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### Recycling carbon fiber composites using microwave irradiation: Reinforcement study of the recycled fiber in new composites

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**ABSTRACT**: With increasing use of carbon fiber reinforced polymer (CFRP) composites in transportation, sports, and many other industries, recycling of the scrap and end-of-life composites has presented both great challenges and opportunities. In this work, we report our study on reclaiming carbon fibers from CFRP using energy efficient microwave irradiation. Different irradiation conditions were used and the optimal conditions were determined based on the surface morphology of the recycled fiber. Polypropylene (PP) and Nylon, representing nonpolar and polar polymers, respectively, were reinforced using the recycled fiber through extrusion and injection molding. For comparison, PP and Nylon reinforced by virgin carbon fibers could improve these properties, they exhibited different reinforcing effects on the two polymers. The recycled fiber outperformed the virgin fiber in reinforcing PP whereas the virgin fiber performed better in Nylon. This was due to the differences in surface roughness, surface bonding, and fiber aspect ratio between the two fibers. This study shows the great potential of recycled carbon fiber and microwave irradiation as an effective recycling technique. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 42658.

**KEYWORDS:** composites; mechanical properties; recycling

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#### INTRODUCTION

Carbon fiber reinforced polymer (CFRP) composites exhibit desirable properties including light weight, high strength, high modulus, and high fatigue resistance. They have been widely used in aerospace industry, sporting goods, performance automobiles, civil engineering, etc. Rapidly increasing end-of-life CFRP products and large amount of CFRP scrap created during manufacturing have increased the urgency for recycling such materials.<sup>1,2</sup> Landfilling and incineration are the traditional methods for disposing of CFRP composites. The polymer matrices (most likely thermoset polymers such as epoxy) of the composites placed in a landfill take hundreds of years to decompose and can release harmful chemicals into the environment as they undergoe this process. Current EU Directive on Landfill of Waste (99/31/EC) classifies CFRP as chemical waste, which has led to their high disposal costs.<sup>3</sup> Incinerating CFRP can result in greenhouse gases and other hazardous chemicals being released into the air. These methods may be subject to stricter regulations in the future due to their negative environmental impacts.

In addition to environmental concerns of landfilling and incineration, producing virgin carbon fiber is energy intensive and therefore reclaiming it from the waste presents an economic advantage. Virgin carbon fiber is produced with an estimated energy intensity of 286 MJ/kg while only 33 MJ/kg is required for steel production.<sup>4</sup> Reclaiming carbon fiber from end-of-life and scrap materials can lead to substantial savings on material and energy costs. Virgin carbon fiber, due to its high cost, is limited in the application areas where the high costs can be justified by a substantial gain in mechanical performance and weight saving (e.g., aerospace and sport applications). Recycled carbon fiber, with its much lower price, could open up new markets and new opportunities in different industries. A series of applications, taking advantage of carbon fiber's high modulus, high electrical conductivity, dimensional stability, and temperature stability, have been developed, including electromagnetic interference shielding,<sup>5,6</sup> thermally conductive fabrics,<sup>7</sup> reinforcement for ceramic brake discs,8 fuel cell applications,9 and many composite applications.<sup>10–12</sup>

Carbon fiber has been reclaimed from waste composite materials mainly using thermal and chemical methods. In thermal recycling, composite materials are treated in hot air (450–550°C) in a fluidized bed process<sup>13,14</sup> or are heated in the absence of oxygen (i.e., pyrolysis).<sup>15–17</sup> A big advantage of

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fluidized bed process is its capability of processing mixed and contaminated materials while the advantage of pyrolysis is that both fiber and liquid/solid condensable chemicals can be recovered from the composites. In chemical recycling, polymer matrices are decomposed into useful chemicals in solvents and carbon fiber is reclaimed with largely unchanged properties. The solvents that have been used to decompose epoxy matrix include tetralin,<sup>18</sup> supercritical/subcritical alcohols,<sup>19–22</sup> nitric acid,<sup>23,24</sup> and supercritical/subcritical water.<sup>25–27</sup>

Electrical or fuel heating is traditionally employed as the energy source in thermal recycling processes. In this study, highly efficient microwave irradiation was used as the heating source. Composite scrap of carbon fiber reinforced epoxy was used as a model CFRP system to study the performance of microwave pyrolysis. The recycled carbon fiber was blended with polypropylene (PP) and Nylon 6 and the properties of the composites were compared with those of the composites reinforced with virgin carbon fiber.

