## Application of Ultraviolet Photografting Technology to Acrylamide/Polyethylene to Improve the Interfacial Adhesion of Polyethylene/Unsaturated Polyester Resin Composites

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**ABSTRACT:** This study was divided into two sections. In the first part, we used ultraviolet (UV) rays in the wavelength range 300–400 nm to remove the hydrogen atom from polyethylene (PE) and worked with a hydrophilic monomer to complete the grafting action. In the second part, we used the best conditions derived from the previous film grafting and applied them to fibers to achieve excellent adhesion for application in composite materials. For the handling process of the PE film, we initially used acrylamide (AM) as the monomer and then added acetone and benzophenone (BP) to form a reactive solution for the advanced photografting process. In general, the optimum concentrations of the monomer solutions obtained from the photografting of PE films were 2 mol/L of AM and 0.2 mol/L of BP. The UV irradiation time was fixed at 30 min. The

#### INTRODUCTION

Finished products derived from polyethylene (PE) materials are cheap and easy to manufacture. Therefore, they are popular for many kinds of applications. However, these materials have a low surface energy  $(30.61 \text{ mJ/m}^2)^1$  and present surface inertia to prevent soaking or partial soaking, which thus decreases the adhesive and coating effects. Therefore, the printing and adhesive qualities of the finished products are markedly inferior. In our research, we aimed to execute a proper method for surface modification and to correct the aforementioned disadvantages.

PE surfaces do not have a functional group or a suitable solvent for dissolving. Although both are meritorious when it comes to applications, they do not produce sound printing, coating, and grafting and require another process to improve the surface quality of the materials. The current surface-modifioptimum grafting conditions achieved in the first part of this research were applied in the photografting process for the PE fiber bundles in the second part. The unsaturated polyester (UP) resins were spread over the outer surfaces of the modified fibers. This was done to strengthen and increase the interface between the UP resins and the modified PE fiber. During the curing experiment of the grafted fiber bundles in the resin coatings, the best material quality was obtained under the following conditions: hardener content = 0.85% (relative to the UP resin weight), oven temperature =  $80^{\circ}$ C, and time frame =  $5 \text{ h. } \odot 2008$  Wiley Periodicals, Inc. J Appl Polym Sci 111: 2507–2517, 2009

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cation methods<sup>2,3</sup> have rough finishing mechanisms; they include solvent, electric,<sup>4</sup> inflammation, acidtreatment, and plasma-induced surface treatments.<sup>4,5</sup> Excluding the modification methods just mentioned, grafting polymerization provides another type of surface modification, which can be divided into chemical grafting and radiation grafting.<sup>6</sup> Chemical grafting requires suitable initial preparation to induce the reaction, whereas radiation [electron beams from electron accelerators or  $\gamma$  irradiation from cobalt (Co-60) sources,<sup>7–9</sup> or ultraviolet (UV) rays from high-pressure mercury vapor lamps] grafting radiates a direct irradiation response and causes the monomer and the active backbone of the molecular chain to mutually interact. Both have their advantages and disadvantages. For example, radiation grafting does not have too many extra treatment steps in the grafting process.<sup>4</sup> Also, with radiation grafting, one can rapidly begin production<sup>10</sup> and does not need to consider the variation in material quality for incompletely eliminating the initiator mixed with the reactant agent.<sup>11</sup> In addition, the steps for radiation (UV-ray) grafting are simple and easy to perform. The whole grafting process is kept at low temperature (nearly 50°C);<sup>12</sup> there is not any

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variation in the internal structure of the base polymer during surface modification. This is the reason we used radiation grafting as the selected method in this study. By contrast, the percentage grafting in the chemical grafting process is higher than that in radiation grafting under the same conditions.

Related developments and applications formed by the chemical grafting method for improving surface qualities have been reported successively. For instance, in 1961, Lazar et al.<sup>13</sup> used the chemical modification of hydrocarbon polymers to make a graft of methyl methacrylate onto polypropylene (PP) and PE. Hitoshi et al.<sup>14</sup> used scanning electron microscopy (SEM) to discuss the surface morphology of a polyolefin grafting plate. Meanwhile, the research conducted by Samay et al.<sup>15</sup> used the process of grafting maleic anhydride onto high-density PE in the presence of various comonomers in an intermeshing corotating twin-screw extruder. Rengarajan et al.<sup>16</sup> offered the technique of solidphase graft copolymerization onto homopolymer PP to produce PP-maleic anhydride graft copolymers. As for the literature on radiation grafting for surface modification, there are also some reports introduced as follows. For example, in 1959, Gerald et al.<sup>17</sup> used the photografting procedure to irradiate plastics to produce a graft between pressure-sensitive tape and styrene monomer. Yang and Ranby<sup>18</sup> proposed a photografting process for producing dual-layer compounds. Chen et al.<sup>19</sup> used radiation ( $\gamma$ -ray) techniques for grafting PE onto conductive carbon black.

