

**A simple method to enhance the interface properties of carbon fiber/polyamide 6
composites: Grafted caprolactam-blocked 3-isocyanatopropyltriethoxysilane on
carbon fiber surface**

Na Sun ^a, Bo Zhu ^{a,*}, Xun Cai ^{b,*}, Xiaomin Yuan ^a, Ye Zhang ^a, Jiaqi Zhou ^a, Shuhan
Yan ^a, Chengsen Li ^a, Mingzhe Zhou ^c, Qi Zheng ^a

*^aKey Laboratory for Liquid Solid Structural Evolution and Processing of Materials
(Ministry of Education), School of Materials Science and Engineering, Shandong
University, Jinan 250061, China*

*^bSchool of Computer Science and Technology, Shandong University, Jinan 250101,
China*

*^cSchool of Metallurgy and Materials, University of Birmingham, Birmingham B15 2TT,
Edgbaston, United Kingdom*

Corresponding Authors: E-mail: zhubo@sdu.edu.cn (B. Zhu) caixunzh@sdu.edu.cn (X.
Cai)

Experimental section

FTIR was tested in the range of 400-4000 cm^{-1} scanning wave number by KBr tablet method.

TG-DSC was tested in a nitrogen atmosphere, at a heating rate of 10°C/min, in the range of 30-600°C.

SEM used 15KV test voltage, and the sample was pretreated with gold spraying for 30s before testing.

The wettability of fiber and resin was tested by the capillary principle, and the test equipment is shown in Fig. S1 [1]. Specifically, put the fiber bundles in a capillary that is 70mm long with a 2:5 ratio between the fiber volume and capillary volume. Suspend one end of the fiber from an electron densitometer (ZMD-2) connected to a gravity sensor. The other end of the fiber can be brought into the PA6/formic acid solution by altering the lifting platform's height (the fiber end's depth into the resin solution is 0-2mm). Record how the gravity value changes over time until the gravity value stays within 10s. The record time for termination is T. When recording data, selects a data point every 1s within 0-T/10, 5s within T/10-T/5, and 10s within T/5-T. Finally, process the data and a curve equation defining the infiltration property is created by fitting the data. The fitting equation is

$$y_n = A_n(x) \pm B_n \quad (\text{Eq. S1})$$

where x is time and y_n is gravity value. The larger the value of A_n , the faster the fiber can infiltrate the resin and the better the infiltration performance. The greater the y_n value, the higher the resin adsorption capacity.

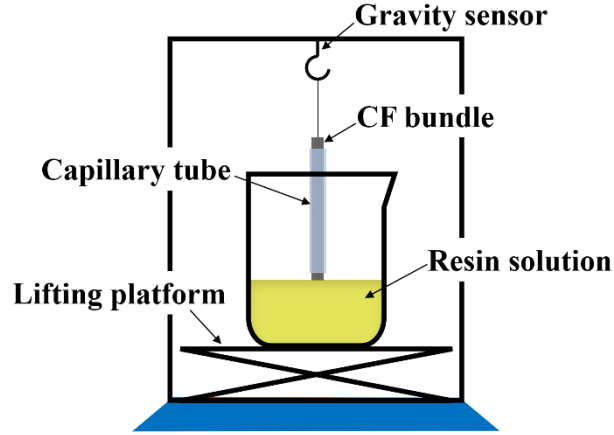


Fig. S1. Diagram of resin wettability test equipment

Single-fiber tensile [2]:

The schematic of single-CF tensile sample preparation is shown in Fig. S2. The single-fiber was separated from the CF fabrics, and both ends were fixed on the test paper. The test span is 20mm. The paper was fixed on the fixture of the fiber tensiometer, and the tensile rate was 2 mm/min. At least 35 single-fibers were tested for each sample, and taken the average. The strength calculation formula was calculated using the following equation:

$$\sigma = 4F/\pi d^2 \quad (S2)$$

where σ is single-CF tensile strength (Pa), F is maximum breaking load (N), and d is single-CF diameter (m).

The results were statistically analyzed using the Weibull distribution model.

