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Interfacial Performance of Carbon Fiber Reinforced Bismaleimide Composites with Different Sizing Agents Under High Temperature and Humidity Condition

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This study addresses the crucial need to understand the interfacial behavior of GQ4522 carbon fiber reinforced bismaleimide (BMI) resin under conditions of high temperature and humidity, with a particular focus on the role of sizing agents. Through an integrated characterization approach that includes scanning electron microscopy, atomic force microscopy and surface energy measurements, combined with mechanical testing, this research elucidates how sizing agents influence fiber surface topography, specifically by reducing groove depth and enhancing uniformity, which affects interfacial compatibility. Key findings indicate the optimal sizing content necessary for achieving a balance between resin infiltration and fiber protection. Notably, GQ4522-7# fiber demonstrates a higher surface energy (43.45 mN/m), which is beneficial for wetting, while GQ4522-8# fiber exhibits a uniform sizing distribution, despite the presence of surface protrusions attributed to high viscosity. Mechanical testing results reveal distinct compatibilities: 7# sizing agent increases the resistance to high temperature and humidity of BMI-I composites, whereas 8# sizing agent improves the mechanical performance of BMI-II composites. This work contributes foundational insights into the interplay between fiber surface characteristics and sizing chemistry, establishing pivotal benchmarks for the advancement of high-performance composites in aerospace applications.

Keywords: sizing agent, interfacial performance, high temperature, humidity condition.

1. INTRODUCTION

In the recent years, carbon fibers reinforced thermoset composites have been widely used in aerospace fields due to their high specific strength, high specific modulus and excellent designability [1 – 3]. The numerous approaches concerning the surface treatment of carbon fibers have been studied proposed to improve the bonding between fibers and resin, while sizing agent is a good way to improve the interfacial properties. The sizing agents can change the carbon fiber surface properties, and enhance the interfacial performance between carbon fibers and resin through chemical bonding, physical anchoring, as well as the wettability and chemical reactions with matrix [4 – 6], thereby improving the mechanical properties and durability of the composite material.

Researchers have studied the interfacial properties of carbon fiber and bismaleimide (BMI) resin. For instance, Zhu et al. [7] analyzed the effects of temperature on mechanical properties of carbon fiber/BMI resin composites. Chen et al. [8] studied the bonding ability of carbon fiber and resin, with the increase in temperature, the bonding ability of the carbon fiber-BMI resin interface becomes weak, and debonding and delamination of resin are obvious. Sun et al. [9] investigated the correlations of the interphase with interfacial reinforcement and damage monitoring of carbon fiber/BMI composites after treatment at different temperatures, the partial detachment and decreased surface activity appeared with increasing temperature.

Although numerous studies [10, 11] have focused on the interfacial properties of carbon fiber/BMI resin systems, there remains a lack of comprehensive comparative research on the interfacial bonding behavior between GQ4522 carbon fibers and high-performance BMI thermoset resin matrices, as well as the effects of different sizing agents on the interfacial performance of GQ4522 carbon fiber-reinforced thermoset composites.

The present work proposes to characterize the sizing agents of carbon fibers and investigate the mechanical properties of carbon fibers reinforced BMI resin composites under ambient temperature and high temperature/humidity conditions, respectively, with the goal to assess the compatibility of sizing agents and BMI resins. The work aims to establish critical performance references for the application of GQ4522 carbon fibers in high-performance composite materials. These results provide valuable reference data of carbon fibers reinforced BMI resin composites in the aerospace fields.

2. EXPERIMENTS

2.1. Materials

Four batches of GQ4522 carbon fibers were used in this study, supplied by Weihai Tuozhan Fiber Co. Ltd., China. Meanwhile, two types of sizing agents were used, named as 7# and 8#, respectively. The former two batches of fibers were coated with 7# (named CF1-7# and CF2-7#), while the latter two were coated with 8# (named CF3-8# and CF4-8#).

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Two types of BMI resins used were also supplied by Weihai Tuozhan Fiber Co. Ltd (named BMI-I and BMI-II), which are both 180 – 225 ℃ cured with a service temperature range of - 59℃ – 204℃.