#### EXPERIMENTAL

#### Materials

The CFRP composites used in this study were scrap of carbon fiber-epoxy composites prepared by vacuum assisted resin transfer molding using 8603 epoxy resin (Huntsman) and GA130 carbon fiber (Hexcel). The virgin carbon fiber was Panex 35 (Zoltek), which was produced to be compatible with engineering thermoplastics such as Nylon and polycarbonate. The carbon fibers from Hexcel and Zoltek show comparable tensile strength and Young's modulus (4137 MPa and 242 GPa, respectively) according to manufactures' data. PP (model 3825) and Nylon 6 (Ultramid 8202) were purchased from Total and BASF, respectively. All the materials were used directly without further treatments.

#### **Pyrolysis Temperature**

The CFRP scrap was cut into  $\sim 9 \text{ mm}^3$  cubes using a tile wet saw before pyrolysis. Approximately 250 g of the cubes were loosely placed in a crucible and the filled crucible was placed into a microwave furnace (CEM MAS 7000 Microwave Ashing Oven). The temperature of the furnace was increased to a preset temperature (400, 500, or 600°C) and held constant for 30 min. The specimens were allowed to cool down to 150°C before they were removed from the furnace. During the whole process, 25 SCFM of nitrogen flow, which was predetermined to be sufficient to protect the specimens from oxidation, was supplied to the furnace continuously. The surface of the recycled fiber was examined using SEM (JEOL JSM-6490LV) to investigate the effects of the pyrolysis temperature on fiber surface morphology. An optimal temperature was determined based on the results.

#### **Composite Fabrication**

The recycled carbon fiber was compounded with PP or Nylon using a Leistritz Micro 18 twin screw corotating extruder without any further treatment. All the materials were vacuum dried at 80°C for 24 h before compounding. The composites containing 0, 10, 20, and 30 wt % of the carbon fiber were prepared. The extrusion temperatures were set at 210 and 290°C for PP and Nylon, respectively. The extrusion speed was 100 rpm for all the formulations. The composites were extruded out through a filament die head, cooled in a water bath, and pelletized. For comparison, the PP and Nylon composites containing the virgin carbon fiber were also produced under the same conditions.

The pellets of all the composites were dried in a vacuum oven and then injection molded into ASTM standard tensile, flexural, and impact specimens using a plunge injection molder (Mini-Jector Model #45, Miniature Plastic Molding, Solon, OH). The molding temperatures were 290 and 235°C for Nylon and PP, respectively.

#### Characterization

Three point bending tests were performed following ASTM D 790-03 to evaluate the flexural properties of the specimens. The procedure "b" method was used to determine the strain rates used for each test. Tensile testing was performed following ASTM D638-03. Izod impact tests were conducted following ASTM D256-10 (notched samples). For all three tests, five replicates were tested for each sample. The fracture surfaces of the specimens after tensile and impact tests were analyzed using SEM (JEOL JSM-6490LV) to determine the failure mechanisms. The extrusion and injection molding processes can change the length of the carbon fibers. To determine their actual length and diameter in the composites, composite samples were heated in a muffle furnace at 400°C for one hour to remove the PP or Nylon matrices. The residual carbon fibers were collected and imaged using SEM. Fiber length and diameter were obtained from the SEM pictures based on 100 measurements using ImageJ software (National Institutes of Health).

#### **RESULTS AND DISCUSSION**

#### **Optimization of Pyrolysis Temperature**

Figure 1 shows the SEM micrographs of the recycled fiber after pyrolyzing the CFRP composites at 400, 500, and 600°C. At the lowest temperature, a large amount of pyrolytic residual is visible on the fiber surface after the thermal treatment. At 500°C, the fiber surface is much cleaner but some residual is still evident. Even at the highest temperature, residual can still be spotted at some locations [Figure 1(c)], while signs of fiber damage have appeared as shown in Figure 1(d), where cavities on the fiber can be noticed. The damage is most likely due to oxidation of the carbon fiber. As observed by Yang et al., the content of oxygen atoms on the fiber surface increases after pyrolysis even when O<sub>2</sub> level is very low during the reaction.<sup>10</sup> Based on these observations, 500°C was chosen as the pyrolysis temperature in this study as an acceptable compromise between residual removal and fiber damage. The carbon fiber reclaimed under this temperature was directly used in composite compounding without any further surface treatment, which lowers recycling cost but may lead to poor interfacial bonding between the fiber and the polymers, as will be discussed later.

