

High Temperature Experiments and Properties of Carbon Polyimide Composites

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Abstract. The interlayer shear tests of AC741/M55JB carbon fiber reinforced composite at room temperature, 400°C, 600°C and 800°C were carried out. The longitudinal and transverse shear tests of AC741/CCF800 carbon fiber reinforced composite were carried out at room temperature, 400°C, 600°C and 800°C dry conditions, as well as the experiments to calculate material property parameters. The results showed that the main failure mode of the sample in the interlayer shear test was stratified damage, and the fracture of the upper surface material was not obvious. This is due to the different types of tests, the interlayer shear pattern is short and small, and it is not easy to occur the fracture damage of the material without stratification. In addition, with the increase of temperature, the interlaminar shear strength also decreased significantly. In crossbar shear test, the maximum in-plane shear stress, maximum in-plane shear strength and in-plane shear modulus of the material gradually decrease with the increase of temperature, thus changing the bearing property of the material.

Keywords: Polyimide, composite material, high temperature test, mechanical properties.

1. Introduction

For nearly a century, resin matrix composites have been widely studied and applied in aerospace, rail transit and other industrial and civil fields because of their advantages such as low density, high mechanical strength, chemical corrosion resistance and strong design versatility^[1,2]. Among them, carbon fiber reinforced composite (CFRP) and glass fiber reinforced composite (GFRP) have become the two most used composite materials respectively for their outstanding structural strength and extremely attractive cost effectiveness^[3]. Polyimide composites not only have outstanding thermal stability, mechanical properties, radiation resistance and solvent resistance, but also have excellent performance in high temperature, high pressure and high speed and other harsh environments, it also plays a vital role in rocket engine shell, wound formation pressure vessel, sports equipment, automotive lightweight and other fields^[4,6]. On the other hand, polyimide composites have been widely used in printed circuit boards, building materials, wind turbine blades and other fields^[7,8]. For some high strength and high modulus high temperature resistant polyimide fiber is a kind of high performance organic fiber developed rapidly in recent years^[9]. Ke H^[10] et al. studied and prepared toughened PI (TPI/PI) blend, which showed that TPI/PI blend maintained the curing behavior and characteristics of PI oligomers, and the modified PI resin system had good thermal stability, excellent heat resistance and high toughness. Zhang H^[11] et al. maintained their high flexibility by attaching flexible blocks to rigid polymer structures to create thermoplastic structures. In this paper, the interlayer shear tests and crossbar shear tests of ISA-CCT800 carbon fiber reinforced composites were carried out at room temperature, 400°C, 600°C and 800°C dry conditions.

2. Experimental part

2.1. Preparation of Test Samples

The composite material used in this paper is carbon fiber reinforced resin-based prepreg with carbon fiber content $\geq 70\%$, which is used to make composite laminate samples. Clean the glass plate with alcohol, evenly wipe the defilm agent on the surface of the glass plate 3 to 5 times each vertically

and horizontally, and lay the layer according to the layering method of $[0^\circ/45^\circ/-45^\circ/90^\circ]_2$ s to prepare vacuum bags. The vacuum degree is about 100MPa. The purpose of vacuuming the laminate before prepreg curing is to make the laminate as dense as possible and reduce defects such as pores in the laminate. After that, the laminate was kept in a vacuum state, and the prepreg was heated to 120°C in a YB-WG-1000-D non-standard oven, kept for 1h30min, and then cooled to room temperature naturally to complete the whole curing process, as shown on the left in Figure 1. In the vacuum process, pay close attention to observe the vacuum degree to prevent air leakage during the oven heating process, so that the vacuum degree is reduced, so that the laminate vacuum is not tight, there are a large number of bubbles and other defects. Take out the test master plate cooled to room temperature, and use the LTJ2040-5A intelligent five-axis water knife cutting system to cut the laminated plate sample into $300\text{mm}\times 300\text{mm}$, as shown in Figure 1.



Fig. 1 The composite motherboard was vacuumed and cured in the oven.

