Journal of Sustainable Mobility (ISSN 2053-2350)

Citation Details

Citation for the version of the work held in 'OpenAIR@RGU':

SILVA, F., SACHSE, S., ZHU, H., PIELICHOWSKI, K., LESZCZYNSKA, A. and NJUGUNA, J., 2014. The effect of matrix and reinforcement material selection on the tensile properties of hybrid composites. Available from *OpenAIR@RGU*. [online]. Available from: http://openair.rgu.ac.uk

Citation for the publisher's version:

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The Effect of Matrix and Reinforcement Material Selection on the Tensile Properties of Hybrid Composites*

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In order to develop advanced materials as lightweight structures three-phased composites were prepared using nano-fillers, nano-silica (nano-SiO_2) and nano-clay and micro glass-spheres to reinforce two kinds of glass-fibre reinforced thermoplastic matrices, polypropylene (PP) and polyamide (PA6). These novel composites were examined for: 1) morphological property using Transmission Electron Microscopy (TEM) and X-ray diffraction; 2) the nature of surface fracture using Scanning Electron Microscopy (SEM); and 3) the mechanical property using Instron 5500R universal testing machine. The results showed that for glass-fibre-PA6 composites, the addition of MMT and glass-spheres enhanced the stiffness, whereas the addition of nano-SiO_2 increased the maximum elongation. In contrast the addition of the secondary filler had little effect on the mechanical properties of PP composites.

- Nanocomposites
- Polymer-matrix composites (PMCs)
- Short-fibre composites
- Injection moulding

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* This work was funded by the European Commission (FP7 Project- CP-FP; Project Reference: 228536-2). The authors also gratefully acknowledge the EPSRC for providing testing equipment, as well as Grado Zero Espace SRL and Laviosa Chimica Mineraria SPA for the preparation of the materials.

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THERMOPLASTIC POLYMERS SUCH AS POLYPROPYLENE (PP) and polyamide (PA6) are widely used in the automotive industry, as they exhibit a valuable combination of good mechanical properties, processability and low cost [1]. On the other hand, application of thermoplastics in lightweight structures is limited because of their relatively poor mechanical properties [2]. Addition of micro-size fillers is a common method to increase the mechanical properties, such as stiffness, strength or impact resistance [3]. However, the increase in rigidity by addition of micro-fillers is usually at the cost of reduced ductility [4]. This effect is caused by the existence of stress concentration regions in a vicinity of the reinforcement. The use of nano-size fillers has been shown to cause a significant reduction of stress concentration regions, while maintaining or even improving ductility [5]. Moreover, it has been proved that addition of nano-sized fillers, rather than micro-sized fillers, can significantly enhance the mechanical properties of polymeric materials at low filler content. This is usually achieved at loadings of 0.5–5% [6–9], whereas in the case of micro-fillers the reinforcing effect is achieved at loadings typically higher than 20% [10].

In order to enhance physical and mechanical properties of a resin material, silica (SiO_2) nano-particles can be introduced [11]. Several researchers have reported significant improvements in properties such as tensile strength, strain to failure, Young's modulus and impact strength after reinforcing the material with silica nano-particles [12–15]. In the manufacturing process of nanocomposites, an appropriate dispersion of nano-filler within a matrix is very important. The presence of agglomeration regions, which can act as stress concentrators, may lead to an impairment of composite performance [16; 17]. Zheng et al. [18] compared the mechanical properties of nanocomposites with different quality of filler dispersion. They found that the material with uniformly distributed particles exhibited significant property improvements, while that with poorly distributed particles exhibited only a slight improvement in the mechanical properties.

A second commonly used nano-filler is nano-clay in the form of plates, so named as they have only one dimension in nanometre scale. The most commonly used nano-plate material is silicate clay mineral, Montmorillonite (MMT), due to its high surface area and surface reactivity [19]. To achieve the best mechanical properties of MMT reinforced composite, a large surface contact area between MMT and the resin material, ideally ~ 1000 m^2/g, is required. Thus MMT has to disperse well within a matrix, without significant breakage. In practice, this is often difficult to achieve because of the large shear stresses induced during the mixing process, resulting in an aspect ratio of 30 to 300 [20].