## Determination of Carbon Fiber Length and Diameter in the Composites

The length of the carbon fibers are reduced after composite processing because of the mechanical forces exerted on the fibers during the extrusion and injection molding processes. To better understand their reinforcing effects in the composites,





Figure 1. SEM micrographs of the carbon fibers recycled from the CFRP composites using microwave irradiation. The pyrolyzing temperatures are (a)  $400^{\circ}$ C, (b)  $500^{\circ}$ C, and (c,d)  $600^{\circ}$ C.

their actual length, and diameter need to be determined. Figure 2 shows the SEM pictures of the carbon fibers in PP and Nylon carbon fiber composites after thermal removal of the polymer matrices. The fibers appear to be smooth and clean. Except being cutting shorter, no apparent further damages by the mechanical forces are noticed. The length and diameter of all the fibers were measured and the results are reported in Table I. The virgin fibers are longer than the reclaimed fibers in both PP and Nylon composites, although the distribution of the fiber length is wide. All the fibers exhibit similar diameters. The sizes of the carbon fibers are very important to the properties of the composites, as will be discussed later.

#### **Mechanical Properties**

Figure 3 compares the reinforcing effects of the recycled fiber and the Zoltek virgin fiber on the flexural strength of PP and Nylon. Both fibers increase the strength of the two polymers, with the recycled fiber showing stronger reinforcement on PP whereas the virgin fiber showing stronger reinforcement on Nylon. Especially, the recycled fiber almost doubles the strength of PP at 30 wt % fiber ratio while the virgin fiber only leads to a minimal increase. Both fibers also increase the moduli of the two polymers and the same preferential reinforcement on PP by the recycled fiber is observed (Figure 4). All the samples were also subjected to tensile testing to verify the preferential reinforcement of the two types of carbon fiber. Figures 5 and 6 compare the tensile strength and Young's modulus of PP and Nylon that contain different ratios of recycled and virgin carbon fibers. The recycled fiber continues to perform better in reinforcing PP than in reinforcing Nylon and the virgin fiber still performs better in reinforcing Nylon. It is worth noting that the tensile strength of PP is slightly decreased by the addition of the virgin fiber, an indication of poor fibermatrix interfacial bonding. Despite this strength decrease, Young's modulus of the composites increases monotonically with fiber concentration. This is because the modulus depends much more on the volume effect (i.e. rule of mixture - the increase in composite modulus is solely from the introduction of high modulus rigid fibers) than on interfacial bonding.

The effects of the two types of carbon fiber on the strength and modulus of the two polymers are more clearly presented in Figures 7 and 8, where the percentage changes in the properties are compared. Figure 7 clearly shows that for PP the recycled fiber substantially outperforms the virgin fiber in increasing all the four mechanical properties, whereas Figure 8 shows that for Nylon the virgin fiber performs only slightly better than the recycled fiber in flexural and tensile strengths and moderately better in moduli. These findings demonstrate the great potential

![](_page_2_Picture_10.jpeg)

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![](_page_3_Figure_3.jpeg)

Figure 2. The carbon fibers in PP and Nylon carbon fiber composites after the removal of the polymers. (a) PP/recycled fiber, (b) PP/virgin fiber, (c) Nylon/recycled fiber, and (d) Nylon/virgin fiber.

of the recycled carbon fiber as an effective replacement for the virgin fiber in reinforcing polymers, especially nonpolar PP. The differences of the two types of fiber in reinforcing PP and Nylon are due to their different bonding strength to the polymer matrices, which is further discussed below.