In the first part of this research, we performed UV photografting techniques to improve the surface qualities of low-density polyethylene (LDPE). Varied concentrations of hydrophilic acrylamide (AM) monomer were selected and matched with benzophenone (BP) to determine the optimum UV irradiation time. In the first part, we used LDPE as a raw material for making film. This film, made by a single-screw extruder accompanying the T-die and chilled roller device, had a texture composed of two phases,<sup>20,21</sup> crystalline lamellae and amorphous portions. The previously mentioned LDPE, as a common PE, contained 1000-3000 carbon atoms and had the following chemical structure:  $-(CH_2-CH_2)_n$ -. The LDPE molecular chains extending from the crystalline lamellae passed through several crystals and several amorphous regions and held the whole structure together under stress. As for the second part, we used fiber bundles made of ultrahighmolecular-weight PE. These molecular chains in the fiber texture had an ultrahigh degree of orientation in alignment. This ultrahigh-molecular-weight PE (weight-average molecular weight >3,000,000 for commercial products) fiber went with the three-step process for acquiring the internal molecular chain full orientation.<sup>22</sup> This three-step process<sup>23–26</sup> included (1) dissolution, (2) spinning, and (3) highdraw-ratio drawing. The three-step process as described (called *gel spinning*) provided a treated fiber with excellent physical properties,<sup>27</sup> and this is the main reason we considered the ultrahigh-molecular-weight PE fiber the best choice reinforcement for complexing unsaturated polyester (UP) resins after fiber surface modification and the addition of covalent functional groups.

At the onset, in this research, we first focused on the PE films, chiefly because they are low cost and easily acquired, and this directly hits the research key point of the universal learning of the photografting technique. Also, it is easier to judge the grafting yield<sup>28</sup> under a certain film status. The corresponding grafting conditions derived from the PE film could be directly applied to PE fiber in the same manner as methylene units  $(CH_2)$  for improving the hydrophobic characteristics of the original fiber surface. As a result, this allowed the PE fiber surface to produce a significant covalent bonding effect for UP resins after the grafting process and caused the original excellent ultrahigh-molecular-weight PE fiber to have notably increased physical properties. The content of this research focused on the developing film technology in relation to the fiber conditions. To date, however, little related literature can be found on the relevant applications of this type of indirect application method in composites research.<sup>18,29-33</sup> Thus, taking our research in this direction contributes to the development of composites and aims to provide another processing technology as a helpful reference in the interface research field.

#### **EXPERIMENTAL**

#### Materials

#### Manufacturing of the PE films

The LDPE plastic pellets required the use of Paxothene NA20766 produced by the USI Far East Corp. (Taiwan) with a melt flow index of 8.0 and a density of 0.923 g/cm<sup>3</sup>. The equipment used in the film making was Brabender Plastrograph single-screw extruder (Brabender Co., Germany). The temperatures between the screw extruder and barrel (from the starting point to the feed end) were successively set as 160, 170, 170, and 160°. The temperature of the extruder as it contacted the nearby circular chilled roller was fixed at 20°. The thickness of the manufactured film was 0.15 mm.

#### PE fibers

All of the fiber materials used were Spectra 630 denier and were highly intensified PE fibers produced by Allied Signal Co. (Signal, U.S.A). The fibers required a density of 0.925 g/cm<sup>3</sup>, and the average diameter was 0.03 mm.

#### Refined grafting monomer

To guarantee that the AM monomer had a higher purity for grafting, the following purification procedure was used. First, AM was dissolved in chloroform with a dissolving ratio of 0.1 g to 1 mL. Next, the temperature was simultaneously increased as the AM was stirred until it was completely dissolved. Finally, the solution was poured into a ceramic funnel attached to a flask and filter paper. This was sucked by a water vacuum pump device to recrystallize the dissolved substance. This procedure chiefly served to remove the impurities in AM and to produce a purifying effect. In addition, the filtration process was operated in coordination with the flask circled with ice cubes to boost the crystallization of the AM solution.