2.2. Experimental Method

Before the test, the appearance of all samples was visually checked, and the samples were qualified without obvious dents, cracks, notches, surface irregularity, voids and delamination. Use an oil-based white marker to mark the sample, measure the sample size according to the test standard and record it. Experimental equipment and operating system: This experimental equipment uses the high-temperature furnace heating equipment of the Institute of Advanced Structural Technology of Arctic University of Technology, which is composed of the modified universal testing machine, water tank, vacuum pump and operating box, as shown in Fig 2.



Fig. 2 High temperature test equipment and operating system diagram

Specific test steps and experimental methods: Select the indenter and support, adjust the span of the support according to the thickness of the sample, align the loading head and support, make the

axis of the surface of the cylinder parallel to each other, and make the loading head located in the center of the two supports. The parallelism of the fixture can be adjusted by means of a flat plate with parallel grooves, and when fully aligned, the loading head and support fit with the grooves. Place the specimen in the center of the support, and make the long axis of the specimen perpendicular to the loading head and the support, as shown in Fig 3.



Fig. 3 Assembly test sample

Displacement loading is adopted, and the loading speed is 0.5mm/min. When the maximum strain on the outer surface of the specimen reaches 0.05 mm/mm, or the specimen breaks before reaching the maximum strain, the test is stopped. The failure load and failure mode of the specimen were recorded. Heat to the specified temperature for half an hour, then cool to room temperature and take out the sample.

3. Experimental Results and Analysis

The interlaminar shear strength, longitudinal shear strength and shear stress of the material were calculated according to formula (1) ~ (3)

$$F = 0.75X \frac{P_m}{bh} \quad (1)$$

$$S = \frac{P_{max}}{2wh} \quad (2)$$

$$\tau = \frac{P}{2wh} \quad (3)$$

Where: P_m is the peak load, l is the span of the sample, b is the width of the sample, h is the thickness of the sample, S is the shear strength, τ is the shear stress, P_{max} is the maximum load where the shear strain is equal to or less than 5%, P is the load borne by the sample, w is the width of the sample, h is the thickness of the sample.

The interlayer shear test data of AC741/M55JB at room temperature, 400°C, 600°C and 800°C are shown in Table 1, Table 2, Table 3 and Table 4, respectively.

Table 1. Interlaminar shear test data under dry condition at room temperature

Number	Length /mm	Width /mm	Thickness /mm	interlayer shear strength /MPa	Peak load/N	Failure mode
1	20.05	6.05	1.92	34.23	530.20	Stratification and fracture
2	20.04	6.08	1.75	43.11	611.60	Stratification and fracture
3	20.03	6.07	1.89	35.47	542.60	Stratification and fracture

Table 2. Interlaminar shear test data under 400°C dry condition

Number	Length /mm	Width /mm	Thickness /mm	interlayer shear strength /MPa	Peak load/N	Failure mode
1	20.05	6.05	1.92	31.24	480.36	Stratification and fracture
2	20.04	6.08	1.75	30.18	478.26	Stratification and fracture
3	20.03	6.07	1.89	31.48	482.61	Stratification and fracture

Table 3. Interlaminar shear test data under 600°C dry condition

Number	Length /mm	Width /mm	Thickness /mm	interlayer shear strength /MPa	Peak load/N	Failure mode
1	20.03	6.03	1.91	28.81	442.40	Stratification and fracture
2	20.02	6.03	1.81	29.91	435.20	Stratification and fracture
3	20.02	6.05	1.80	28.44	413.20	Stratification and fracture

Table 4. Interlaminar shear test data under 800°C dry condition

Number	Length /mm	Width /mm	Thickness /mm	interlayer shear strength /MPa	Peak load/N	Failure mode
1	20.04	6.03	1.76	24.42	345.60	Stratification, surface damage
2	20.03	6.07	1.85	22.44	336.00	Stratification, surface damage
3	20.03	6.09	1.79	24.27	352.80	Stratification, surface damage

The damage diagram of AC741/M55JB in the interlayer shear test under the dry condition of 800°C shows stratification and fracture, as shown in Fig 4.



