



Layered silicates nanocomposite matrix for improved fiber reinforced composites properties

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ABSTRACT

Three-phase glass fiber reinforced composites (GFRP) consisting of traditional woven glass fiber and polyamide-6 (PA6) matrix dispersed with organically modified layered silicates were prepared and investigated in this study. The fabrication of GFRP with different weight percentages of layered silicates was successful when the matrix contains less than 5 wt% of the layered silicates. The improvement due to the high aspect ratio and high stiffness of the layered silicates is illustrated through the matrix-controlled properties of the GFRP. The results showed that the GFRP with 5 wt% layered silicates offer the largest improvement of approximately 30% increase in both flexural strength and compressive strength at elevated temperatures. On the other hand, the in-plane shear properties measured from $[\pm 45]_s$ laminates revealed that the layered silicates help improved both the in-plane shear strength and modulus appropriately. By utilizing a nanocomposite matrix, improvement of stiffness and strength, as well as thermal and barrier properties is obtained without any change in processing temperature of the fiber composites.

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1. Introduction

In recent years, layered silicates or nanoclays have attracted great interest as the outstanding reinforcement for various types of engineering polymers due to the possibility of exceptional mechanical properties enhancement as well as added multifunctionality at low filler content. The incorporation of nanoclays into the polymers is also expected to result in the improvement of the mechanical properties of three-phase continuous fiber reinforced composites. The key to such performance depends on the ability to exfoliate and disperse individual, high aspect ratio silicate platelets within the polymer matrix. Many processing methods such as in-situ polymerization, solution mixing and melt mixing are adopted to produce such polymer/layered silicates nanocomposites. The technique of melt processing is particularly attractive due to its versatility and compatibility with existing processing infrastructure. Many research studies show that melt processing in a well-designed twin-screw extruder [1–6] can produce well exfoliated nanocomposites for many types of thermoplastic

matrix. Although researches on two-phase polymer/clay nanocomposites have been extensively studied for many types of polymers and based on several dispersion techniques, successful processing and characterization of three-phase composites have been rarely reported.

In earlier works, these nanoclay-filled epoxy resins often did not lead to an improvement of the fiber composite strength [7–9], apparently due to problems with fiber impregnation or because the fibers were damaged in the process. However, recently several results have been presented on fiber reinforced epoxy nanocomposite where the expected improvement in matrix-dominated properties, among which the increase in flexural, compressive, shear and transverse tensile strength, was indeed achieved [10–16].

Most of the research on the concept of three-phase GFRP is primarily confined to only epoxy-layered silicate nanocomposites except a study by Akkapeddi [17] on short fiber reinforced PA6 nanocomposites and our previous published research on continuous fiber reinforced PA6 layered silicate nanocomposites by Vlasveld et al. [18,19]. The study on a nanomaterials reinforced matrix for fiber composites shows that the flexural and compressive strength is indeed achieved. However, the full potential of layered silicates for improving matrix-dominating properties of the fiber composites was not reached, apparently since the nanoclays were not fully exfoliated in the matrix phase in order to compromise with the ease of fiber impregnation.

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The main goal of this research is to improve the matrix-dominated mechanical properties of GFRP laminates by using organically modified layered silicates. As the conventional GFRP laminates have high enough tensile properties, exceptional strength improvement of the resin in tension is not necessary. However, nanocomposites based on PA6 can retain a relatively high modulus at temperatures above T_g [20] and in the presence of absorbed moisture [21], and have a strongly reduced creep compliance [22]. Nanocomposites can eliminate or reduce the disadvantages associated with PA6 as matrix material for fiber composites.

In this study, the melt mixing approach was used to disperse organically modified layered silicates in PA6 matrix. Due to high viscosity of the exfoliated nanocomposite resin, which can lead to filtering of the nanoparticles by the fiber bundles, porosity, incompletely wetting, etc., temperature and pressure cycle during processing of three-phase GFRP laminates was experimentally investigated. The matrix-controlled properties of the GFRP with a nanocomposite matrix at various temperatures and after moisture conditioning are presented. The effect of organoclay contents on the flexural, compressive and shear properties of the three-phase fiber composites is discussed. Microscopy is used to investigate the fracture behavior in this new type of fiber composites.

2. Experimental

2.1. Materials

As a reference material, polyamide-6 Akulon®K222D from DSM, the Netherlands, which is a medium-viscosity extrusion grade PA6 with nucleating agents, was used in this study.