#### Photografting reaction

After radiation with 300–400-nm UV rays, the BP in the solution produced radicals under the active state. These BP radicals swiftly initiated further reaction with the PE molecular chain. All relevant reaction mechanisms are shown in Figure 1.<sup>12</sup> When the radicals and AM produced a chain reaction, this produced a homo–poly structure like a long chain. If these long chains connected with a film or fiber or the activated BP directly reacted with AM or the PE film or fiber, the UV photografting effect was obtained.

In the entire UV photografting process, the reactive solutions included acetone, AM, and BP. Excluding the acetone solvent, the three variables, namely, the monomer-producing AM, reactive BP concentration, and UV irradiation time, mutually formed a connection that had an enormous effect on the photografting rate. The grafting rate<sup>28,34–36</sup> can be expressed as

Grafting (%) = Weight of the grafts/  
Weight of the PE film 
$$\times$$
 100% (1)

The grafting rate was based on the significant weight changes before and after the film grafting reactions.

#### Processing

Photografting of the PE film surface

The purified AM monomer, reactive BP, and acetone (solvent) were individually fed into a flask on the basis of the tabulation ratio (see Table I). This mixture was passed through a magnetic rotor that stirred it until it was completely dissolved. Then, a



**Figure 1** Total reaction for the surface grafting of the PE film or fiber (RH = solvent or impurities with active hydrogen atoms, PH = homopolymer, PG = graft copolymer).

transparent and soft plastic film was used to seal the flask for later use. In another grafting process, after the PE film was cleaned with acetone, a clear marked film was placed between the glass vessel and the Pyrex plate (as shown in Fig.  $2^{12}$ ). The vessel rim and the Pyrex plate were tightly sealed with a screwing clamp. When the aforementioned mixed solution was poured into the glass vessel, it stayed in the glass vessel infused with nitrogen gas for about 3 min. Then, it was subjected to UV rays 300-400 nm in wavelength for 30 min, and the distance between the PE film and mercury lamp was set at 11 cm. When the process was finished, the PE film was sequentially dipped into freshwater and acetone. It was then dipped into acetone again for 8 h and rinsed with clear water to fully clean the unreacted monomers. Afterward, the treated film was left on filter paper until it completely dried out.

Photografting of the PE fiber surface

The photografting process of the PE film surface was repeatedly performed to search for the best conditions. The following conditions were assigned: 50 mL of acetone solution, a monomer AM concentration of 2 mol/L, and a reactive BP concentration of 0.2 mol/L. The PE fibers poured into a glass vessel (see Fig. 2) were exposed to UV irradiation for 30 min. The photografting treatment of fiber bundles was performed at the end of this process.

	Fixed BP and time					Fixed AM and time				
Sample code	A-1	A-2	A-3	A-4	A-5	B-1	B-2	B-3	B-4	B-5
AM (mol/L) BP (mol/L) Acetone (mL) Time (min)	0.5 0.2 50 30	1.0 0.2 50 30	1.5 0.2 50 30	2 0.2 50 30	2.5 0.2 50 30	2 0.05 50 30	2 0.1 50 30	2 0.2 50 30	2 0.3 50 30	2 0.4 50 30
	Fixed AM and BP									
Sample code	C-1	C-2	C-3	C-4	C-5	C-6				
AM (mol/L) BP (mol/L) Acetone (mL) Time (min)	2 0.2 50 5	2 0.2 50 10	2 0.2 50 20	2 0.2 50 30	2 0.2 50 45	2 0.2 50 60				

TABLE I Specifications of the Specimens

#### UP resin coating of the PE fiber surface

The grafted and ungrafted PE fiber bundles coated with various percentages of UP resins and squeezed externally with a polyurethane-based roller were sent to an electrical oven for resin curing.

#### Testing

#### Contact angle testing

The contact angle of the liquid (distilled water) on the surface of the PE film was tested with a goniometer (Rame-Hart Instrument Co., model 500F1, U.S.A) to determine the degree of improvement in the hydrophobic PE interfacial modification after application of the photografting techniques.