2.2. GQ4522/BMI composites

The four batches of GQ4522 fibers, which were coated with 7#and 8# sizing agents, reinforced two types of BMI prepregs were prepared in unidirectional tapes. Prepregs were laid-up following specified stacking sequence, which depend on samples requirement in each testing. Using an autoclave, the composites were manufactured with the same curing cycles, including three stages, which are 120 ℃ dwell 45 min, followed by 180 ℃ dwell 360 min, and then freestanding post-curing at 225 ℃ for 360 min. The bagging procedure is given in Fig. 1. Fiber volume fractions of all composite samples are controlled to be 60 %.

2.3. Characterization

2.3.1. Properties characterization of sized carbon fibers

According to GB/T 3362 standard, resin-impregnated continuous GQ4522 sized carbon fiber tows were prepared and tested to determine their tensile properties. Meanwhile, the linear density and volume density of sized fibers tows were measured following the same standard. Sizing content was evaluated by GB/T 26752.

2.3.2. Surface morphology of sized carbon fibers

To evaluate the effect of different sizing agents on the morphology aspects of carbon fibers, the specified amount of sizing agents were submitted on four bathes of GQ4522 fibers surface to make specimens. The specimens were fixed on metal stubs and then gold sputtered under a vacuum atmosphere, followed by examination via Quanta^{TM} 250 FEG Scanning Electron Microscope (SEM), focusing on the homogeneity along the fiber surface. The specimens were scanned under magnification varying 4000× to10000×. The unit operated at 20 KV.

2.3.3. Surface roughness of sized carbon fibers

Atom force microscopic (AFM) was performed to describe the surface roughness of sized carbon fibers, with a scanning region of 3 μm × 3 μm, using Veeco D3000 equipment. The value of Ra was calculated from AFM images using Nano Scope Analysis Software. At least 20 valid data were collected for each type of sized carbon fibers, and the standard deviation of *R*a was in the scope of 10 nm.

2.3.4. Surface energies of sized carbon fibers

Dynamic contact angles were characterized on sized carbon fibers via DCAT21 Dynamics Contact Angle Analyzer manufactured by Data Physics Corporation. Two different polar liquids, including deionized water and non-polar diiodomethane, were chosen for the testing. The surface energy (γ), which is considered as a sum of independent terms, representing a particular intermolecular force, can be divided into a polar term (γ^p) and a dispersive term (γ^d), as shown in Eq. 1. The surface tension of two testing liquids was listed in Table 1.

$$\gamma = \gamma^p + \gamma^d. \tag{1}$$

According to OWRK method, the surface energy of fiber can be calculated by Equation (2) and (3), in which the contact angle can be obtained from testing above.

$$\gamma_l(1 + \cos\theta) = 2(\gamma_s^d \gamma_l^d)^{1/2} + 2(\gamma_s^p \gamma_l^p)^{1/2}; \tag{2}$$

$$\gamma_s = \gamma_s^d + \gamma_s^p, \tag{3}$$

where γ_s, γ_s^d, γ_s^p are the surface energy, dispersive term and polar term of carbon fibers; γ_l, γ_l^d, γ_l^p are the surface energy, dispersive term and polar term of testing liquid, respectively.

2.3.5. Mechanical properties of GQ4522/BMI composites under high temperature and humidity

In order to evaluate the effect of moisture and temperature on the interfacial performance, mechanical properties of GQ4522 /BMI composites were tested on Instron 5967 equipment with an environmental chamber under room temperature and high temperature (150 ℃), respectively. The details for testing was shown in Table 2, where "150 ℃/WET" means the samples were immersed in water for 72 h before testing in a 150 ℃ environmental chamber, and "RT" represents room temperature. At least 6 samples were used in each test. It should be noted that 150 ℃ is the temperature that the load-bearing structure made of BMI resin matrix composites for supersonic aircraft needs to withstand over an extended period [12].

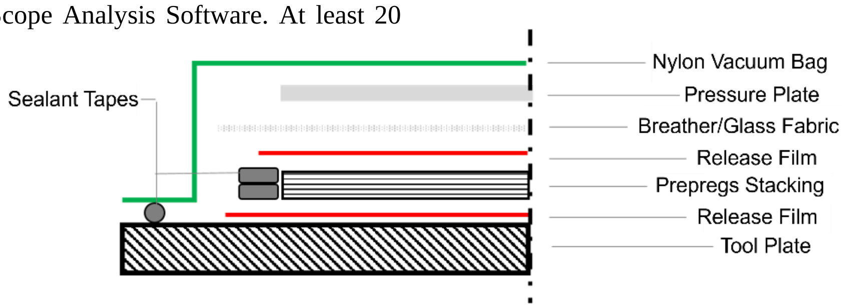


Fig. 1. Diagram of bagging procedure for GQ4522/BMI stacking

Table 1. Details of testing liquids in contact angles measurement

Testing liquids	γ^p, mN/m	γ^d, mN/m	γ, mN/m	Polarity γ^p/γ
Deionized water	50.7	22.1	72.8	0.7
Diiodomethane	0	49.2	49.2	0

