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COMPOSITES THEORY AND PRACTICE

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STRUCTURAL EXAMINATIONS OF WOOD-POLYMER COMPOSITES

This paper presents the results of examinations of wood-polymer composites based on mixed polypropylene (PP) and sawdust. Composites with a different volumetric content of wood were prepared (25, 50 and 70% filler). Injection molding technology was used to obtain the research samples. The authors also employed X-ray diffraction, an optical microscope and a transmission scanning microscope. Based on the X-ray diffraction analysis, the presence of several polymorphic forms of the polypropylene were found in the composites used in the study. The crystalline fraction of the matrix in the studied composites is composed of an α phase which crystallizes in a monoclinic lattice and a β phase which crystallizes in a hexagonal system. Microscopic analysis revealed irregular distribution of the wood particles in the matrix of the tested composites.

Keywords: wood-polymer composites, X-ray diffraction

BADANIA STRUKTURY KOMPOZYTÓW POLIMER-DREWNO

W artykule przedstawiono wyniki badań struktury kompozytów polimer-drewno, gdzie jako napełniacz wykorzystano trociny drzew liściastych i iglastych. Osnowę kompozytu stanowił termoplastyczny polipropylen. Do badań przygotowano kompozyty o różnej frakcji zastosowanych trocin i zmieniającą się zawartością napełniacza (25, 50 oraz 70% obj.). Próbki przygotowano z wykorzystaniem technologii wtryskiwania. Do badań wykorzystano dyfraktometr rentgenowski oraz mikroskopię optyczną i elektronową mikroskopię skaningową. Przeprowadzone badania rentgenowskie pozwoliły zidentyfikować fazy krystaliczne występujące w polimerowej osnowie. Widoczne na dyfraktogramach refleksy dyfrakcyjne pochodzą od odmian polimorficznych polipropylenu: α (jednoskośna) i β (heksagonalna). Analiza mikroskopowa wykazała nierównomierne rozmieszczenie cząstek drewna w osnowie badanych kompozytów.

Słowa kluczowe: kompozyty polimer-drewno, badania rentgenowskie

INTRODUCTION

The worldwide crisis and increasingly high prices of raw materials has intensified efforts to find more innovative and cheaper solutions in such domains of science as material engineering. For this reason, increasingly more composites with plant-based fillers have been manufactured in recent years. A good example is wood-plastic composites (WPC). These composites are frequently composed of a matrix made of thermoplastic polymers and ground wood particles. These materials can be used wherever traditional wood in its pure form is unsuitable since WPC show many advantages, such as aesthetical aspects, little or lack of water absorptivity, lack of susceptibility to warping and resistance to atmospheric factors. With the above properties, these composites are gaining in popularity in different sectors of such industries as the construction or automotive industry.

WPC composite products are often obtained by means of the methods of extrusion or injection methods. An important factor in processing of wood-plastic composites is the temperature, which should not be greater than 190 to 200^{o}C. The application of higher processing temperatures might lead to thermal decomposition of the wood in the plasticizing system of the injection molding machine. Therefore, WPC use thermoplastic polymers as a matrix, with a melting point below 200^{o}C. They include polypropylene (PP), polyethylene (PE) and polyvinyl chloride (PVC) [1-4]. When choosing the material for matrices in WPC processed with the injection molding method, another important factor is the mass flow rate (MFR > 15 g/10 min). Adding a filler might cause a considerable reduction in the MFR value, which in consequence might lead to incomplete filling of the mold cavity [5, 6].

MATERIALS AND EXPERIMENTAL PROCEDURE

The present study was based on a polypropylene with a mass flow rate of MFR = 25 g/10 min (manufac-

tured by Slovnaft). The filler, in the form of sawdust (that originating from both coniferous and deciduous trees), was characterized by a high water content. Therefore, before processing, it was dried for 6 hours at a temperature of 105°C. The material in the form of sawdust was sieved and divided into fractions using sieves with a mesh size of 1.5 and 2 mm. When sieving, the wood dust was separated. The fractions prepared using this procedure were then added to the plastic so that the sawdust content was 25, 50 and 70% of the total volume of the composite. The components of WPC were measured in a laboratory vessel and weighted by means of laboratory scales with the accuracy of 0.002 kg. The density of the dry wood was assumed at the level of 480 kg/m^3. After preparation of suitable proportions of the matrix and filler, the components were mixed for about 4 minutes in a drum mixer. The research samples were prepared by means of a Krauss - Maffei KM - 65 130C4 injection molding machine for thermoplastic materials.

The aim of the study was to analyze the structure of wood-plastic composites. Composites with a polypropylene matrix with different degrees of sawdust filler and different sawdust fractions were used in the study. Examination of the WPC structure was based on an X-ray diffractometer, optical microscopy and scanning electron microscopy.