As nano- and micro-sized fillers can reinforce polymer property in different aspects, combining both into one three-phase composite is increasingly considered as a promising solution for future lightweight structures. Despite the fact that the mechanical properties of two phase composites have been extensively studied in the available literature, it is very difficult to predict how a three phase material will behave under the same conditions. This present study aimed to investigate the morphology and the mechanical properties of PP- and PA6-based three phase composites, filled with different nano or micro materials and short glass-fibres. The effects of the polymer matrices and reinforcement materials on the mechanical properties of injection moulded composites were studied and discussed in detail.

Experimental

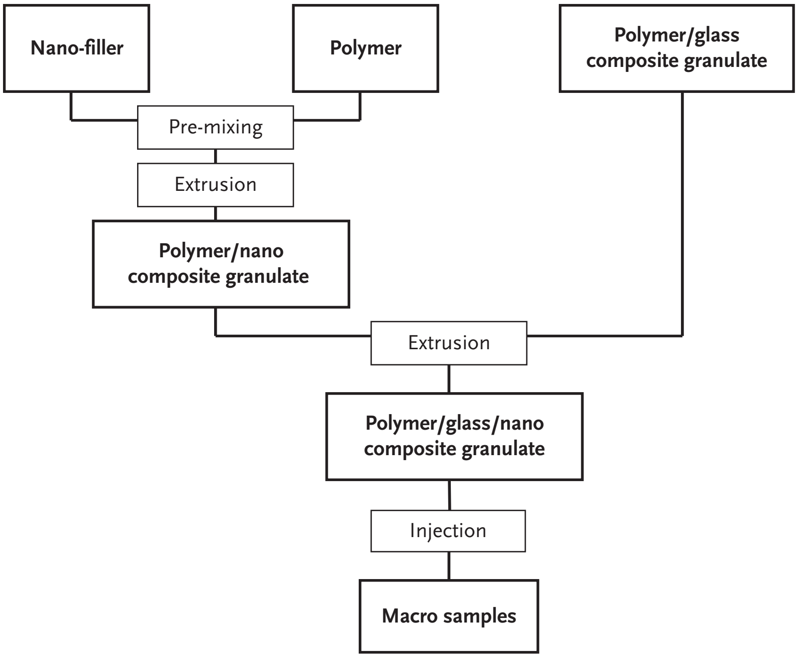
Materials

Polypropylene (PP) Moplen HP500J and polyamide 6 (PA-6) Tarnamid T-30 were obtained from Basell Polyolefins and Zakłady Azotowe w Tarnowie-Mościcach, Poland, respectively. MMT including organically modified MMT Dellite 72T, designed for nonpolar polyolefin matrices, and Dellite 43B, designed for polar matrices, were obtained from Laviosa. Fumed silica nanoparticles AEROSIL 974 with hydrophobic properties (surface modified with dichlorodimethyl silane (DCMS) for nonpolar polyolefin matrices), and AEROSIL 200 with hydrophilic properties for polar polymer were obtained from Degussa. Glass-fibre reinforced polyamide MM-PA I 1F30 and polypropylene MM-PP BI 24, as well as glass-spheres reinforced polyamide MM-PA I 1K30 and polypropylene MM-PP HE25 were supplied by MACOMASS Verkaufs AG Germany.

Preparation of nano-composites

Preparation of nano and glass reinforced polymer composites was conducted in three main steps: preparation of nano-composite granulate, mixing and extrusion of nano and glass reinforced composite granulates and injection moulding of macro-samples. A flow chart showing the preparation process is presented in Figure 1.

Figure 1 Samples preparation process



In order to warrant the highest homogeneity of the composition, nanofiller and polymeric matrix both in solid (powder) form, were premixed before extrusion. This activity was performed by the use of a turbomixer with rotatory blades. The pre-mixing phase consisted of two steps, the first one at lower speed (1,500 rpm) and the second one at higher speed (3,000 rpm). This choice was made in order to ensure the maximum homogeneity of the premix and, on the other hand, to subject the polymer to a small temperature stress, to improve binding between polymeric matrices and added reinforcements. Subsequently the premixed materials were fed into the twin-screw extruder at a constant predefined rate. Once properly fed to the extruder, the materials were processed according to the predefined temperature profile. The choice of a (pilot plant) corotation twin screw extruder characterised by a high length/diameter ratio (52) warranted the best dispersion and highest homogenisation within the composite materials in the melt phase.

