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EXPERIMENTAL INVESTIGATION ON THE CONSOLIDATION OF POLYPROPYLENE-CLAY NANOCOMPOSITES FIBERS

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Introduction

Polymers like polypropylene lack sufficient mechanical properties to be used in many engineering applications. To improve their mechanical properties, two main approaches have traditionally been employed [1]. The first and most common approach consists in adding foreign fillers or fibers such as talc or glass fiber. More recently, nanoparticles of clay and carbon nanotubes have also been used to enhance the mechanical properties of polypropylene [2,3]. A second approach that can be used to improve the mechanical properties consists in producing parts from oriented fibers of a polymer. The production of such all-polymer composites has been shown to be successful for several polymer materials including polypropylene [4,5].

In this study, the results of an experimental investigation on the consolidation of polypropylene (PP) and polypropylene nanocomposite (PPNC) fibers is presented. Two reinforcing effects were targeted by using polymer fibers reinforced with nanoparticles of clay. First, by carefully selecting the temperature, it should be possible to keep some of the orientation of the polymer chain in the composite. Secondly, the presence of nanoparticles is expected to increase the mechanical properties of the laminate.

Experimental

Material

To produce fibers, PP and PPNC were extruded at 220°C through a 37.75 mm (1.25 in.) extruder, followed by a gear pump and fiber die. The die contained 150 holes having a diameter of 380 µm and spaced by 0.5 mm. A constant mass flow rate of 4 kg/hr was used. A draw down ratio of about 22 was used to orient the fibers in air at the exit of the die before collection.

Unidirectional composites were produced by hot compaction of PP and PPNC fibers. During consolidation, fibers were heated to between 160 and 167°C, held at this temperature for a period varying from 5 to 15 minutes and then cooled to room temperature at a rate of 10°C/min. A constant pressure of 2.4 MPa (350 psi) was applied during the entire molding cycle.

Flexural tests were performed at 23°C using a three point bending set-up. These tests were performed using a span-to-depth ratio of 16 on 12.5 mm wide specimen. Differential scanning calorimetric (DSC) experiments were conducted using a Perkin Elmer scanning apparatus at a rate of 20°C/min. The degree of crystallinity of each specimen was then determined from the melting enthalpy, ΔH_m. Structure of PP and PPNC fibers was evaluated using X-ray diffraction. The PP and clay crystalline axes orientation factors [6] were determined from wide-angle X-ray diffraction pole figures of using a Bruker AXS X-rays goniometer equipped with a Hi-STAR two-dimensional area detector. The generator was set up at 40 kV and 40 mA and the copper K_α radiation (λ=1.542 Å) was selected using a graphite crystal monochromator.

Results

The influence of the molding parameters on the void distribution was investigated for PP and PPNC unidirectional plates. For a holding time of 5 minutes, large porosities were observed between fibers, indicating insufficient resin melting to fill the gaps between the fibers. For a holding time of 15 minutes, only small voids were observed between fibers, indicating a significant improvement of the consolidation quality. Similar results were also observed for the influence of the molding temperature, since void content was seen to decrease with increasing temperature.

The influence of the holding time and forming temperature on the flexural properties measured in the longitudinal (LD) and transverse (TD) directions was determined. Results were obtained for holding times of 10 and 15 minutes. A sharp increase in flexural modulus was measured between low and high forming temperature for PPNC. However, for PP, the flexural modulus remained nearly constant for the entire range of temperature under investigation. Further investigations are needed to explain this difference. A maximal flexural stress in the longitudinal direction (LD) was nearly two times superior to that measured in the transverse direction (TD). The lower strength obtained in the transverse direction can be attributed to the remaining voids observed between fibers; these voids were acting as defects to initiate failure. For holding time of 15 minutes, a nearly constant flexural modulus is measured throughout the entire range of molding temperature investigated. Increasing the holding time therefore allow increasing the temperature processing window without reduction of the flexural modulus. The maximum flexural stress measured in the TD was seen to increase from 40 to 50 MPa for holding time of 15 minutes compared to 10 minutes.