The examinations by means of X-ray diffraction analysis were carried out using a SEIFERT XRD 3003 T – T diffractometer. The authors used filtered radiation from acuprum anode lamp with radiation wavelength $\lambda_{CuK\alpha}$ = 0.15418 nm. The operating parameters for the lamp were: U = 40 kV, I = 30 mA. A stepwise method of measurement was employed for scattering angles 2θ from 10° to 40° with a step of 0.1°. A numerical computer analysis was carried out for the obtained diffraction curves using Analyze software. The analysis consisted in decomposition of the obtained curves into diffraction reflections from polypropylene crystalline phases and diffraction maximum of the amorphous phase and determination of the best match to the experimental curves.

Observation of the metallographic specimens was carried out by means of an Axiovert 25 optical microscope with digital image recording and magnification ranging from 100 to 250x. Furthermore, the authors carried out observations using a Joel JSM 6610LVz scanning electron microscope with the digital image recording option. Before the examination, the specimens were sprayed with a layer of gold.

RESULTS AND ANALYSIS

Phase composition in a composite considerably affects its functional properties, hence the necessity for detailed determination of this parameter. In practice, phase analysis (both qualitative and quantitative) is performed using X-ray diffraction examinations. Based on this analysis, the authors determined the angular locations of the reflection points and diffraction maximums and their full widths at half maximums and integral intensities. The content of the propylene crystalline phase (crystallinity degree) was calculated as the quotient of the sum of the integral intensities of crystalline reflections to the integral intensity of the diffraction curves. The quantitative analysis revealed that the value of the crystallinity degree in all the studied specimens was above 70%. There are several strong diffraction reflections in the diffractograms obtained in the study (Figs. 1 and 2). Based on the literature data [7-10] concerning locations of characteristic diffraction reflections for polypropylene, they were identified as reflections from the polymorphic forms of polypropylene, such as α (monocyclic) and β (hexagonal) forms and from the amorphous polypropylene phase. The diffraction reflections presented in the diffractograms originate from lattice planes: (110), (040), (130) as well as (111) and (131), which are typical of the α form of polypropylene and from the crystallographic plane (300). It is a diffraction reflection characteristic of the β form of polypropylene.

Based on the obtained values of full width at half maximum and using the Scherrer equation [11], the authors calculated the dimensions of the L_{hkl} crystallites in the directions perpendicular to the lattice planes with Miller indices of (110), (040), (130), (111) and (131) for the α phase and (300) for the β phase. The calculated dimensions of crystallites for phases α and β of polypropylene are presented in Table 1.

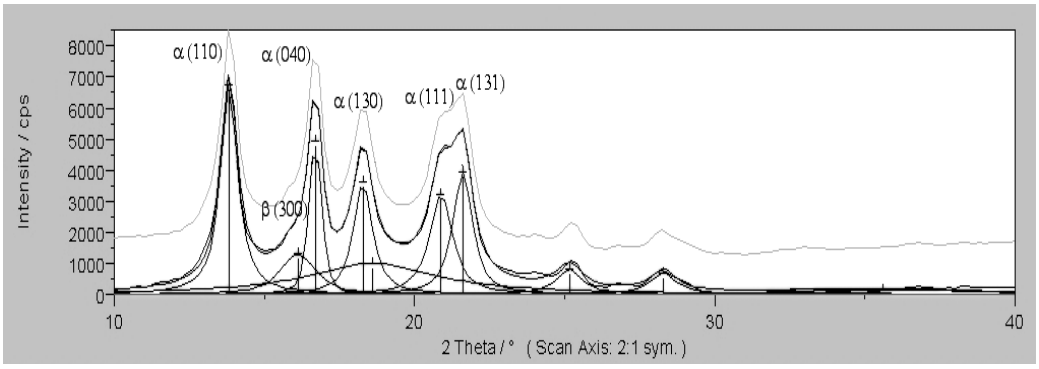


Fig. 1. X-ray diffraction sample of WPC: 75%PP/25% filler, fraction 1.5

Rys.1. Dyfraktogram rentgenowski kompozytu WPC: 75%PP/25% napełniacz, frakcja 1,5

