Energy absorption characteristics of nano-composite conical structures

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Abstract. The effect of the filler material on the energy absorption capabilities of polyamide 6 composite structures is studied in details in the present paper. The axial dynamic and quasi-static collapse of conical structures was conducted using a high energy drop tower, as well as Instron 5500R electro-mechanical testing machine. The impact event was recorded using a high-speed camera and the fracture surface was investigated using scanning electron microscopy (SEM). The obtained results indicate an important influence of filler material on the energy absorption capabilities of the polymer composites. A significant increase in specific energy absorption (SEA) is observed in polyamide 6 (PA6) reinforced with nano-silica particles (SiO$_2$) and glass-spheres (GS), whereas addition of montmorillonite (MMT) did not change the SEA parameter.

1. Introduction

Thermoplastic polymers, such as polyamide 6 (PA6), are widely used in the automotive industry because of their good mechanical performances, processing properties and low cost. However, their application as structural materials is limited due to their low impact resistance and energy absorption capabilities [1]. Incorporation of glass-fibres and various nano-sized fillers such as: nano-particles, carbon nano-tubes, and clay nano-plates; can be an appropriate solution to that problem [2; 3]. In case of light-weight structures, made of polymer composites, the most widely used nano-reinforcements are silica based particles, due to their good mechanical properties and high thermal stability [4].

For the purpose of measuring the energy absorption in composite structures, tube crashing experiments are the most prevailing. The ability of a composite structure to absorb energy was found to be highly dependent on the mode of fracture. Materials which fail in a progressive manner, with extensive delamination and fragmentation, are able to absorb much higher energies than those which tend to fail in a brittle manner. Mamalis et al. [5] studied polyester cones, cylinders and tubes, reinforced with random orientated glass fibres, divided failure of the samples into four different modes: progressive crashing with micro-fragmentation (Mode I), brittle fracture with catastrophic failure (Mode II and III, depending on the crack form), progressive folding and hinging, similar to metallic tubes (Mode IV). Each of these modes is characterized by different energy absorption capabilities.

Numerous researches have been conducted to study the influence of nano-particles on the mechanical behaviour of polymer composites and main factors influencing their enhancing capabilities were outlined. This includes key parameters such as: shape [6] and size [7] of the nano-fillers, matrix and reinforcement material [8; 9], interfacial strength and interphase characteristics [10], as well as
volume fraction [11] and quality of dispersion within the matrix [12]. However, there is a lack of crash experiments conducted on nano-composites presented in the literature [2]. Energy absorption capabilities of nano-composites have been mainly characterized by means of compression [13], flexural [14] and Charpy or Izod impact testing [15]. That is why the relation between mechanical properties of nano-filled materials and energy absorption characteristics of nanocomposite structure is not fully understood.

2. Experimental

2.1. Materials
Polyamide 6 (PA6) (Tarnamid T-30 from Azoty Tarnow Poland), was used as a matrix material. As nano-filler two different types of material were used: silica-particles (SiO$_2$) (AEROSIL 200 from Degussa) and montmorillonite (MMT) (Dellite 43B from Laviosa). Additionally, two different glass-reinforced composite materials, supplied by MACOMASS Verkaufs AG Germany, were used to prepare nano and glass-reinforced composite samples: glass-fibre (GF) reinforced polyamide 6 (MM-PA I 1F30) and glass-spheres (GS) reinforced polyamide 6 (MM-PA I 1K30).

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 granulate and injection moulding of the structural cones. In the first step nano-reinforcement and polymeric matrix, all in solid (powder) form, were premixed before extrusion, in order to warrant the highest homogeneity of the composition. Subsequently, the premixed materials were fed into the twin-screw extruder. In the second step, nano-composite granulates and glass-fibre reinforced polymers were mixed in the extruder. As a result eight different composite materials were prepared as shown in Table 1. In the third step, crash cones were produced using injection moulding machine (Engel ES200/60 HL ST).

| Table 1. PA6 composites. |
|---------------------------|
| Name | PA/GF | PA/GF/GS | PA/GF/SiO$_2$ | PA/GF/MMT |
| Matrix | PA | PA | PA | PA |
| 1$^\text{st}$ filler [wt%] | GF [30%] | GF [30%] | GF [30%] | GF [30%] |
| 2$^\text{nd}$ filler [wt%] | - | GS [12%] | SiO$_2$ [2%] | MMT [2%] |

2.2. Methods
Quasi-static compression testing of the crash cones was conducted using Instron 5500R universal machine, at a crosshead speed of 0.1mm/s. The load was measured using a 100kN load cell. Impact tests of the crash cones were carried out on a high energy capacity drop tower machine at the velocity of 6.2m/s. The impactor mass of 54kg was constant in all experiments, giving an overall impact energy of 1050J. The load was measured using a 200kN load cell, placed underneath the sample. In order to measure the displacement of the falling mass, the linear variable differential transformer (LVDT) displacement transducer was used, with precision of 0.01mm and a maximum displacement speed of 10m/s.