Fabrication of macro-samples

Fabrication of glass-fibre and nano reinforced composite structures has been conducted in two steps. In the first one, granulates of the nano-particle reinforced polymers and glass-fibre reinforced polymers were mixed in the extruder. Nanocomposites based on polypropylene were obtained at temperatures 180, 180, 185, 185, 190, 190, 190, 190, 195, 195, 200, and 200°C of 1st–12th barrel zones, respectively, with melt pressure of 35–40 bar. The processing parameters for extrusion of PA6-based nanocomposites were: temperatures of barrel zones (1st–12th) 240, 250, 250, 250, 260, 260, 260, 270, 270, 270, 280, 280°C and melt pressure 30–35 bar. The screw speed was set at 400 rpm for all prepared compositions. As a result eight different composite materials were prepared as shown in Table 1. In the second step, the macro-samples were produced using injection moulding machine (Engel ES200/60 HL ST).

Table 1 Manufactured composites

PP composites				
Name	**PP/GF**	**PP/GF/GS**	**PP/GF/SiO$_2$**	**PP/GF/MMT**
Matrix	PP	PP	PP	PP
1st filler [wt%]	GF [30%]	GF [30%]	GF [30%]	GF [30%]
2nd filler [wt%]	–	GS [12%]	SiO2 [2%]	MMT [2%]
PA6 composites				
Name	**PA/GF**	**PA/GF/GS**	**PA/GF/SiO$_2$**	**PA/GF/MMT**
Matrix	PA	PA	PA	PA
1st filler [wt%]	GF [30%]	GF [30%]	GF [30%]	GF [30%]
2nd filler [wt%]	–	GS [12%]	SiO2 [2%]	MMT [2%]

Characterisation

Microscopy

TEM images of various nanocomposites were obtained at 120 kV, using Philips CM20 scanning Transmission Electron Microscope. In order to prepare TEM samples, the composite granulates were compression moulded into thin films. Afterwards, the films were embedded into epoxy resin and cured overnight at room temperature. Finally, ultra thin sections were cut using ultra microtome and deposited on copper grids.

The fracture surface of the tensile bars was examined with FEI XL30 field emission scanning electron microscope (SEM). The operating voltage was in the range of 10–20 kV and the specimens were gold sputtered to minimise charging of the sample.

X-ray diffraction

X-ray diffraction experiments were carried out on a Philips X-Pert diffractometer, with graphite monochromator placed in the front of detector – $\lambda_{Cu} = 1.5418$ Å. The measurements used for determination of crystallographic structure of polymers were carried out at an angle range of 3–60° at 0.1° step. Material used for all structure analysis was in a shape of rectangle bars made by injection moulding.

Quasi-static and dynamic tensile tests

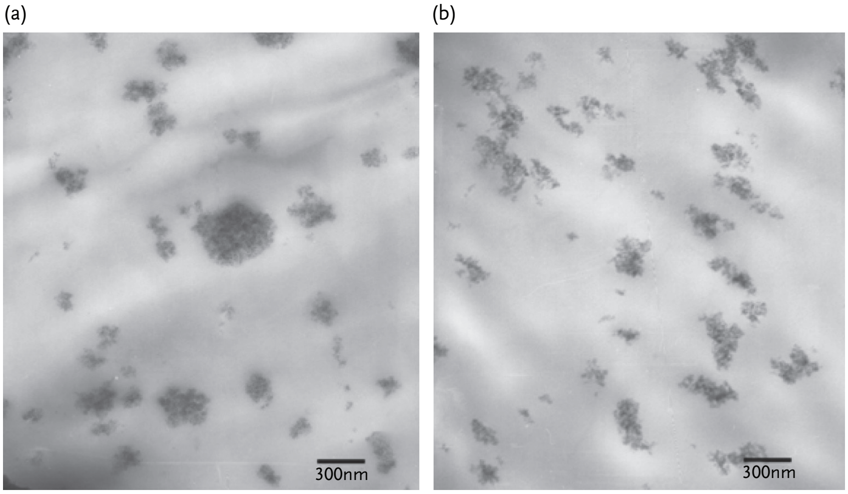
Quasi-static and dynamic tensile tests were carried out using Instron 5500R universal testing machine. All experiments were conducted according to ISO-527 standard, using specimen type A. Samples were machined from injection moulded plates in two different directions: longitudinal (along fibres) and transverse (cross-fibre). Five samples of each material were tested at two different crosshead speeds, 0.1 mm/min and 1000 mm/min. The load was measured using a 100 kN load cell. In order to measure the strain a laser extensometer was used.