Table 2. Testing properties of GQ4522/BMI composites

Properties	Testing condition	Standard	Stacking sequence
90° compression strength	RT	ASTM D 6641	$[90]_{16}$
	150°C/WET		
Flexure strength	RT	ASTM D 790	$[0]_{16}$
	150°C/WET		
Interlaminar shear strength (ILSS)	RT	ASTM D 2344	$[0]_{24}$
	150°C/WET		
In-plane shear strength	RT	ASTM D 3518	$[\pm45]_{5S}$
	150°C/WET		
Bearing strength	RT	ASTM D 5961	$[+45/0/-45/90]_{4S}$
	150°C/WET		
Compression strength after impact (CAI) 6.67 J/m)	RT	ASTM D 7137	$[+45/0/-45/90]_{5S}$

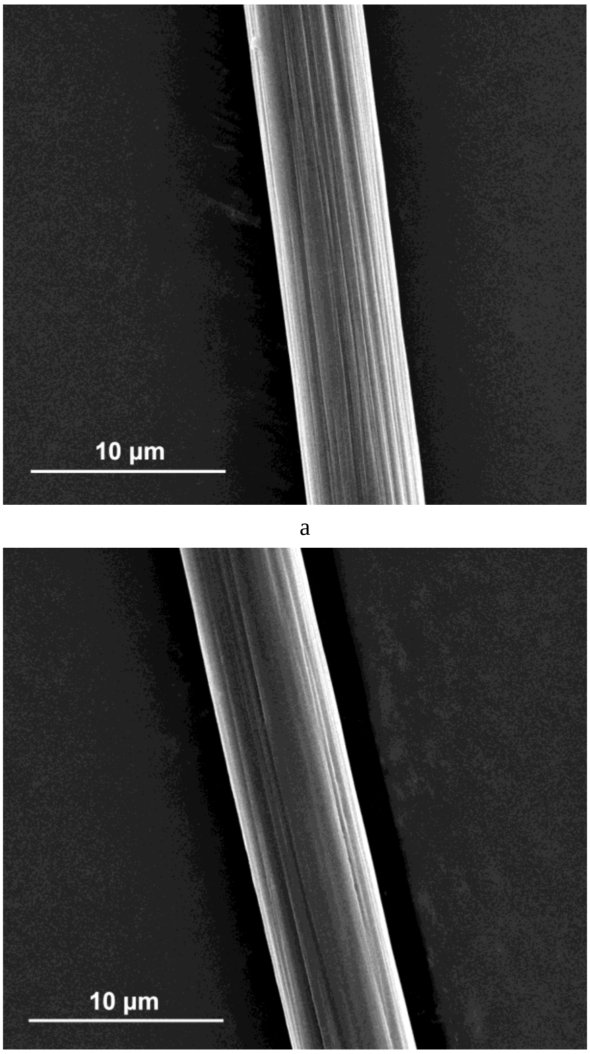
3. RESULTS AND DISCUSSION

3.1. Properties characterization of sized carbon fibers

In order to further understand the relationship between carbon fibers and sizing agent, Table 3 shows the characterization results of GQ4522 carbon fibers coated with sizing agent 7# and 8#. It is indicated that fibers coated with 7# has a slightly higher sizing content due to 7# presenting a lower viscosity at 30 °C, which leads to a solid content of 39.25 % for 7#, compared to 50.75 % for 8#. According to the previous study, the sizing agent is an important factor for composite manufacturing, acting as an intermediate phase to enhance the bonding between fibers and resins, preventing fiber surface damage and promoting the increase of interfacial properties [13]. However, excessive sizing content is disadvantageous to spread the fibers to meet the resins, leading to poor resin infiltration, which will be defects in composites. From tensile properties measurement, it is shown that the four batches of GQ4522 carbon fibers have consistent modulus, elongation and density, while tensile strength has a small change but still in the normal range between 4834 and 5133 MPa. Therefore, appropriate sizing content is required to obtain superior properties of carbon fiber.