#### **Composite Morphology**

Figure 9 shows the SEM micrographs of tensile and impact fracture surfaces of PP reinforced with the recycled and virgin fibers. In all four micrographs, extensive fiber pull-out is evident and the fiber surface is free of polymer residual, indicating relatively weak interfacial binding between the fiber and PP. The PP matrix undergoes extensive plastic deformation (i.e., fibrilla-

**Table I.** Diameter and Length of the Recycled and Virgin Carbon Fibers(CF) in the PP and Nylon Composites

	Fiber diameter		Fiber length	
Sample	Mean (µm)	S. D.	Mean (µm)	S. D.
PP/Recycled CF	6.5	0.58	74.6	56.7
PP/Virgin CF	7.0	0.78	94.0	68.1
Nylon/Recycled CF	5.6	0.83	57.9	39.4
Nylon/Virgin CF	6.7	0.91	76.3	70.4

tion) in tensile testing [Figure 9(a,c)] and a brittle fracture in high rate impact deformation [Figure 9(b,d)]. The absence of carbon fiber fracture on the surfaces implies that the tensile stress applied on the carbon fiber through interfacial shear stress transfer is smaller than the failure stress of the fiber.<sup>28</sup> However, the tensile stress on the fiber is still larger than that on the PP matrix, which leads to the increases in the tensile and flexural strength of the composites (containing the recycled fiber) as shown in Figures 3 and 5. PP is a non-polar polymer that has negligible chemical and physical interactions with the carbon fiber (especially the virgin fiber, which is treated to be compatible with Nylon). The level of interfacial shear stress transfer between the two phases depends primarily on the mechanical friction force on the interface exerted by thermal shrinkage of PP. The results in Figures 3 and 5 demonstrate that for the recycled fiber, the shrinkage-induced friction force alone is able to cause an interfacial shear stress that is large enough to reinforce PP. The reinforcement caused by the recycled carbon fiber, which does not occur on the virgin fiber, is most likely due to the former's higher surface roughness (compared with the latter's) caused by the thermal process [Figure 1(d)], which effectively increases the friction force on the interface. This hypothesis is strengthened by the fact that the virgin carbon fiber is longer (Table I) and stronger<sup>10,17</sup> than the recycled one

![](_page_3_Picture_11.jpeg)

![](_page_4_Figure_3.jpeg)

Figure 3. Flexural strength of carbon fiber reinforced PP and Nylon at varying fiber mass fractions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

![](_page_4_Figure_5.jpeg)

Figure 4. Flexural modulus of carbon fiber reinforced PP and Nylon at varying fiber mass fractions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

![](_page_4_Figure_7.jpeg)

Figure 5. Tensile strength of PP and Nylon at varying fiber mass fractions. [Color figure can be viewed in the online issue, which is available at wileyon-linelibrary.com.]

![](_page_5_Figure_3.jpeg)

Figure 6. Young's modulus of PP and Nylon at varying fiber mass fractions. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

(pyrolysis reduces fiber strength), which, based on the composites theory, should lead to larger reinforcement to the matrix polymer, assuming equal level of interfacial bonding in the two composites. For the virgin carbon fiber, its stress concentration effect, which decreases the strength of the composites, is believed to dominate its reinforcing effect due to the extremely poor fiber-PP interfacial bonding. As a result, the tensile strength of PP/virgin fiber composites is decreased.

The fracture surfaces of carbon fiber reinforced Nylon composites are very different from those of the PP composites (Figure 10). First, the lengths of the fibers that have been pulled out from the Nylon matrix are much shorter than those from the PP matrix, indicating that most of the fibers have fractured instead of simply being pulled out from the matrix. Second, the fiber-polymer interfacial bonding in Nylon is stronger than that in PP. This is clearly demonstrated in Figure 10(b,d), where no fiber-Nylon interfacial debonding can be seen. In contrast, fiber-PP debonding and pullout is widespread in Figure 9(b,e). The strong fiber-Nylon interfacial bonding allows large shear stress transfer onto the fiber and hence increases the tensile stress of the fiber. As a result, many fibers fracture under this elevated tensile stress. Two factors are believed to cause the stronger interfacial bonding in the Nylon composites. First, Nylon's high polarity allows strong physical attractions between the polymer and the polar reaction products (or coatings) on the recycled (or virgin) carbon fibers. Second, Nylon has a larger thermal shrinkage rate than PP,<sup>29</sup> which causes a higher pressure on the fiber.