Results and discussion

Morphology studies

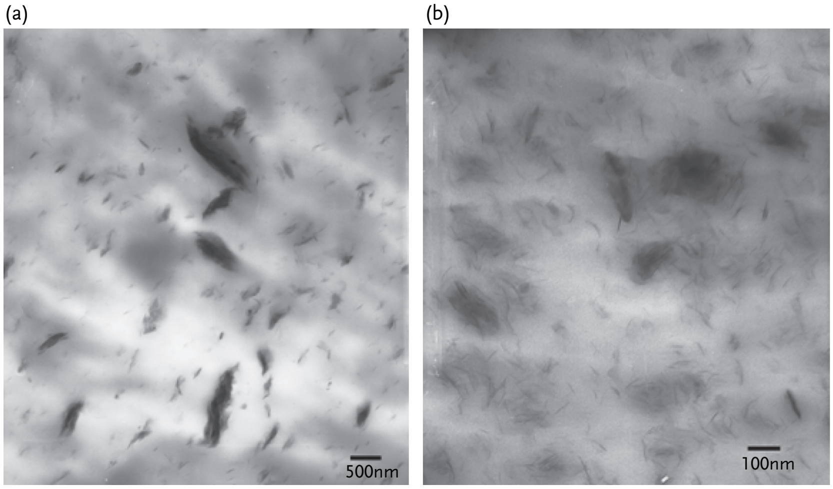
Representative TEM images of SiO_2 filled polymer composites are shown in Figure 2. A generally good state of dispersion was observed in both PP and PA matrices. There was a visible tendency of the particles to create small clusters within the polymers. Some of these clusters tended to create larger structures known as agglomerates. The size of the agglomerates was in the range of 50 to 300 nm, whereas the size of the small clusters was in the range of 10 to 50 nm. Contrary to the PA composite, the agglomerates in the PP matrix were more likely to form chain-like branched structure.

Figure 2 TEM images of SiO_2 reinforced (a) PA (b) PP



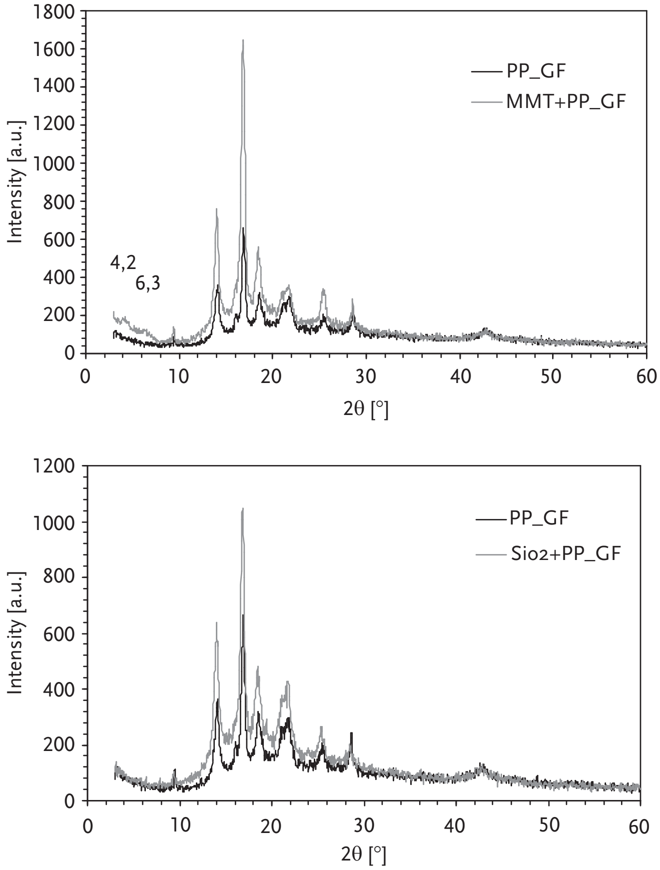
The morphology of MMT filled composites is shown in Figure 3. From the micrographs it could be observed that in both matrices, MMT was partially intercalated and exfoliated. Therefore, the polymer was present between the nano-plates, which were randomly distributed. However, there were visible agglomerates within the polymer, meaning that some of the nano-plates were not fully separated from each other. Comparing the morphology of the PP and PA based composites it was observed that agglomeration regions were more significant in PA based composites. Moreover, the size of these agglomerates was observed to be bigger in relation to those in the PP matrix, and therefore, the interlayer spacing between the plates was reduced as well. The aspect ratio of nano-plates was in the range from 1:10 to 1:50, in both composites.