3.2. Surface morphology of sized carbon fibers

SEM was employed to characterize surface morphology of 7# and 8# sized carbon fibers (GQ4522-7# and GQ4522-8#), as shown in Fig. 2. The results demonstrate that both 7# and 8# sizing agents form continuous and uniform coatings on the surface of carbon fibers, closely adhering to the surface of the carbon fibers without obvious agglomeration or peeling phenomena.



a

b

Fig. 2. Surface morphology of sized carbon fibers: a – GQ4522-7#; b – GQ4522-8#

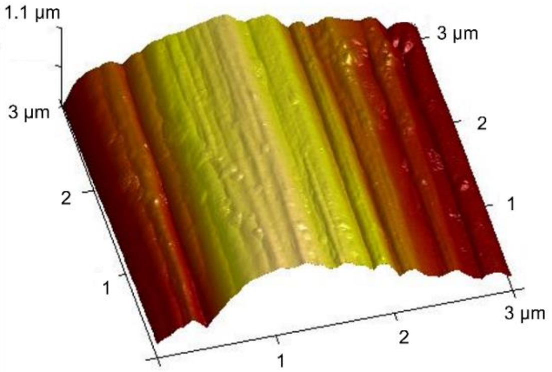
Table 3. Characterization of GQ4522 carbon fibers coated with sizing agent 7# and 8#

Content	Tensile strength, MPa		Tensile modulus, GPa		Elongation, %		Linear density, g/km	Volume density, g/cm^3	Sizing content, %
	Average	CV%	Average	CV%	Average	CV%			
CF1-7#	4851.35	2.95	251.20	1.28	1.94	2.53	800.15	1.80	1.26
CF2-7#	5107.13	2.51	258.33	0.95	1.98	2.36	795.83	1.80	1.34
CF3-8#	4834.12	2.91	251.07	3.45	1.92	3.58	802.93	1.80	1.22
CF4-8#	5133.50	2.37	261.28	1.10	1.94	2.28	800.94	1.80	1.08

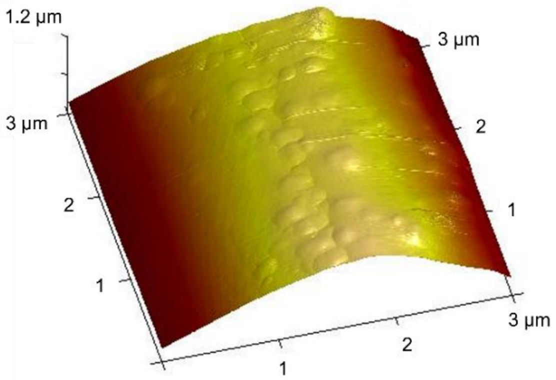
There are grooves along the fiber axis on both fiber surfaces, which are caused by the production process of PAN-based carbon fibers during the wet spinning process of PAN precursor. At this stage, the diffusion of the solvent and the traction force both cause the surface of the carbon fibers to collapse. Compared to GQ4522-7#, it can be seen from Fig. 2 b that the longitudinal grooves become shallower and more uniform on GQ4522-8#, which shows an even distribution of sizing agent.

3.3. Surface roughness of sized carbon fibers

More details about the surface topography of sized carbon fibers were obtained by AFM, as shown in Fig. 3.



a



b

Fig. 3. Surface topography of sized carbon fibers: a – GQ4522-7#; b – GQ4522-8#

The surface roughness was calculated, and the standard deviation was also listed in Table 4. As shown in SEM results, AFM results also convinced that there are obvious grooves along the fiber axis on GQ4522-7#, while GQ4522-8# shows shallower grooves, which is probably generated after the sizing step. The roughness of GQ4522-7# is 35.2 nm, higher than GQ4522-8# (17.2 nm), while the standard deviation is 9.3 nm (GQ4522-7#) compared to 4.6 nm (GQ4522-8#). It is also observed that a certain amount of protruding spots exist on GQ4522-8# fiber surface, which can be attributed to the higher viscosity of 8# sizing agent, as mentioned above.

