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Mechanical and impact performance of three-phase polyamide 6 nanocomposites

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ABSTRACT

In this work, three-phase nanocomposites using multiscale reinforcements were studied to evaluate the influence of nanofillers on static and dynamic mechanical properties at varying temperature conditions. In particular, short-fibres reinforced polyamide 6 (30 wt.%) composites with various weight fractions of montmorillonite (OMMT) and nanosilica (SiO_2), manufactured and investigated. Quasi-static tensile properties were investigated at room temperature and also at 65 °C just above the polyamide 6 (PA6) glass transition temperature. The low velocity impact tests were conducted on the manufactured cone-shaped structures to evaluate the crash behaviour and energy absorption capability. The study results shows that the increase of the weight percentage level of OMMT in PA6/glass fibre (30 wt.%) composite made the nanocomposites more brittle and simultaneously deteriorated the tensile properties. SiO_2 nanofiller at 1 wt.% was found to be the optimum ratio for improving tensile properties in silica-based nanocomposites studied. It was further noted that for both types of nanofillers, the crashing behaviour and energy absorption in dynamic properties were improved with increase in nanofillers weight percentage in the composites. The study also shows that the brittleness behaviour of the nanocomposites investigated is associated to the fibre/matrix interaction which is dependent on the nanofiller type and has significant effect on crash modes observed.

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1. Introduction

A significant number polymer and polymer composites are increasingly being used in various industrial applications such as, aerospace, automotive and chemical industries. This is because these materials provide high strength/weight ratio in comparison to classic material. In addition, most classic polymer materials have limited structural applications due to their low mechanical, thermal and impact resistance properties. Therefore reinforcements are often used to improve their properties [1,2].

Today, various types of polyamides covering a wide range of properties are commercially available. Polyamide 6 (PA6) is one of the polyamide grades most widely used in the automotive industry. PA6 is a high performance engineering thermoplastic used in electrical/electronics, automobile, packaging, textiles and consumer applications. However, limitations in mechanical properties, the low heat deflection temperature, high water absorption and dimensional instability of pure PA6 have prevented its wide range applications in load bearing applications such as under-the-hood automotive applications [3]. Hence, polyamide 6 reinforcement using nanofillers to further widen and increase its application range in impact/crash applications is beneficial [4].

In brief, nanocomposite materials are an attractive technology because using nano-fillers allows great improvements of the polymeric materials compared to micro-reinforcement and suit to the industrial/technological goals [5]: e.g. to produce lighter, thinner, stronger and cheaper structures [6]. The nano-size of the fillers increases the area of contact between matrix and filler and so, reduces stress concentration around the filler. Also, the nano-size presents a very large surface area to volume ratio. For example, it augments the surface area to volume ratio up to 10^3 times for a nanofibre compared to a microfibre [6]. It is also significant to note that only 5 wt.% of nanofillers can significantly improve behaviour and properties of a neat polymer [7], compared to at least 20 wt.% with glass fibre reinforcement, which allows a reduction of weight and cost. For the nano-reinforced composites, however, it is essential to control and stabilize a desired type of morphology in polymers in order to generate polymeric materials with favourable properties.

Some of the key properties that are improved include strength, stiffness, heat-distortion temperature, scratch resistance, thermal,

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oxidative and dimensional stability, water and thermal permeability, corrosion resistance, surface hardness, barrier properties, flame retardancy and electrical conductivity [8,9]. Some studies in the literature have focused their research on the influence of modified or unmodified clay on polymer nanocomposites' properties [10–14]. For example, enhancement of mechanical properties of nanocomposites and three-phase nanocomposites are often confirmed. Mishra et al. [13] had shown that Young modulus, tensile strength and elongation at break are increasing with the augmentation of organically modified montmorillonite (OMMT) content (until 3 wt.%) into a polyamide-66 matrix. Silva et al. [15], reported that an increase of 32% for the elongation at break for a 30 wt.% glass fibre/polyamide-6 filled with 2 wt.% of SiO_2 nanoparticles, compared to a classical polyamide-6/glass fibre. Wu et al. [16] found that a polyamide-6/clay with 30 wt.% of glass fibre had an enhanced tensile strength of 11% and a tensile modulus enhancement of 42% compared to polyamide-6/30 wt.% glass fibre. Further, several parameters were demonstrated to have an influence on these mechanical properties (stiffness, modulus) such as interaction between the matrix and the fillers [17], fillers' size [18], fillers' volume fraction [19], and filler's shape [9]. Others advantages are the cost which is low considering that only a small amount of filler is necessary, and the ease of manufacture without need to change the conventional processing conditions in order to manufacture new products [6].