Figure 3 TEM images of MMT reinforced (a) PA (b) PP



Structure analysis of glass-fibre reinforced panels fabricated from PP and PA6 based nanocomposites was carried out using WAXD method. Figures 4–6 compare the diffractograms of dust produced from glass-fibre reinforced panels composed of neat polymer and those containing MMT, SiO2 and GS nanofillers, in a range of 2θ from 3 up to 60°. The diffraction peaks observed at 4.2° and 6.3° for PP/GF/MMT sample can be correlated with the presence of lamellar intercalated arrangement of MMT in produced powder. The crystal structure of PP was not significantly affected by the mechanical processing and resembled the pattern of neat PP in a bulk form. Stronger peaks were observed for dust of nanocomposite panels, indicating more perfect and higher content of polypropylene crystal phase. There are no changes in basic diffraction patterns of dusts prepared of glass-fibre reinforced panels independently on the matrix type; namely, dusts made of PA6 panels and PA6 based nanocomposite panels showed similar course. The application of nanocomposite materials as a matrix for glass-fibre reinforced panels changed the susceptibility of the material to undergo structural changes under mechanical degradation. However in the presence of nanoparticles, especially MMT, the peak derived from the γ crystal phase of PA-6 was strengthened, indicating higher content of polymer crystal phase in dust made of nanocomposite panels. Such robust crystal morphology could origin from the processing condition, where polymer was subjected to high shearing and cooling rates which favoured the formation of γ crystals.

Figure 4 WAXD diffractograms of dust produced from (a) PP and PP/MMT (b) PP and PP/SiO_2 panels reinforced with glass-fibre



Tensile properties

Tensile properties of eight different glass-fibre reinforced polymer composites, filled with nano and micro particles, are presented in Table 2. It was shown that the addition of a second filler caused a decrease in modulus, strength and elongation to brake. The only exception was the addition of SiO_2 nanoparticles to PP-based composite, which resulted in the improvement in elongation to brake. The biggest drop in all parameters of PP composite was caused by the presence of GS micro-filler. The negative effect of the secondary reinforcement may be attributed to the properties of the PP material itself. Good bonding between PP matrix and reinforcement is difficult to achieve without an appropriate coupling agent, which assures a chemical coupling between non-polar polymer and polar reinforcement [21]. Another issue associated with PP composites is to achieve a good dispersion of nano-reinforcement within a matrix. Usage of untreated nano-reinforcement without a compatibilizer in PP matrix can lead to a poor dispersion and creation of the agglomeration regions, and therefore even lower ability of the material to achieve a strong interphase bonding [22].