Previous research has unequivocally established that sizing agents play a pivotal role in the performance of carbon fiber reinforced composites. These agents, applied to the surface of fibers, modify surface roughness, thereby significantly influencing the interfacial properties between fibers and resin [14 – 16]. Surface roughness is a critical determinant of mechanical interlocking and adhesion strength at the fiber-resin interface. When a sizing agent alters fiber surface texture, it directly affects the resin's wetting ability and adhesion to the fiber. Additionally, sizing agents can effectively bridge surface defects present on the fibers, such as micro-cracks and uneven grooves, that may act as stress concentrators under external loading. By filling these irregularities, sizing agents diminish stress concentration, subsequently reducing the external load experienced by the fiber during stress circumstances. Uniform distribution of sizing agents across the fiber surface is particularly advantageous, as it promotes a more consistent and smooth surface profile. This uniformity increases the contact points between the fibers and resins, resulting in a larger effective bonding area, which enhances interfacial bonding strength and improves overall load transfer efficiency between the fiber and the matrix. Conversely, the distribution of sizing agents along the fiber axis is critical; and nonuniform distribution can lead to several detrimental effects. An uneven application may create voids or areas of insufficient coverage, potentially trapping air during the composite manufacturing process and forming gaps at the interface between fiber and matrix. Such gaps disrupt continuous contact, impair wettability, and hinder the resin's ability to fully encapsulate and bond with the fiber, ultimately compromising interfacial properties. Consequently, the mechanical performance of the composite, including its tensile and shear strength, may be significantly compromised [17 – 19].

3.4. Surface energies of sized carbon fibers

It has been reported that the sizing agent can improve the wettability of fibers and protect their reactivity [20], which is essential to form the strong interface between fibers and resins. In order to estimate the wettability of the sized fiber surface, the contact angle of fibers was measured in two testing liquids, deionized water and nonpolar diiodomethane, as listed in Table 5.

Table 4. Surface roughness of two types of sized carbon fibers calculates from AFM images

	*R*a, nm	Standard deviation, nm
GQ4522-7#	35.2	9.3
GQ4522-8#	17.2	4.6

Table 5. Contact angles and surface energy of sized GQ4522 carbon fibers

Fiber type	Contact angle, °		γ^p, mN/m	γ^d, mN/m	γ, mN/m
	Deionized water	Diiodomethane			
GQ4522-7#	69.10±1.8	48.69±5.81	9.50	33.95	43.45
GQ4522-8#	73.44±8.3	60.98±11.99	9.71	27.17	36.88

Based on the results, surface energy, its dispersion component and polar component were calculated following Eq. 2 and Eq. 3. It is clear that GQ4522-7# has smaller contact angles for both deionized water (69.10°) and nonpolar diiodomethane (48.69°) compared to GQ4522-8#. The variation of contact angle in two testing liquids is ascribed to the polarity, which is 0.7 for deionized water and 0 for nonpolar diiodomethane.

As listed in surface energy results, both sized fibers have similar polar component, 9.50 mN/m for GQ4522-7# and 9.71 mN/m for GQ4522-8#, relative to the similarity of polar function groups on the sized fiber surface. However, it described a significant decrease in dispersion component, from 33.95 mN/m for GQ452-7# to 27.17 mN/m for GQ4522-8#, which is attributed to the roughness variation, since GQ4522-8# has shallower grooves and more uniform surface, as shown in the above results. Therefore, GQ4522-7# has a higher surface energy (43.45 mN/m) compare to GQ4522-8# (36.88 mN/m), which is beneficial to good wettability, resulting in better bonding and interface between fibers and resins.

3.5. Mechanical properties of GQ4522/BMI composites under high temperature and humidity

To evaluate the compatibility of GQ4522 carbon fibers with BMI resins, the composites were fabricated using GQ4522 fibers in conjunction with two types of sizing agents and two types of BMI resins across four batches. Mechanical performance was assessed through tests outlined in Table 2, considering the effects of temperature and humidity. The variation in test results is illustrated in Fig. 4. The results indicate that GQ4522 carbon fibers coated with 7# sizing agent demonstrate good compatibility with BMI-I type resin, exhibiting higher strength retention under elevated temperature and humidity conditions. In contrast, GQ4522 carbon fibers coated with 8# sizing agent display favorable compatibility with BMI-II type resin [21], as illustrated in Fig. 5.

The synergistic effect of the surface grooves of carbon fibers and the sizing agent significantly affects the interfacial properties of the composite material. The proper retention of the surface grooves enhances mechanical engagement between the fibers and the matrix. Meanwhile, the presence of the sizing agent effectively increases the contact area between the fibers and the resin, and at the same time reduces the stress concentration points. It improves the interfacial bonding strength by enhancing the wettability and the ability of chemical bonding.