As the use of thermoplastics in the automotive industry increases, the need to determine their impact responses to ensure safety and stability of designed structures is important. As such, the impact test should be ideally designed to simulate the loading conditions to which the composite component is subjected to in operational service and then reproduce the failure modes and mechanisms likely to occur in real conditions. More efforts in understanding the impact performance and failure mechanisms of reinforced thermoplastics used in automotive industry are necessary. 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 therefore aims to investigate the morphology and the mechanical properties of 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 particular, the influence of the nano-fillers content and temperature changes in three-phase nanocomposites using multiscale reinforcements were studied. Polyamide-6 reinforced filled with short glass fibre 30 wt.% and with an addition of nanoclay (montmorillonite, OMMT) and/or nanosilica (SiO_2) were tested in order to characterise their tensile properties at room temperature and at 65 °C just above the polyamide 6 glass transition temperature. The quasi-static tensile properties are complemented with crashing behaviour ones and fracture studies were also conducted for completeness.

2. Experiment

2.1. Materials and samples preparation

Glass-fibre reinforced polyamide MM-PA I 1F30 i.e. polyamide-6 (Durethan B30 and B31) reinforced by 30% of glass fibre (ThermoFlow 672) were supplied by MACOMASS Verkaufs AG Germany. Montomorillonite, Dellite 43B, were obtained from Laviosa Chemicals. Fumed silica nanoparticles AEROSIL 974 were obtained from Degussa.

Two types of three-phase nanocomposites were produced: polyamide-6 (Durethan B30) reinforced by 30% of glass fibre (ThermoFlow 672) and particles of SiO_2 (Aerosil R 974), and polyamide-6 reinforced by 30 wt.% of glass fibre (GF) and montmorillonite (Dellite 43B, Laviosa Chemicals). 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. In total, seven materials were manufactured with different content of nano-fillers (Table 1).

The mechanical testing specimens were obtained by direct melting and extrusion in a twin-screw extruder at a maximum temperature of 280 °C. The product was cooled in a water bath, pelletized and then dried. From granulates, test samples (crash cones, tensile bars and plates) were manufactured by injection moulding according to the ISO 527 [20] test standard requirements.

2.2. Mechanical testing

Quasi-static and dynamic tensile tests were carried out using Instron 5500R universal testing machine. All experiments were conducted according to ISO-527 [20] standard, using specimen type A. Samples were machined from injection moulded plates in two different direction: longitudinal (along fibres) and transverse (cross-fibre). Five samples of each material were tested at crosshead speed of 0.1 mm/min. The load was measured using a 100 kN load cell. In order to measure the strain a laser extensometer was used.

Crashing behaviour was analysed with quasi-static and dynamic crash experiments. Quasi-static compression testing of the crash cones was carried out using Instron 5500R universal testing machine. A set of samples of each material were tested at a crosshead speed of 0.1 mm/s. The load was measured using 100 kN load cell and the displacement was measured using a built in crosshead displacement sensor. Impact tests of the crash cones were carried out on a high energy capacity drop tower. Three samples of each material were tested at the velocity of 4.4 m/s. The tests were performed by direct impact of the falling beam. In order to ensure a good distribution of the load, 8 mm thick steel plate was placed on the top surface of the cone. The impactor mass of 54 kg was constant in all tests, giving the overall impact energy of 522 J. The load was measured using 200 kN load cell, placed underneath the sample. In order to measure shortening of the sample (falling beam displacement), the linear variable differential transformer (LVDT) displacement transducer was used, with precision of 0.01 mm and maximum displacement speed of 10 m/s. The impact event was recorded using Phantom high speed camera.

2.3. Scanning electron microscopy

In order to study the materials failure mechanism and the relation between the matrix and the filler, the fracture surface of the tensile bars was examined with FEI XL30 field emission scanning electron microscope (FE-SEM). The operating voltage was in the range of 10–20 kV and the specimens were gold sputtered to minimize charging of the sample.

3. Results and discussion

3.1. Tensile properties

3.1.1. Effect of the filler's type

In this work, tensile properties of polyamide-6/glass fibre nanocomposites were investigated. Fig. 1 illustrates the tensile stress vs

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