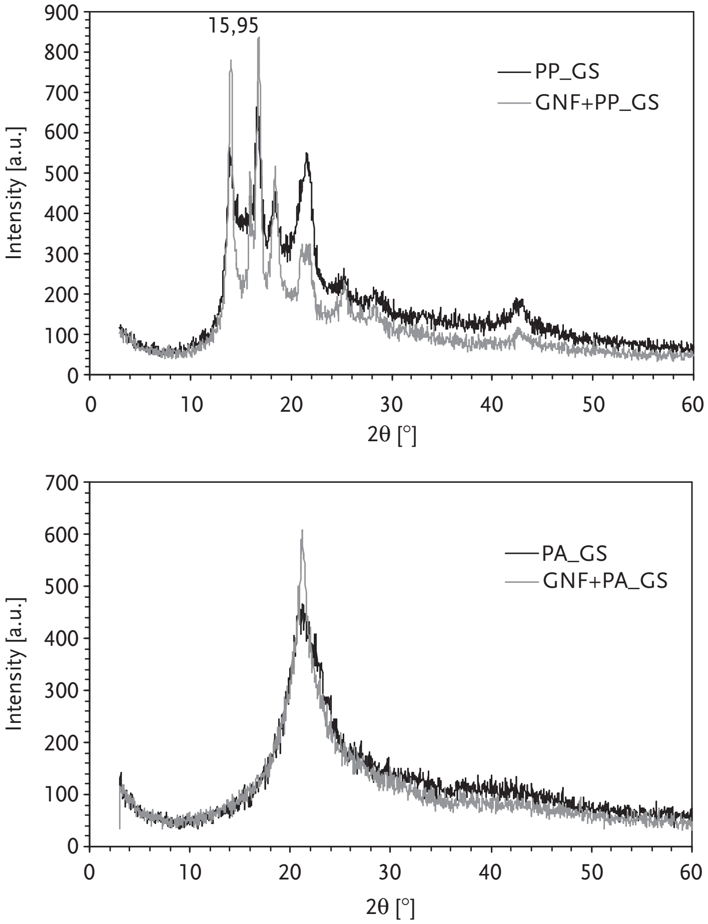
Table 2 Tensile properties

Material	Modulus [GPa]			Strength [MPa]			Elongation [%]		
	Static		Dynam.	Static		Dynam.	Static		Dynam.
	Long.	Trans.	Long.	Long.	Trans.	Long.	Long.	Trans.	Long.
PP/GF	6.6	4.3	8.11	74.1	49.9	102.4	2.8	3.2	2.4
PP/GF/SiO_2	5.9	3.9	7.51	62.4	42.0	90.1	2.9	3.4	2.5
PP/GF/MMT	5.9	3.9	7.66	61.7	41.5	83.7	2.4	2.7	1.8
PP/GF/GS	5.7	3.7	8.10	47.4	31.9	62.8	1.3	1.6	1.1
PA/GF	6.9	4.5	8.70	116.2	78.3	154.4	5.2	6	4.1
PA/GF/SiO_2	7.2	4.4	9.17	105.1	70.8	133.3	6.9	8.0	6.1
PA/GF/MMT	7.6	5.0	9.23	109.7	73.9	140.4	5.1	6.8	4.3
PA/GF/GS	8.6	5.6	9.62	109.9	74.0	142.9	4.8	5.5	3.9

By contrast, the effect of secondary filler in the PA composites was positive. All samples indicated an increase in tensile modulus. The biggest enhancement was observed in PA/GF/GS (25%) and PA/GF/MMT (10%), and the smallest one in PA/GF/SiO_2 (4%). The tensile strength of all composites remained at a similar level. Elongation to brake was found increased in materials reinforced with SiO_2 (33%) and decreased in PA/GF/MMT (-2%) and PA/GF/GS (-8%) composites.

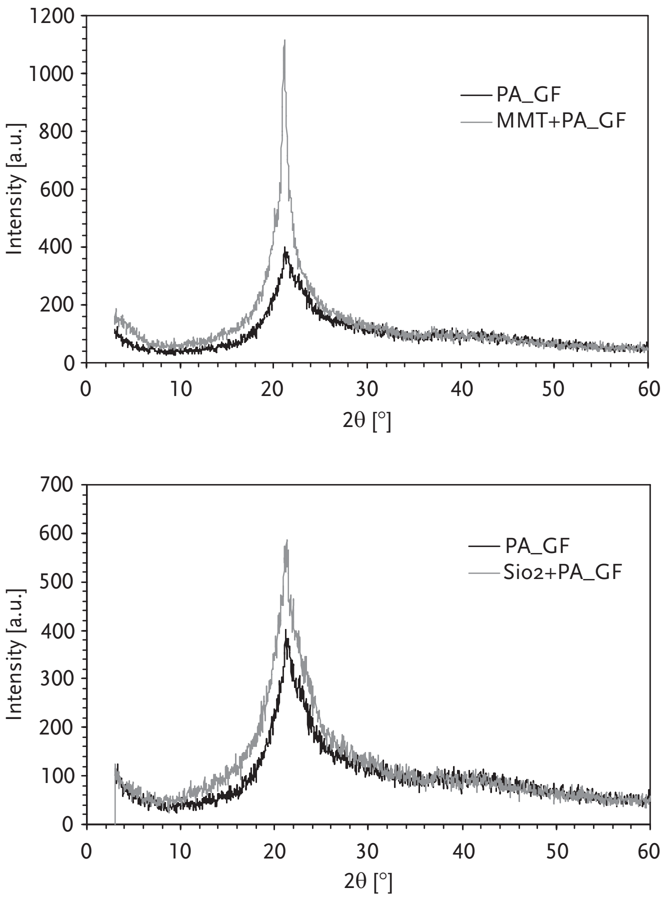
The difference in the mechanical properties of the composites filled with various materials could be due to their different reinforcing mechanisms. A rigid reinforcement often results in an increase in stiffness, at the cost of reduced ductility while retaining the strength of polymer composites. This phenomenon could be caused by transferring of the stress from the flexible matrix to the reinforced rigid composites, leading to an increase in stiffness. At the same time, the ability of the material to deform is reduced, due to the presence of stress concentrations in the nanofiller reinforced matrix. When the cracks initiated in the material reach the stress concentration regions they grow quickly, causing failure of the material at much lower strain in relation to the neat polymer. Such a situation was observed in the MMT and GS reinforced composites. An opposite behaviour was observed in the SiO_2 reinforced composite, which showed a small increase in modulus and strength, and large increase in elongation. This behaviour could result from the nature of the nano-particles, which could significantly reduce the formation of stress concentration regions. Furthermore, nanoparticles may act as crack blockers, leading to an increase in elongation to brake.