4. CONCLUSIONS

This paper aims to study the impact of sizing agents on the interfacial properties of GQ4522 carbon fiber reinforced BMI composites under high temperature and high humidity conditions. By bridging surface defects and modifying roughness, sizing agents synergistically enhance mechanical interlocking and chemical bonding, which are beneficial for load transfer and durability of composite. Carbon fiber tensile tests reveal that appropriate sizing content is required to obtain superior property of carbon fiber.

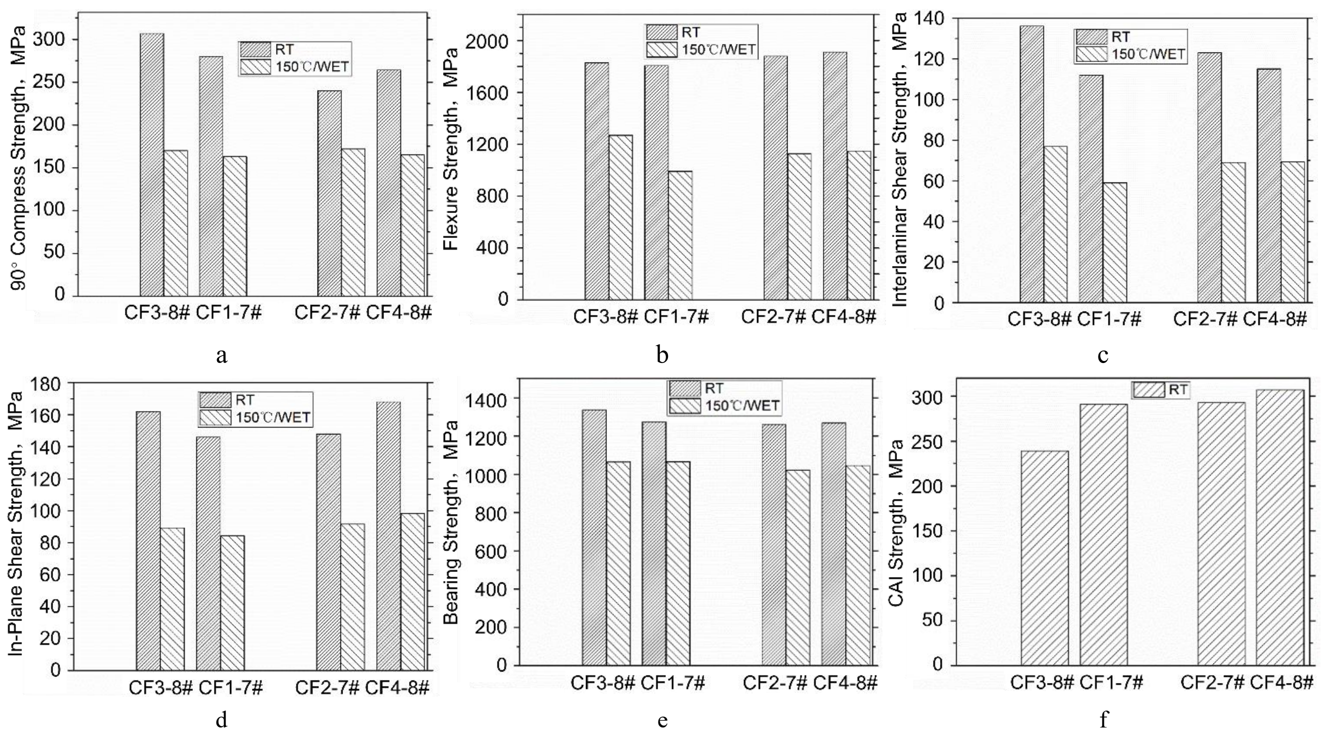


Fig. 4. Mechanical testing results for GQ4522 carbon fibers reinforced BMI-I type resin composites under room temperature and high temperature/humidity conditions: a – 90 ° compression strength; b – flexure strength; c – interlaminar shear strength; d – in-plane shear strength; e – bearing strength; f – CAI strength