Results obtained in flexion are summarized in Figure 1. Compared to the PPNC "annealed" plates, a significant increase in flexural modulus is obtained for

PPNC unidirectional laminates. If the flexural modulus of the PPNC "annealed plates" is taken as a reference, the increase in modulus is of approximately 70%. When the flexural modulus of PP unidirectional laminates are compared to the one of the PPNC unidirectional laminates, a significant increase of 15% is measured. This shows that PPNC fibers can be molded into laminates to produce composites with higher performance. For flexural stress, the maximum in the longitudinal direction was 30% higher than values obtained for the randomly oriented plates. In the transverse direction, the maximum stress was 15% lower than for the randomly oriented plates. The reduction in mechanicals is thought to be related to consolidation of the materials, voids observed between the fibers acting as defects to initiate the failure.

DSC Results showed that the initial fraction of crystallinity was 44% for both types of fibers. Similar experiments were then performed on the PPNC and PP unidirectional laminates. Results of the experiments gave crystallinity of 55% for both materials. This is similar to what was previously observed by others [7] for PET.

Finally, orientation of PP crystalline reflections (110) and (040) in PP and PPNC fibers and laminates as well as that of clay (001) axis (normal to the clay platelets plane) in the PPNC fibers and laminates were determined from X-ray diffraction pole figures and results are shown in Figure 2. Results on the orientation of the clay 001 axis (normal to the clay platelets plane), shown in Figure 2a, indicate that it is oriented in the normal direction in the PPNC fibers and that the orientation is maintained after hot-compaction. Figure 2 also shows that orientation of PP crystalline axes of PP is maintained after hot-compaction for PP and PPNC. PP crystalline orientation is actually seen to increase slightly as a result of recrystallization during the hot-compaction process.

Conclusions

Composite laminates were produced from PP and PPNC fibers using hot-compaction process, flexural modulus in the longitudinal direction (LD) was seen to result in a 70% improvement in performance, as compared to composites produced from randomly oriented fibers. Flexural modulus of unidirectional polypropylene nanocomposite laminates were also seen to be two times those obtained from plates made from randomly oriented polypropylene fibers.

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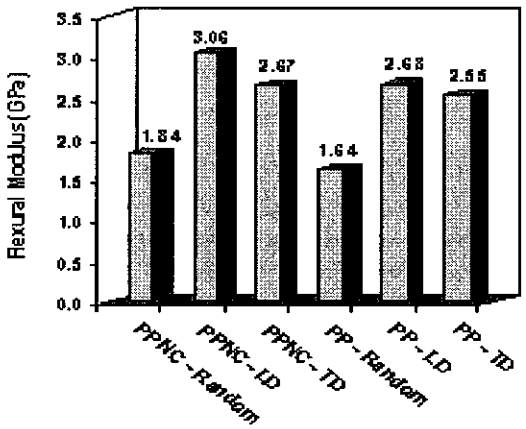

Figure 1: Flexural modulus obtained from unidirectional and randomly oriented plates.

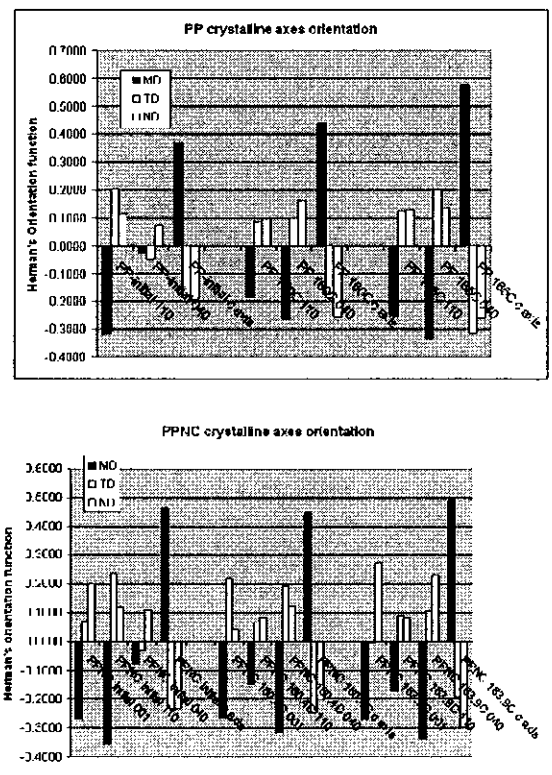

Figure 2: Herman orientation factor of PP reflections (110) and (040) in PP fibers and laminates and in PPNC as well as clay (001) reflection in PPNC.