Figure 5 WAXD diffractograms of dust produced from (a) PP and PP/GS (b) PA and PA/GS panels reinforced with glass spheres



An interesting phenomenon observed in this study was that, at macro-scale, the SiO_2 particles changed the mechanical properties of the matrix in all directions of the material. This was confirmed by the comparison of the results from the tensile tests conducted in both in-flow and cross-flow directions. It was clearly visible that all mechanical properties were changed in both directions by almost the same fraction of the initial value. This leads to a conclusion that, at macro-scale, nano particles reinforced matrix can be considered as a quasi-isotropic material. Furthermore, for the macro-modelling purposes, the nano-silica reinforced three-phase composite can be assumed as a two phase composite (polymer and glass-fibres) with enhanced mechanical properties of the matrix. A similar behaviour was observed in glass-spheres filled polymers, in which the change in the mechanical properties was similar in both directions. That is why this material can also be considered as quasi-isotropic. However, due to the large size of the reinforcement, the composite should not be considered as a two phase material even at macro-scale. This is because the secondary

Figure 6 WAXD diffractograms of dust produced from (a) PA and PA/MMT (b) PA and PA/SiO_2 panels reinforced with glass-fibre



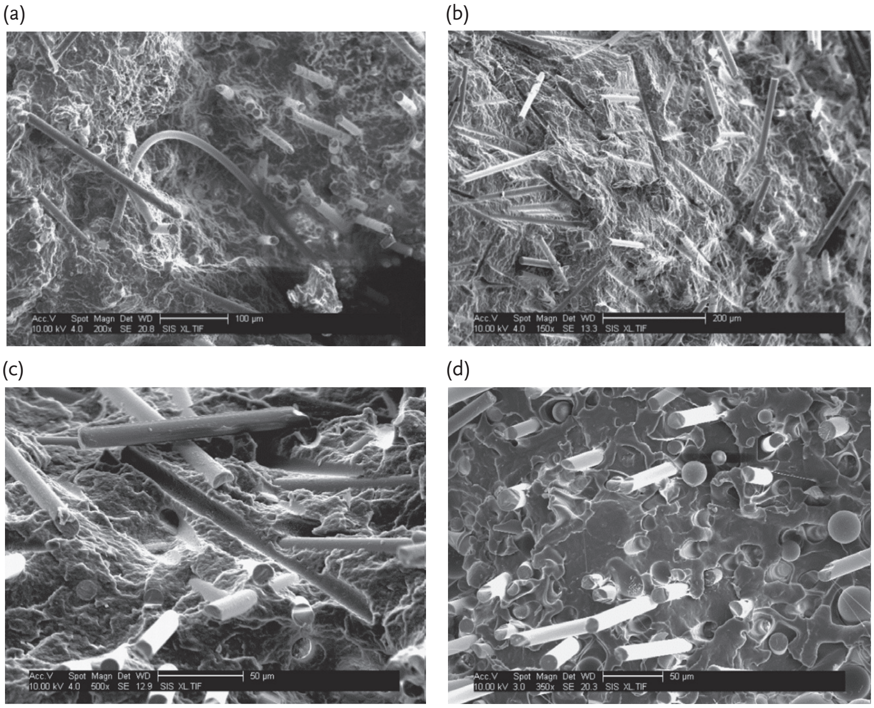
filler has changed the fracture mode of the composite, but it has not changed the mechanical properties of the matrix, as observed in silica filled composite. This effect was visible on the micrographs of the fractures surface obtained using SEM technique (see Figure 8). Regarding the material filled with MMT, the change in the mechanical properties was different in each direction. This is due to the plate-like shape of the MMT particles, which tend to align in the inflow direction, similarly to the glass-fibres. In this case the enhancement in material properties was different in both material directions.

Fracture analysis

The fracture surface of the tensile samples tested under the dynamic load was examined using SEM. Figure 7 shows the micrographs of several PP-based composites. From these pictures it was possible to observe that the failure mode was a combination of matrix and fibre cracking, fibre pull-out and debonding.