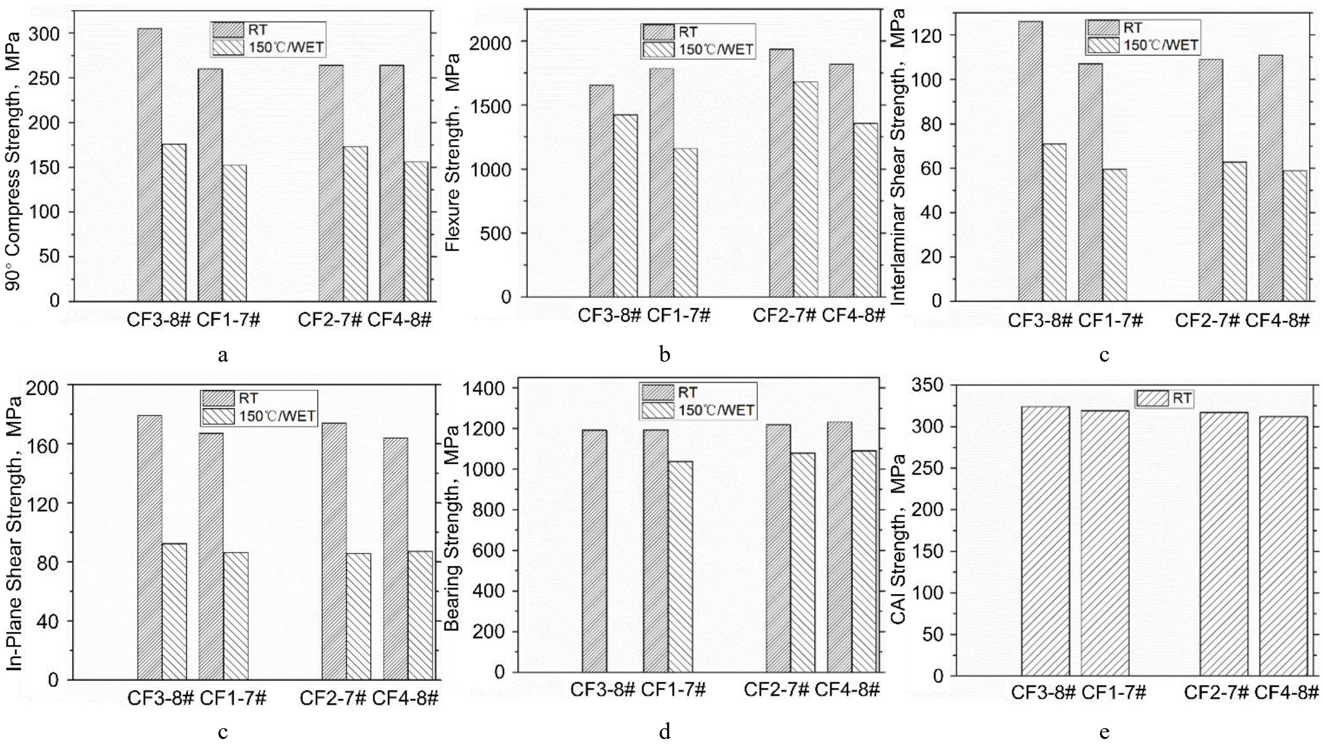


Fig. 5. Mechanical testing results for GQ4522 carbon fibers reinforced BMI-II type resin composites under room temperature and high temperature/humidity conditions: a – 90 ° compression strength; b – flexure strength; c – interlaminar shear strength; d – in-plane shear strength; e – bearing strength; f – CAI strength

SEM and AFM results shows that the longitudinal grooves become shallower and more uniform on GQ4522-8#, which shows an evenly distribution of sizing agents comparation to GQ4522-7#. It is also observed that a certain number of protruding spots exist on GQ4522-8# fiber surface, which can be attributed to higher viscosity of 8# sizing agent. The surface energy results indicates that GQ4522-7# has a higher surface energy (43.45 mN/m) compare to GQ4522-8# (36.88 mN/m), which is beneficial for good wettability, resulting in better interfacial bonding between fibers and resin. The mechanical testing results shows that GQ4522 carbon fibers coated with 7#sizing agent has a good compatibility with BMI-I type resin, while GQ4522 carbon fibers coated with 8# sizing agent has a good compatibility with BMI-II type resin. This study highlights the significance of aligning the properties of sizing agents with the chemistries of resin to enhance environmental resistance performance. The identified correlations among surface topography, surface energy, and mechanical performance provide a foundation for the development of next-generation carbon fiber composites, particularly in relation to GQ4522 fibers.

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