#### Mechanical properties testing

The tensile properties of the fiber bundles were tested with a universal tensile tester (Zwick/Materialprufung, model 1456, Germany) with a driving speed of 30 mm/min in accordance with ISO 2062-1993.

#### Surface observation of the fibers

Surface observation was carried out with SEM (Hitachi, model S-3000N, Japan) to determine the difference between the grafted and ungrafted fibers coated and uncoated with UP resins.

#### Determination of the functional groups

Fourier transform infrared (FTIR) spectroscopy (Thermo FTIR Nexus 870, U.S.A) was used to determine the existence or nonexistence of functional groups in the molecular chains, and the test parameters were set as follows: number of sample scans = 32 and resolution = 4.000 cm<sup>-1</sup>.

#### **RESULTS AND DISCUSSION**

#### Optimum reactions obtained in photografting

Figure 3 shows that when the AM concentration and irradiation time were kept constant, the percentage grafting increased with increasing BP concentration.



**Figure 2** Surface photografting device: (a) 1000-W highpressure mercury lamp, (b) interference filter, (c) Pyrex plate, (d) reacting solution, (e) PE film, (f) glass vessel, and (g) clamping screw.



**Figure 3** Effect of the BP concentration on the grafting percentage with a PE film irradiation time of 30 min and an AM concentration of 2.0 mol/L.

Figure 4 shows that the minimum contact angle portion had a starting point in the plateau section of the BP concentration–angle curve. This starting point had a corresponding value of 0.2 mol/L in the BP concentration scale. This means that after grafting, liquid drops effectively moistened the film. Grafting indeed increased the surface energy of the PE film and formed a rather high ratio of hydrogen bonds for coupling with the liquid drops. Figures 3 and 4 reveal that a 0.2 mol/L BP concentration already achieved optimum results. Even when the grafting



**Figure 4** Relationship between various concentrations of BP in solution and the contact angle at a fixed concentration of AM (2 mol/L) and with a fixed irradiation time (30 min).



**Figure 5** Effect of the AM concentration on the percentage grafting for a PE film irradiation time of 30 min and a BP concentration of 0.2 mol/L.

process was repeatedly applied, it did not demonstrate any significant change in the dampness of the PE surface (contact angle).

Again, Figures 5 and 6 show graphical representations of the fixed BP concentration (0.2 mol/L) and irradiation time (30 min) vis-à-vis variations in AM concentration, percentage grafting, and contact angle. As shown in Figure 6, we know the contact angle demonstrated a minimum value when the AM concentration was 2 mol/L. At this point, the corresponding percentage grafting tended to settle at a



**Figure 6** Relationship between various concentrations of AM in solution and the contact angle at a fixed BP concentration (0.2 mol/L) and with a fixed irradiation time (30 min).



**Figure 7** Effect of the irradiation time on the grafting percentage for a PE film with AM and BP concentrations of 2.0 and 0.2 mol/L, respectively.

fixed value. The two diagrams show that the best AM concentration was 2.0 mol/L.

Figures 7 and 8 show graphical representations of the time and percentage grafting, as well as the time and contact angle, under fixed AM (2.0 mol/L) and BP (0.2 mol/L) concentrations. The two curves conformably show that the optimum UV irradiation time was 30 min. On the other hand, Figure 7 proves that there was no significant increase in the percentage grafting with the initial UV irradiation, chiefly because the oxygen absorbed by the PE film surface hindered the AM monomer's grafting reaction and further increased the induction period for the grafting polymerization process.<sup>17</sup> After a certain period of time, the content of nitrogen gas in the glass vessel increased. Also, AM monomer exposed to UV rays had increased impact energy and drove the oxygen out of the PE film in succession. At that instant, the AM monomer began its enormous grafting onto the film. This explains why a larger percentage grafting took place behind the transition point of the grafting curve. Integrating the experimental results obtained from the previous diagrams, we concluded that the best PE photografting conditions included a 2.0 mol/L AM concentration, a 0.2 mol/L BP concentration, and a UV irradiation time of 30 min. These values set an irradiation parameter, which was applied in the next stage, photografting in fiber bundles.