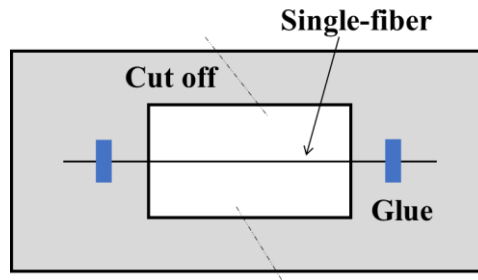


Fig. S2. Schematic of single-fiber tensile sample preparation

ILSS test:

The preparation schematic of CF/PA6 composite laminates is shown in Fig S3(a). 12 layers of unidirectional CFs were cut to size and stacked in a flat mold, which was pre-sprayed with a release agent. PA6 films were stacked alternately with CF at a mass ratio of 55:45. Then the mold was inserted into the curing press preheated to 240°C and closed under a low contact pressure of 0.5 MPa. After 10 minutes, the pressure was increased to 5 MPa and maintained for 6 hours. Turn off the heat, cool the mold to room temperature and unmold. Then the CF/PA6 composite laminates were cut into 20×10×2mm samples as shown in Fig. S3(b). The sample was placed on the supports, and a bending load was applied evenly at the center of the supports at a 1mm/min loading speed, as shown in Fig. S3(c). When the sample failed or the load reached the maximum value, stop the test and record the sample's failure mode and maximum load.

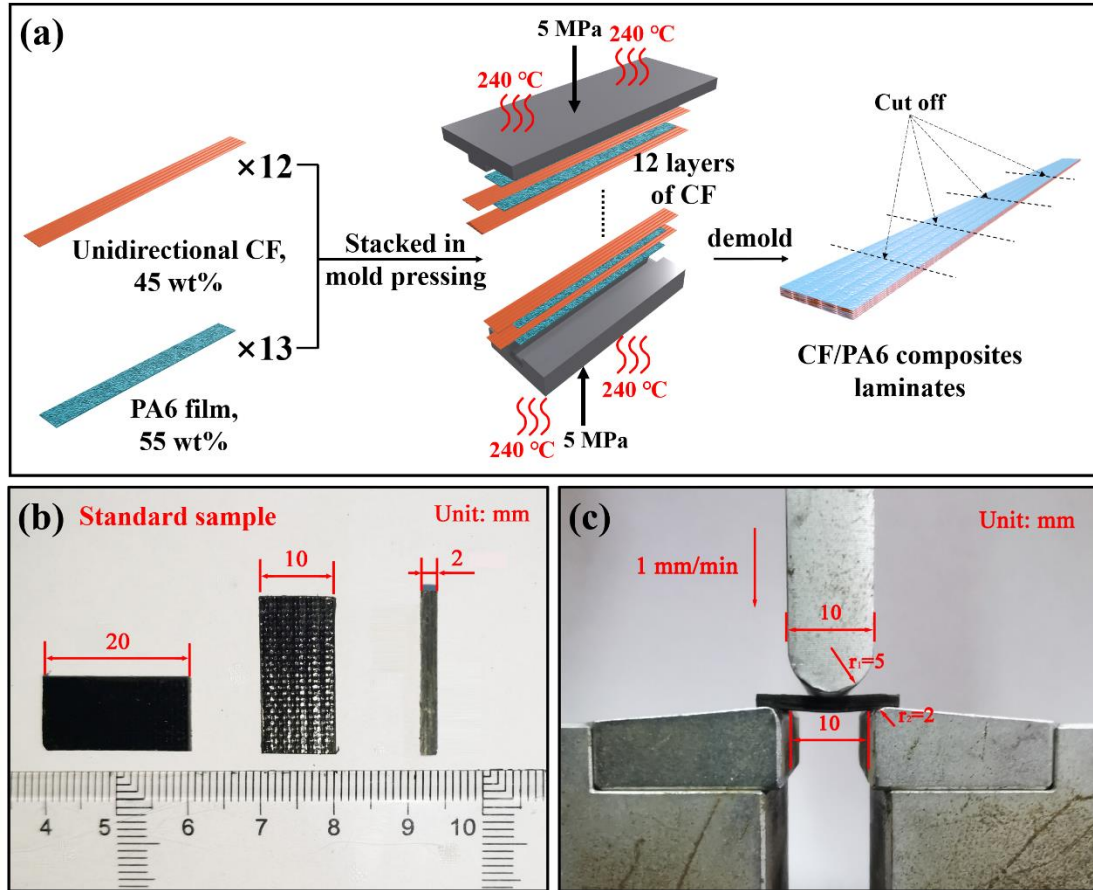


Fig. S3. (a) Preparation schematic of CF/PA6 composite laminates, (b) ILSS samples and (c) ILSS test diagram.

