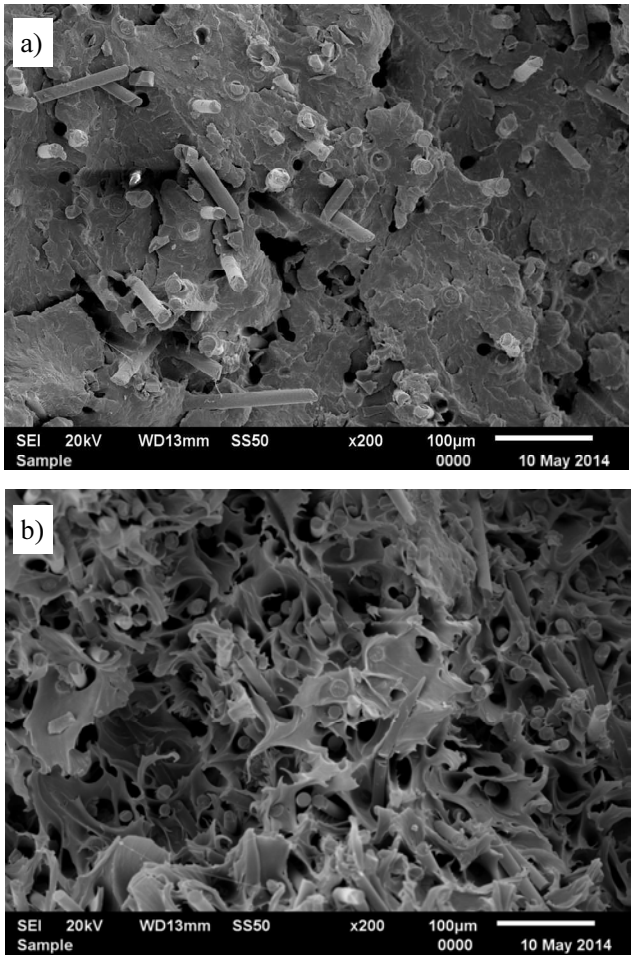

Fig. 4. Fracture microstructure: a) specimen No. 2 (PA6 + 15% WS), b) specimen No. 3 (PA6 + 30% WS); magnitude 200x

Rys. 4. Mikrostruktura przełomu: a) próbki nr 2 (PA6 + 15% WS), b) próbki nr 3 (PA6 + 30% WS); powiększenie 200x

This fact might suggest poorer adhesion between the components. A similar observation was made in the case of the specimens with the addition of quartz sand where lack of good contact between the matrix and the quartz sand particle surface is noticeable in several places.

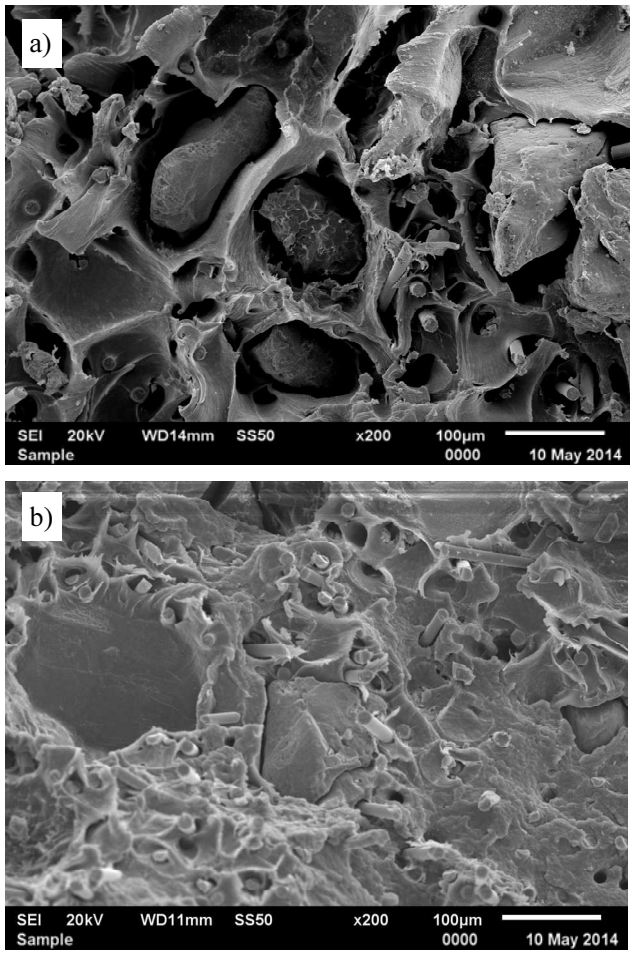

Fig. 5. Fracture microstructure: a) specimen No. 4 (PA6 + 7.5% PK + 7.5% WS), b) specimen No. 5 (PA6 + 15% PK + 15% WS); magnitude 200x

Rys. 5. Mikrostruktura przełomu: a) próbki nr 4 (PA6 + 7,5% PK + 7,5% WS), b) próbki nr 5 (PA6 + 15% PK + 15% WS); powiększenie 200x

CONCLUSION

The study found an increase in tensile strength for the specimens with the addition of glass fibre of 15 and 30% from 42 MPa for polyamide 6 to 76 MPa and 118 MPa, respectively. The increase in the quartz sand content causes a decline in tensile strength. Replacement of 7.5 and 15% glass fibre with a respective amount of quartz sand causes a decline in tensile strength to 56 and 63 MPa, respectively. The poor reliability of the Shore durometer measurement method was found during determination of the properties of the composites studied. All the composite specimens were characterized by hardness at a similar level of 79 to 80 degrees on the Shore scale. An increase in hardness of the composites measured by means of the ball indentation test was observed compared to polyamide 6. The addition of glass fibre of 15 and 30% causes an increase in hardness from 102 MPa for polyamide 6 to 150 and 162 MPa, respectively. With the increase in quartz, an increase in hardness of the composite was observed. Replacement of 7.5 and 15% glass fibre with a respective amount of quartz sand causes an insignificant

decline in HB hardness to 146 and 159 MPa, respectively. The microscopic examinations found structural defects resulting from problems in adhesion between the matrix and fillers used in the study.

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