After the PE film was irradiated with UV rays at 300-400 nm for 30 min, FTIR spectroscopy was used to examine the surface grafting level. Figure 9 illustrates the spectra from before and after the reactions. According to these spectra, two peaks were noted: the carbonyl C=O stretching frequency at 1660  $\text{cm}^{-1}$ and the amine N-H stretching frequency at 3310 cm<sup>-1</sup>. These introduced peaks, the carbonyl (CO) and amine (NH) groups, increased the surface energy and produced a hydrophilic base capable of improving the original hydrophobic PE surface quality. Also, in the grafted film, the intensity of the characteristic bands of PE diminished and the broadening of some other peaks took place, which showed that AM was introduced as a graft onto the PE film. The experimental results also show that the grafting was effective in achieving a covalent bond between the PE film surface and the AM monomers [see



**Figure 8** Relationship between various irradiation times and the contact angle at fixed concentrations of AM (2 mol/L) and BP (0.2 mol/L).



Figure 9 FTIR spectra of the grafted/ungrafted PE films.



**Figure 10** Surface of the ungrafted PE fiber  $(1500 \times)$ .

reaction (2)]. When it contacted other hydrophiliccovered substances (e.g., UP resins), neither poor absorption nor block coating occurred:

$$P_{C}(CH_{2}-CH_{2}]_{n} + C = CC(=O) N \xrightarrow{hv} -[CH_{2}-CH]_{n} \xrightarrow{hv} P_{3}^{P_{3}} \xrightarrow{hv} -[CH_{2}-CH]_{n} \xrightarrow{hv} CH_{2}-CH_{2}-C(=O) NH_{2} \xrightarrow{hv} OR -[CH_{2}-CH]_{n} \xrightarrow{hv} CH_{2}-CH-C(=O) NH_{2} \xrightarrow{hv} CH_{2}-CH_{2}-C(=O) NH_{2} \xrightarrow{hv} CH_{2}-CH_{2}-CH_{2}-C(=O) NH_{2} \xrightarrow{hv} CH_{2}-CH_{2}-CH_{2}-C(=O) NH_{2} \xrightarrow{hv} CH_{2}-CH_{2$$

*h* is Planck's constant, *V* is frequency.

# Experiments on the nature and process of the fiber bundle grafting

Figures 10 and 11 illustrate the surface conditions of both the ungrafted and grafted PE fibers magnified at  $1500 \times$  through SEM. For the surface state of the ungrafted PE fibers, there were clearly line marks caused by the hole edge of the spinnerets during the filament-drawing process. On the other hand, the grafted PE fibers showed a mark-free, even, and smooth surface in which the line marks from the original fibers were thoroughly connected and covered by the grafted monomer. The hydrophilic PE fiber surface increased its activation energy and moisture absorption capacity and effectively raised its reaction abilities to outer circumstances.



**Figure 11** Surface of the grafted PE fiber  $(1500 \times)$ .

Figure 12 shows that the relative curve revealing the PE fiber bundles that were not subjected to the treatment of photografting withstood external forces at various temperature conditions. This curve evidently reveals that there was no significant change for the fiber bundles to withstand external forces before the ambient temperature of  $40^{\circ}$ . This means that at room temperature, the breaking load of the fiber bundles was 4 kgf. However, the PE fiber



**Figure 12** Relational curve between the environmental temperature and breaking load of ungrafted PE fiber bundles for withstanding external force.

**Figure 13** Relationship between the breaking load and various hardener concentrations when the surfaces of PE fiber bundles were coated with UP resins.

bundles treated with photografting and resin coating (as shown in Fig. 13) had a corresponding value of 12 kgf in breaking load when the hardener concentration was only 0.5%. Even more, it arrived at 16 kgf when the hardener concentration was increased to 0.85%. The main reason was that the hydrophilic resins and fiber bundles formed a covalent bond. The fibers not only were mutually bound together by resins but also effectively dispersed the external



Figure 15 Ungrafted PE fiber bundles coated with UP resins.

force among the fibers and, thus, increased the breaking load. Figure 14 shows that when the photografted fiber bundles were coated with UP resins, the fibers efficiently gathered to form bundles. By contrast, due to the poor wetting capability of the ungrafted PE fibers, the UP resins could not be widely spread among the fibers, and the state of fiber bundles separated into two groups and



Figure 14 Grafted PE fiber bundles coated with UP resins.