The fracture surface of the impacted cones 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.

3. Results and discussion

3.1. Crashing behaviour
Crashing behaviour and energy absorption characteristic of the composite structures were studied by means of quasi-static compression and dynamic impact testing. The results obtained are listed in
Tables 2 and 3. Analysing these results it can be seen that crushing characteristic under dynamic load are different from those subjected to the quasi-static compressive load. All materials tested under dynamic load absorbed similar amount of impact energy. However, a big difference can be seen in the specific energy absorption (SEA) parameter. This discrepancy was caused by the fact that each material failed with a different crushing length. For PA/GF/GS composite the crushing length was much smaller than for the other PA6 composites. As the specific energy absorption is a function of a crushed mass the SEA parameter was the highest in these materials, which absorbed the energy at a small crushing length.

Regarding the loads induced during the impact, the mean crushing load was much closer to the initial peak in case of PA/GF/SiO₂ composite, which had a direct influence on the amount of energy absorbed by the structure. In case of the other PA6 composites, the mean crushing load was significantly smaller than the initial peak indicating weaker energy absorption capabilities (see Figure 1).

![Figure 1. Load-displacement curves (a) static (b) dynamic.](image)

**Table 2.** Quasi-static crashing characteristics.

| Material    | Crash length [mm] | Collapse mode | Initial peak [kN] | Mean crashing load [kN] | Energy absorbed [kJ] | SEA [kJ/kg] | Change in SEA [%] |
|-------------|-------------------|---------------|-------------------|-------------------------|----------------------|-------------|-------------------|
| PA/GF       | 86                | II            | 47.66             | 50.44                   | 4.33                 | 58.1        |                   |
| PA/GF/SiO₂  | 86                | II            | 44.61             | 45.66                   | 4.15                 | 54.5        | -6.1              |
| PA/GF/MMT   | 86                | II            | 54.59             | 40.65                   | 3.23                 | 42.9        | -26.2             |
| PA/GF/GS    | 86                | II            | 55.10             | 45.74                   | 4.11                 | 51.7        | -11.0             |

**Table 3.** Dynamic crashing characteristics.

| Material    | Crash length [mm] | Collapse mode | Initial peak [kN] | Mean crashing load [kN] | Energy absorbed [kJ] | SEA [kJ/kg] | Change in SEA [%] |
|-------------|-------------------|---------------|-------------------|-------------------------|----------------------|-------------|-------------------|
| PA/GF       | 60.5              | I             | 19.99             | 5.64                    | 0.35                 | 7.7         | -                 |
| PA/GF/SiO₂  | 57.56             | II            | 26.51             | 8.98                    | 0.43                 | 9.8         | 27.0              |
| PA/GF/MMT   | 62.61             | I             | 38.82             | 4.48                    | 0.37                 | 7.7         | 0.1               |
| PA/GF/GS    | 22.03             | II            | 40.42             | 15.58                   | 0.32                 | 22.3        | 188.5             |
The loads induced during the impact are directly correlated with the fracture mode and propagation of the cracks. The following main fracture modes could be identified and classified: (i) Mode I - Brittle fracture with large fragmentation. This fracture mode corresponds to unstable and catastrophic failure of the sample. Its characteristic part is formation of large debris due to the propagation of axial cracks. These cracks become initiated at the early stage of the impact event and cause a significant decrease in post-failure strength and stability of the structure. This mode indicates weak energy absorption and was observed in PA/GF and PA/GF/MMT composites tested under dynamic load (see Figure 2a and 2c). (ii) Mode II - Brittle fracture with progressive crashing and medium fragmentation. In this mode propagation of the axial cracks, initiated at the early stage of the impact event, stops quickly after the formation. Therefore, the size of the generated debris is significantly smaller than the debris size observed in Mode I. Additionally, a delamination effect was observed, as a separation of the composite layers. That is why the structure does not suffer catastrophic failure, indicating relatively good energy absorption, compared to Mode I. This mode was observed in PA/GF/SiO$_2$ and PA/GF/GS composites tested under the dynamic load (see Figure 2b and 2d), as well as in all PA based materials tested under the quasi-static load (see Figure 3).