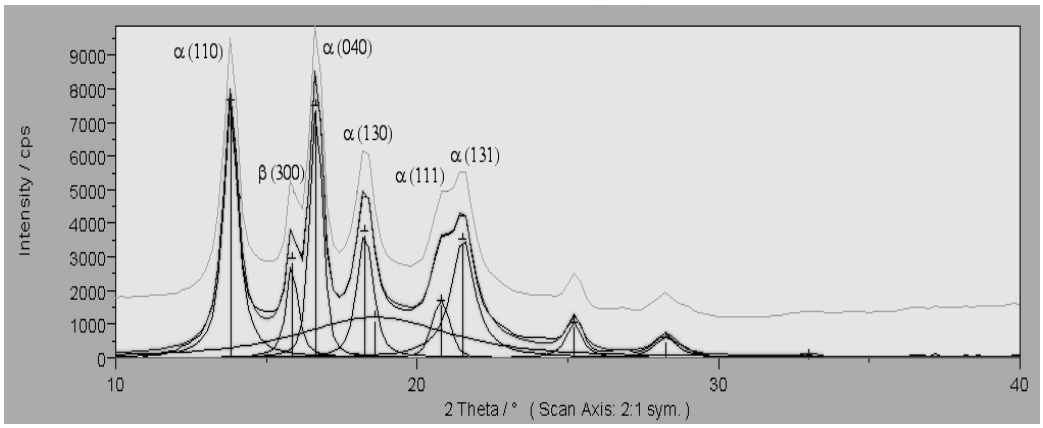


Fig. 2. X-ray diffraction sample of WPC: 25%PP/75% filler, fraction 2

Rys. 2. Dyfraktogram rentgenowski kompozytu WPC: 25%PP/75% napełniacz, frakcja 2

TABLE 1. **Size of crystalline polymorphic form α and β of polypropylene determined by means of X-ray diffraction**

TABELA 1. **Wielkości krystalitów odmian polimorficznych α i β polipropylenu wyznaczone metodą dyfrakcji rentgenowskiej**

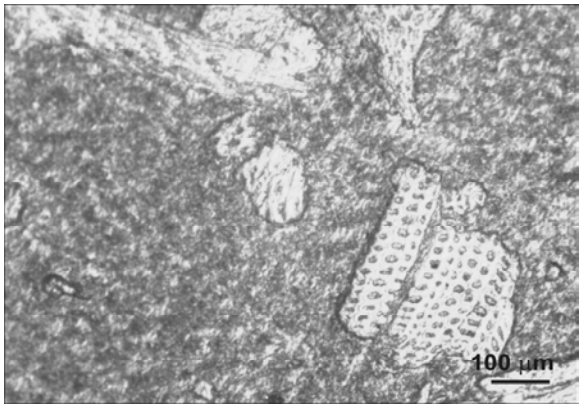
Material	Crystalline size, L_{hkl} [nm]					
	α - monocyclic (hkl)					β (hkl)
	(110)	(040)	(130)	(111)	(131)	(300)
75%PP/25% filler, fraction 1.5	11.80	16.17	10.82	8.53	9.99	5.67
75%PP/25% filler, fraction 2	12.78	16.92	11.78	8.95	9.57	6.56
50%PP/50% filler, fraction 1.5	14.16	16.14	12.61	8.84	9.64	13.47
50%PP/50% filler, fraction 2	14.16	16.52	12.50	8.62	9.57	16.72
30%PP/70% filler, fraction 1.5	12.97	16.32	12.85	7.38	13.60	15.08
30%PP/70% filler, fraction 2	14.31	16.32	12.97	8.62	11.86	19.54

Addition of the filler in the form of sawdust facilitates nucleation of the β form of polypropylene. An increase in the fraction of the volumetric filler causes an increase in the mean size of crystallites in the obtained β phase. It was also found that the use of wood with fraction 2 as a filler is conducive to the growth of higher crystallites compared with the samples where a lower-fraction filler was used. Furthermore, changes in the reflection intensity (300) from the β form indicates that this form is less stable than the monocyclic α modification.

Example results of structural examinations obtained by means of optical and electron microscopy are presented in Figure 3.

The presented microstructures contain individual wood particles which are unevenly distributed throughout the polymer matrix. The cellular structure of the wood particles was revealed in both the optical and scanning electron microscopic pictures.

a)



b)

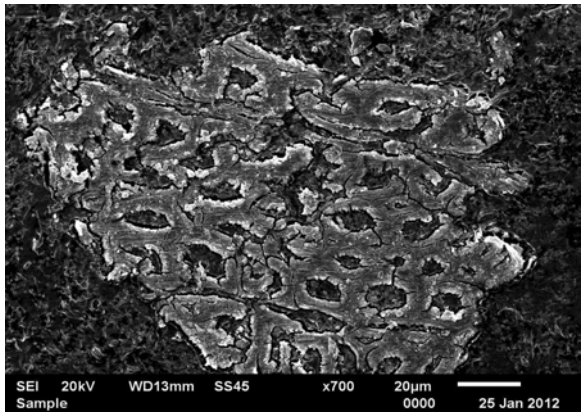


Fig. 3. Microstructure of tested WPC: a) 50%PP/50% filler, fraction 2, optical microscopy, b) 30%PP/70% filler, fraction 1.5, scanning microscopy

Rys. 3. Mikrostruktura badanych kompozytów WPC: a) 50%PP/50% napełniacza, frakcja 2, mikroskopia optyczna, b) 30%PP/70% napełniacz, frakcja 1,5, mikroskopia skaningowa

CONCLUSION

The presence of two polymorphic forms of polypropylene was found in the polymer matrix of the WPC examined in the present study: α (monocyclic) and β (hexagonal) modifications. Higher fractions of the used filler are conducive to the growth of insignificantly larger β phase crystallites. The microscopic observations revealed the non-homogeneous structure of the studied composites.

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