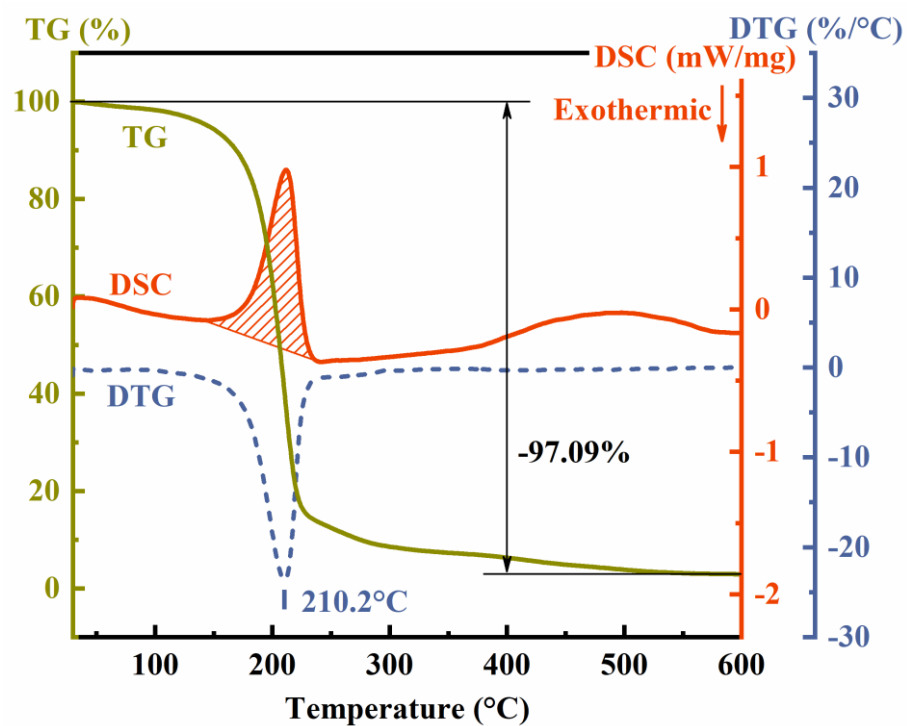


Fig. S4. The TG-DSC simultaneous thermal analysis of C₃IPTS coupling agent

Time	0s	1s	2s	3s	4s	5s	6s	7s	8s	9s	10s	11s	12s	13s	14s	15s
Desized																
CICF-0.1																
CICF-0.4																
CICF-0.7																
CICF-1.0																
CICF-1.3																

Fig. S5. The contact angle optical photographs of desized CF and CICF-

0.1/0.4/0.7/1.0/1.3

Table S1. Linear fitting functions of Weibull distribution.

Sample	Linear regression line	Standard error	Standard error
		of slope	of intercept
Desized	$y=6.03x-8.90$	0.179	0.251
CF-H ₂ O ₂	$y=6.95x-9.85$	0.297	0.401
CICF-0.1	$y=7.01x-10.02$	0.315	0.429
CICF-0.4	$y=7.07x-10.42$	0.186	0.261
CICF-0.7	$y=6.75x-10.28$	0.255	0.371
CICF-1.0	$y=7.15x-11.17$	0.277	0.414
CICF-1.3	$y=5.91x-8.86$	0.119	0.170

Table S2. Raw data for ILSS tests

Sample	Count (MPa)					Average	Standard
	1	2	3	4	5	value (MPa)	error (MPa)
Desized	34.61	41.96	44.84	44.89	45.83	42.43	4.12
CICF-0.1	41.86	44.00	46.12	46.64	46.90	45.11	1.92
CICF-0.4	41.76	49.96	50.28	51.42	51.54	48.99	3.67
CICF-0.7	46.20	51.01	52.42	53.42	53.58	51.33	2.72
CICF-1.0	51.28	51.38	52.08	53.39	54.54	52.53	1.26
CICF-1.3	45.05	49.36	53.09	53.32	53.94	50.95	3.36

References:

- [1]. Qin J, Wang C, Lu R, Su S, Yao Z, Zheng L, Gao Q, Wang Y, Wang Q, Wei H (2020) Uniform growth of carbon nanotubes on carbon fiber cloth after surface oxidation treatment to enhance interfacial strength of composites. *Compos. Sci. Technol.* 195:108198. <http://doi.org/10.1016/j.compscitech.2020.108198>
- [2]. Yao Z, Wang C, Qin J, Su S, Wang Y, Wang Q, Yu M, Wei H (2020) Interfacial improvement of carbon fiber/epoxy composites using one-step method for grafting carbon nanotubes on the fibers at ultra-low temperatures. *Carbon* 164:133-142. <http://doi.org/10.1016/j.carbon.2020.03.060>