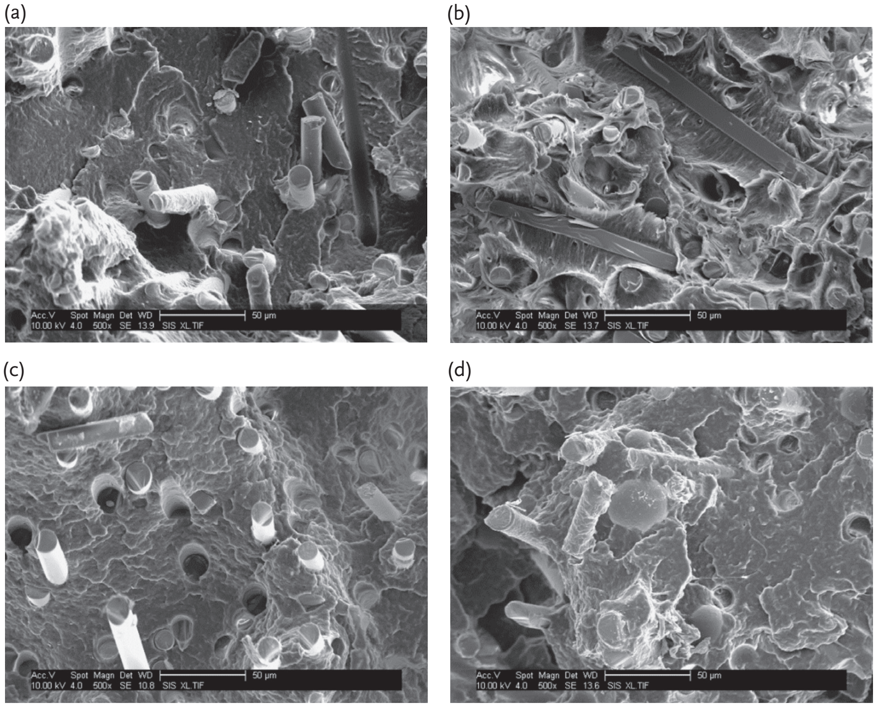
Very little plastic deformation of the matrix is visible, as there are no characteristic deformation paths and fibrous texture of the material. On the other hand, a significant debonding and pull-out of the glass reinforcement are visible. The clean and smooth surface of the glass indicates poor interfacial adhesion. Little difference can be observed between the micrographs of nano-reinforced PP/GF and neat PP/GF composites. This indicated that the secondary reinforcement had little influence on the PP composites failure mode.

Figure 7 PP composites: (a) neat, (b) SiO_2, (c) MMT and (d) GS



The fracture surface of PA-based composites is presented in Figure 8. There is an evident difference in the failure mode in relation to PP-based composites. The fracture is dominated by matrix and fibre cracking, whereas fibres pull out and debonding is of little meaning. The glass reinforcement is covered with polymer residuals, which is a sign of good interfacial adhesion. Moreover, there is a visible difference in the fracture mode between various PA composites. In neat PA/GF and PA/GF/SiO_2 composites the plastic deformation of the matrix is the most evident. An extensive plastic deformation is clearly visible in PA/GF/SiO_2 as a non-smooth texture and characteristic deformation paths, indicating a significant increase in material ductility. Contrarily, in PA/GF/MMT and PA/GF/GS composites, the plastic deformation of the matrix is reduced, due to the transition to more brittle failure as observed in previous studies [23,24].

Figure 8 PA composites: (a) neat, (b) SiO_2, (c) MMT and (d) GS



Conclusion

The morphology and the mechanical properties of various polymer composites were studied in this paper. It was shown that the addition of secondary filler into glass-fibre reinforced polymer composites can have significant influence on the mechanical behaviour of the materials. The following general remarks can be made regarding the morphology and the mechanical properties of the polymer composites: 1) secondary reinforcement in PA composites leads to an increase in mechanical properties, such as stiffness and elongation to brake; 2) all mechanical properties of PP composites have been decreased after the addition of secondary reinforcement, possibly due to a weak filler-matrix interphase region; 3) composites reinforced with particulate material can be treated as quasi-isotropic on a macro-scale, whereas this assumption is invalid for plate-like shaped materials; 4) the SiO_2 particles have changed the mechanical properties of the matrix, causing a significant increase in the material ductility.

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