**Figure 16** Surface coated with UP resins for grafted PE fiber  $(1500 \times)$ .



**Figure 17** Surface coated with UP resins for ungrafted PE fiber  $(1500 \times)$ .

partially peeled off from the resins, as shown in Figure 15. Meanwhile, the scanning electron microscope images (as shown in Fig. 16) revealed that the surface of the photografted PE fiber had uniform and even UP resins after the coating treatment. However, for the ungrafted PE fiber, the surface was glossy, whereas the UP resin coating was no longer set, and only a small amount remained on the surface (as



**Figure 18** Relational curve of the environmental temperature and the elongation of ungrafted PE fiber bundles for withstanding external force.

shown in Fig. 17). This occurrence confirmed that coated resins with higher surface tension did not fully adhere to the fiber surface, and they could partially contract in certain sites on the fiber surface. Anticipatively, one may be unable to acquire the reinforcement effect in a fiber-filled matrix.

Integrating Figures 12 and 18, we found that the breaking load of fiber bundles clearly started to decrease when the ambient temperature was over 80°C. Simultaneously, with this level of temperature, a larger extension occurred with the way the fiber bundles were stretched by external forces. With the fiber bundles used in this experiment, a temperature of 80°C was the critical point of temperature during heat treatment. After the fiber bundle surface was coated with UP resin, the physical properties of the materials decreased at unsuitable curing temperatures. To prevent this, we used the previous method to obtain the optimum heating temperature for the curing stage. A thermocouple was used to identify the exothermic conditions of the UP resins<sup>37</sup> and determine the heating time. For the UP resins in this experiment, we used the aforementioned methods to derive the oven temperature and time frame of 80°C and 5 h, respectively.

In relation to the shear strength between the fiber bundles and UP resins, a tensile tester was used to pull the fiber embedded in the resin matrix (as shown in Fig. 19) and to judge the shear strength of each interface. The experimental results shown in Figure 20 reveal that the shear strength of the grafted fiber bundles was far greater than that of the ungrafted fiber bundles (nearly double). This result confirms that fiber surface grafting effectively



Figure 19 Conceptual diagram for shear strength testing with a universal tensile tester.



**Figure 20** Interfacial shear strength versus the contact length between the grafted and ungrafted fiber bundles and UP resins.

improved the interface strength between the fiber and resins and further produced an effect of reinforcement compounded by the fiber and resin matrix. By examining this diagram, we determined when the contact length of the fiber bundles and resins increased and the shear strength decreased. The main reason was that the strength of the cross section of fiber bundles was fixed, and when the contact length of the fiber bundles and resins increased, the load per unit area of the fiber flank was evidently reduced. Generally speaking, the contact length and the corresponding shear strength show an inversely linear relation. We used the following mathematical relation<sup>38</sup> to explain the relationship between the two:

$$l_c = (\sigma_{fu}/(2\tau_i)) \times d_f \tag{3}$$

where  $l_c$  is the contact length between the fiber and resins,  $\sigma_{fu}$  is the ultimate fiber strength,  $\tau_i$  is the shear strength of the fiber–resin interface, and  $d_f$  is the fiber diameter.

#### CONCLUSIONS

This article reports the use of photografting techniques to provide interfacial improvements to PE film and fiber bundles. On the basis of these results, the following conclusions were drawn:

1. UV photografting technology uses steps that are simple and easy to perform. This method does not have any natural variation in PE internal structure and is an effective surface-modification technique.

- 2. Absorption peaks were clear at 1660 and  $3310 \text{ cm}^{-1}$  in the infrared spectrogram. This confirmed that the AM monomer had an absorption action and was grafted onto the surface of the PE.
- 3. In the photografting process, there were three parameter values, namely, the AM monomer concentration, the BP concentration, and UV irradiation time. These values served to control whether the monomers in this experiment effectively had a covalent bond with the surface of the PE. The experimental results show the best grafting effect was obtained under the following conditions: 2 mol/L of AM, 0.2 mol/L of BP, and a UV irradiation time of 30 min.
- 4. After the grafted PE fiber bundles were coated with UP resin, the breaking load of the grafted fiber bundles was almost three times higher than that of the ungrafted fiber bundles. This means that the grafting treatment had a direct and positive effect on the physical properties of the fiber bundles.
- 5. During the curing experiment on the grafted fiber bundles in the UP resin coating, the best material quality was obtained under the following conditions: hardener content = 0.85% (relative to the UP resin weight), oven temperature =  $80^{\circ}$ C, and time frame = 5 h.

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