Relating the energy absorption characteristic with the crashing characteristics, it can be seen that the materials which fail in a progressive manner, with small local cracks induced (Mode II), are able to absorb much higher energies than those with large continuous cracks (Mode I). This is caused by the fact that the fracture mode has got direct influence on the crushing parameters such as: crushing length, value of the peak loads and mean crashing load. The crushing length of the structure increases if the large cracks and debris become initiated. Additionally, the post-failure strength of the material is also reduced, what was recorded as a decrease in mean crushing load. As a result, the specific energy absorption of the material, which depends on these two parameters, was decreased as well.

![Figure 2: Dynamic collapse mode of PA6 composites (a) PA/GF (b) PA/GF/SiO$_2$ (c) PA/GF/MMT (d) PA/GF/GS](image)

![Figure 3: Static collapse mode of PA6 composites (a) PA/GF (b) PA/GF/SiO$_2$ (c) PA/GF/MMT (d) PA/GF/GS.](image)
Figure 4: PA composites: (a) neat, (b) SiO$_2$, (C) MMT and (d) GS.

Analysing the influence of the secondary filler on the energy absorption of polymer composites, a significant differences could be observed. The SEA parameter increased in SiO$_2$ and GS reinforced composites, whereas it decreased in MMT filled ones. In PA/GF composite the axial cracks were initiated at a relatively low load, indicating low impact resistance of the material. The cracks propagated quickly along the height of the cone, leading to catastrophic failure of the structure (Mode I) and low energy absorption. Incorporation of SiO$_2$ particles did not increase the impact strength of the material but it changed the fracture behaviour. This was observed as a transition from fracture Mode I to Mode II. This change was caused by the significant reduction of the material brittleness, which was observed as an increase in elongation to break. As a result, the strain induced in the structure did not initiate severe cracks, as the material below the crush zone did not reach the failure strain. Moreover, an extensive delamination was observed, increasing the energy absorption capability of the material.

The opposite behaviour was observed in PA/GF/MMT composite. In this case the impact strength of the material was increased, but at the cost of reduced ductility. That is why the nano-composite became even more brittle than neat PA/GF composite. Hence, the strain in radial direction reached the maximum allowable limit and the axial cracks propagated along the height of the structure, leading to a complete failure of the structure. As a result the energy absorption capability of the material
remained on the same level as for the neat PA/GF, in spite of the increase in properties such as: impact strength, tensile strength and stiffness.

The biggest increase in the SEA parameter was found in the PA/GF/GS composite. Similar to the SiO$_2$ reinforced PA/GF, the fracture mode has changed from Mode I to Mode II, after the addition of the secondary reinforcement, but the toughening mechanism was different. In this instance, the properties such as: stiffness, impact and tensile strength were improved, but with reduced elongation to brake, analogously to PA/GF/MMT composite. However, the increase in stiffness was much more significant, and additionally, the material was subjected to delamination effect. As a result the radial stress did not initiate any axial cracks, due to the high resistance of the material and propagation of the interlaminar cracks. That is why the crashing length of the cone was importantly reduced increasing the value of the SEA parameter.

3.2. SEM Analysis
The fracture surface of the crash cones tested under dynamic load was examined using SEM. In all PA6 composites the fracture was dominated by matrix and fibre cracking. The glass reinforcement was covered with polymer residuals, which was a sign of good interfacial adhesion. Moreover, there was a visible difference in the fracture mode between various PA6 composites. In neat PA/GF and PA/GF/SiO$_2$ composites the plastic deformation of the matrix was the most evident and the fibres pull out and debonding was of little meaning. An extensive plastic deformation was clearly visible in PA/GF/SiO$_2$ as a non-smooth texture and characteristic deformation paths. Contrary, in PA/GF/MMT and PA/GF/GS composites, the plastic deformation of the matrix was reduced, due to the transition to more brittle failure. There were also visible signs of fibre pull out and debonding.

4. Conclusion
It has been shown that addition of secondary reinforcement into glass-fibre reinforced polymer composites can have a significant influence on the energy absorption capabilities of the material. The carried out experiments showed that by changing the secondary filler material it is possible to change the micro-mechanism of the crash and therefore control the energy absorption characteristics of the composite. The following general remarks could be drawn, regarding the energy absorption of polymer composites: (i) Secondary reinforcement in PA6 composites leads to an increase in energy absorption capabilities of the structure. (ii) The transition from brittle to ductile fracture mode was clearly demonstrated as a main reason for the increased energy absorption capabilities. (iii) Two different toughening mechanisms were observed. First, due to the increase in elongation to brake. Second, due to the increase in the material impact strength and stiffness.

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