Fig. 4 Failure mode of interlaminar shear test under 800°C dry conditions

The transverse shear test data of AC741/CCF800 at room temperature, 400°C, 600°C and 800°C under dry conditions are shown in Table 5, Table 6, Table 7 and Table 8 respectively, in which the strength and modulus are regularized data.

Table 5. Transverse shear test data under dry condition at room temperature

Number	Length /mm	Width /mm	Maximum in-plane shear stress/MPa	In-plane shear strength 5% /MPa	in-plane shear modulus /GPa	Failure mode
1	250.31	25.41	55.6	55.6	4.72	Working section 45 degrees oblique break
2	250.12	25.53	53.9	53.9	4.52	Root 45 degrees oblique break
3	250.23	25.60	53.8	53.8	4.51	Root 45 degrees oblique break

Table 6. Transverse shear test data under dry condition at room temperature

Number	Length /mm	Width /mm	Maximum in-plane shear stress/MPa	In-plane shear strength 5% /MPa	in-plane shear modulus /GPa	Failure mode
1	25.023	25.21	51.3	32.6	3.68	Root 45 degrees oblique break
2	25.042	25.43	48.0	30.5	3.36	Working section 45 degrees oblique break
3	25.013	25.10	47.8	29.6	3.13	Working section 45 degrees oblique break

Table 7. Transverse shear test data under dry condition at room temperature

Number	Length /mm	Width /mm	Maximum in-plane shear stress/MPa	In-plane shear strength 5% /MPa	in-plane shear modulus /GPa	Failure mode
1	25.014	25.06	44.5	30.6	2.91	Working section 45 degrees oblique break
2	25.033	25.21	45.6	29.8	2.97	Working section 45 degrees oblique break
3	25.011	25.19	44.8	27.4	2.86	Working section 45 degrees oblique break

Table 8. Transverse shear test data under dry condition at room temperature

Number	Length /mm	Width /mm	Maximum in-plane shear stress/MPa	In-plane shear strength 5% /MPa	in-plane shear modulus /GPa	Failure mode
1	25.019	25.23	36.7	29.8	3.07	Root 45 degrees oblique break
2	25.021	25.45	33.8	30.1	2.94	Working section 45 degrees oblique break
3	25.015	25.12	40.8	27.5	2.69	Working section 45 degrees oblique break

The damage diagram of AC741/CCF800 under the dry condition of 600°C shows that the root or working section is broken at 45°, as shown in Fig 5.

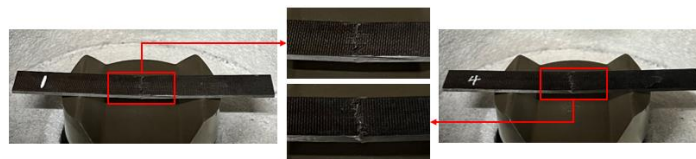


Fig. 5 The failure form was tested by crossbar shear test under the dry condition of 600°C.

4. Conclusion

In the interlayer shear test of AC741/M55JB at room temperature, 350°C, 600°C and 800°C dry conditions, the main damage form of the sample is stratified damage, and the fracture of the upper surface material is not obvious, which is due to the different types of tests, and the interlayer shear pattern is short and small, which is not easy to occur the fracture damage of the material without stratification. In addition, with the increase of temperature, the interlaminar shear strength also decreased significantly. The main failure mode of AC741/CCF800 under the dry conditions of room temperature, 400°C, 600°C and 800°C is 45° oblique break in the working section. With the increase of temperature, the compressive strength and modulus of the material decrease and the fracture strain of the material increases. The maximum in-plane shear stress, the maximum in-plane shear strength and the in-plane shear modulus of the material gradually decrease with the increase of temperature. In this paper, the mechanical test, analysis and summary of AC741/M55JB and AC741/CCF800 composites were carried out at room temperature, 400°C, 600°C and 800°C dry environment, which provided data support for material development and application.

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