The different reinforcement results of the recycled and the virgin fibers to PP and Nylon worth further discussion. The virgin fiber possesses longer length and higher strength/modulus than the recycled fiber. In theory, it should lead to higher reinforcement to the composites. However, the results for PP have shown the opposite. This is due to the poor interfacial bonding between PP and the two fibers, which results in fiber pullout before the tensile stress on the fiber even approaches its failure stress. In other words, the strength and the length of the fiber is not the controlling factor of the strength of the composites.

![](_page_5_Figure_9.jpeg)

**Figure 7.** Comparison of the effects of the recycled and virgin fiber on the mechanical properties of PP. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

![](_page_5_Figure_11.jpeg)

Figure 8. Comparison of the effects of the recycled and virgin fibers on the mechanical properties of nylon. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

![](_page_6_Figure_0.jpeg)

Figure 9. Tensile (a and c) and impact (b and d) fracture surfaces of PP comprising 30 wt % carbon fiber.

![](_page_6_Figure_2.jpeg)

Figure 10. Tensile (a and c) and impact (b and d) fracture surfaces of Nylon comprising 30 wt % carbon fiber.

![](_page_7_Figure_3.jpeg)

Figure 11. Impact strength of PP and Nylon composites comprising different contents of carbon fiber. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Instead, surface roughness of the fiber, which determines stress transfer on the interface, dictates the properties of the composites. The recycled fiber exhibits higher surface roughness than the virgin fiber and therefore allows larger load to be transferred to the fiber, leading to higher reinforcement.

However, this is not the case when interfacial bonding is strong as in the Nylon/carbon fiber system. In this system, the fibers (recycled or virgin) predominantly fracture because the tensile stress on the fibers exceeds their failure stresses due to high stress transfer on the interface. Therefore, the strength of the fiber becomes critical to the strength of the Nylon composites. As a result, the virgin fiber shows higher reinforcement than the recycled fiber due to its higher strength (and longer length).

It should also be pointed out that the virgin carbon fiber does show a clear advantage over the recycled fiber in increasing the impact strength of both PP and Nylon. Figures 11 and 12 show that the virgin fiber causes higher impact strength than does the recycled fiber at the same fiber concentration. This is mainly due to the virgin fiber's longer length and higher strength. During impact deformation, fracture of the matrix, fiber pullout and fiber fracture occur simultaneously (Figures 9 and 10). Long fiber length causes high energy consumption for fiber pullout and high fiber strength results in large energy consumption for fiber fracture. Moreover, long fiber tends to more effectively deflect or stop crack propagation. As a result, more energy is required to fracture the virgin fiber composites and therefore their impact strength is increased more.

The comparative studies conducted in this article show that recycled and virgin carbon fibers can affect the properties of the composites in different ways. Which fiber to use in a composite application would depend on the type of polymer matrix, targeted composite properties and cost considerations. The recycled fiber, if without any further surface treatment, lacks compatibility with the matrix and depends primarily on surface roughness for interfacial stress transfer. Its insensitivity to the chemistry of the matrix polymer enables it to reinforce polar and nonpolar polymers alike in terms of tensile/flexural/impact properties. The virgin fiber, due to its surface treatment for an intended matrix polymer, is very selective about the polymer to be used within. Strength of the material can even be decreased after the addition of the fiber if an unsuitable matrix polymer is used. However, when the intended polymer is used, the virgin fiber outperforms the recycled fiber in reinforcing the material.

#### CONCLUSION

The high value of the recycled carbon fiber is best realized through an efficient CFRP recycling method and a thorough understanding of the properties of the recycled fiber. Through this study we found that microwave irradiation was a flexible, easy-to-control, efficient technique to reclaim the carbon fiber from CFRP composites. Without any further treatments, the

![](_page_7_Figure_12.jpeg)

Figure 12. Comparison of the effects of the recycled and virgin fibers on the impact toughness of PP and Nylon. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

recycled fiber can be directly used as reinforcement in the new polymers (PP and Nylon) using traditional composite processing technology. Most of the mechanical properties were significantly increased by the fiber. The property comparison further showed that the recycled fiber performed better in nonpolar PP while the virgin fiber dominated in polar Nylon. This was ascribed to the different fiber surface roughness and surface bonding characteristics of the two fibers. This discovery provides guidelines for intelligent use of the recycled carbon fiber.

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