The nanocomposite contains 2.5, 5 and 10wt% Somasif®MEE synthetic mica layered silicate nanoparticles from Coop Chemicals, Japan in PA6 Akulon®K222D. Somasif® MEE (or MEE) is organically modified synthetic fluorine mica based on the Somasif®ME-100, which is synthesized by heating talc with Na_2SiF_6 . The crystal structure is similar to montmorillonite. The organic surfactant molecules on this layered silicates are coco-bis(2-hydroxyethyl) methyl ammonium. Due to the organic modification the layered silicates are often referred as organoclays.

Ten Cate Advanced Composites SS0303, eight Harness satin weave E-glass fibers, 300 g/mm^2 , (Style 7781) ($E = 77 \text{ GPa}$) were provided for this research.

2.2. Preparation

The nanocomposite was made via melt mixing in a Werner and Pfleiderer ZSK 30/44D co-rotating twin-screw extruder operated at a temperature of 240°C , at a rotation speed of 200 rpm and a feeding rate of approximately 25 ml/min followed by palletizing or film extrusion. The nanocomposites with 2.5wt%, 5wt%, and 10wt% of the Somasif®MEE organoclays were produced for this study. Dumbbell shaped samples according to ISO 527 standards were produced by injection molding of the nanocomposite pallets on an Arburg Allrounder 221-55-250 machine. The feeding zone was heated to 150°C , the melting and mixing zones heated to 240°C and the nozzle was heated to 270°C .

The 12-ply GFRP laminate of $45 \text{ cm} \times 45 \text{ cm}$ were made by film stacking in a flat platen press at 270°C and 200 kN. The composite is build up by alternating layers of matrix material films and woven fiber reinforcement. The GFRP laminates have a fiber volume fraction of about 50% and average thickness of $3.2 \pm 0.14 \text{ mm}$. The composite samples were cut with a water-cooled diamond saw and further dried in a vacuum oven at 80°C for at least 48 h before testing. The moisture-conditioned samples were stored in a climate chamber at 62% RH and 70°C (ISO 1110). These conditions result in a moisture content of 3wt% in the PA6 phase [21,23].

2.3. Testing

2.3.1. Tensile tests on the pure matrix materials

The modulus of the injection molded matrix samples was measured in tension at a crosshead speed of 5 mm/min at 20°C intervals from 20– 160°C according to the ISO 527 standard on a Zwick 1445 universal test machine with a 10 kN load cell. The machine was equipped with a temperature chamber to test the samples at elevated temperatures.

2.3.2. Flexural tests on the 12-layer glass fiber composites

The 3 point bending tests were done according to the ASTM D790M test standard on a Zwick 1445 universal test machine with a 10 kN load cell. The machine was equipped with a temperature chamber to test the samples at elevated temperatures. The test speed was 5 mm/min. For each temperature and composition five samples were tested to determine the average values. The dry and moisture-conditioned composites with a PA6 and nanocomposite matrix were tested at 20, 80 and 120°C .

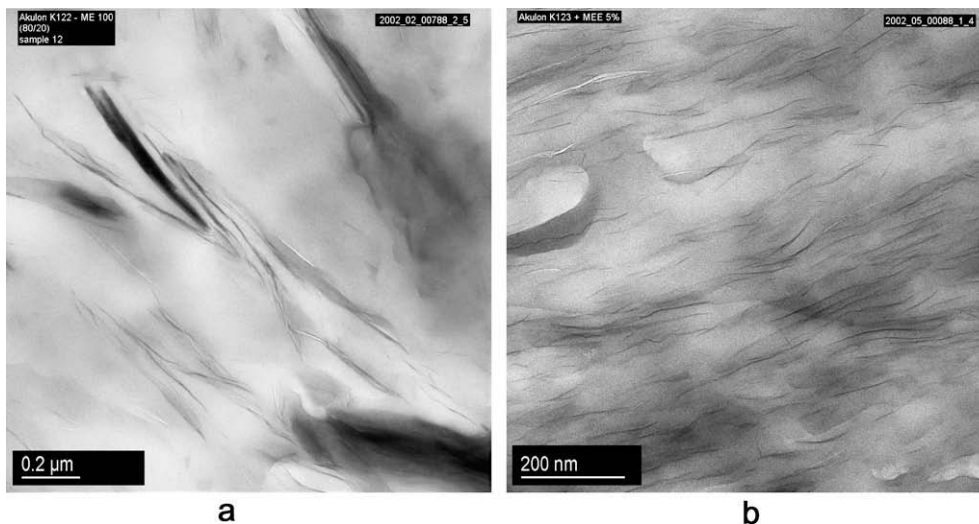


Fig. 1. TEM-images of PA6 nanocomposite with 5 wt% nanoparticles: (a) Somasif®ME-100 and (b) Somasif®MEE.

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