AGING EFFECTS OF CARBON FIBER/BISMALEIMIDE COMPOSITE SUBJECTED TO THERMAL CYCLING

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ABSTRACT

Carbon fiber/bismaleimide composite are of interest for aerospace vehicle because of its excellent thermal stability, high specific modulus and high specific strength. In low earth orbit, satellites and aerospace planes passes in and out of the earth's shadow exposed to periodic sharp temperature changes. In this work, micro-cracks and mechanical properties changes of composites at high temperature and aged with thermal cycling were studied compared with that at room temperature. Short-beam shear tests were carried out at 200℃. Thermal cycling was conducted in the temperature range between -120℃ and 200℃. The method simulates the real environmental temperature condition in low earth orbit. Then reference and aged specimens were tested with three point bend method to investigate interlaminar properties of laminates. Cross and side section were polished carefully and observed with scanning electron microscope. No visible cracks were found on the specimens but micro-cracks were detected both on the cross and side section. Most of micro-cracks existed in the resin and interface between matrix and fibers. It could be found debonding of interface and fragments around fibers. It could be found that interlaminar shear strength decreased after specimens were subjected to thermal cycling. Interlaminar shear strength which is closely related to resin properties is weaken due to development of micro-cracks. Consequently, initiation of micro-cracks resulting from thermal cycling should be taken into consideration for aerospace applications.

1 INTRODUCTION

Fiber-reinforced polymer composites (FRPCs) have been widely used in aviation and aerospace field, where advantages of these materials including high specific stiffness and designability make them suited for weight-limit demand. However, FRPCs may be suffered severe environment in service such as temperature changes. In low earth orbit (LEO), satellites and aerospace planes passes in and out of the earth's shadow exposed to periodic sharp temperature changes [1, 2]. Consequently, FRPCs serving under this condition should have the ability to resist thermal-mechanical load at both high and low temperature. Bismaleimide (BMI) is a high-performance thermosetting polymer with excellent thermal stability, high specific modulus and high specific strength. Compared with epoxy which is often used below 150℃, BMI can resist high temperature environment better. Meanwhile BMI has high formability than polyimide although the latter one can be employed at higher temperature. As above states, carbon fiber/BMI composites have achieved more interests and is widely applied in aerospace field by reason of aggressive environments [3].

Thermal ageing effects on FRPCs were studied by researchers using different methods. FTIR analysis results showed that molecular chain changes occurred during thermal-oxidative aging process[4]. Mass loss presents anisotropic phenomenon [1, 5, 6, 7] and flexural properties changes due to thermal ageing [8, 9] were studied. High temperature created new challenges to predict composite structures'' service life. Thermal cycling effects on FRCPs were studied widely also. Matrix-dominant properties were focused on to exanimate [10]. Microscopy observation, C-scan and acoustic emission were used to monitor cracks development [11, 12]. Thousands cycles were conducted to age FRPCs then mechanical, chemical properties along with observation were conducted to evaluate the effects.

In this paper, thermal cycling has a more wide temperature range from -120℃ to 200℃.Based on the previous study[13], matrix-dominated properties of FRPC are sensitive to temperature. This study intends to evaluate effects of thermal cycling on interlaminar properties of carbon fiber reinforced BMI composite.

2 EXPERIMENTS

2.1 Materials

The studied carbon fiber/ bismaleimide composite was manufactured by stacking multiple layers of unidirectional ZT7H/QY9611 prepregs. Three kinds of layup are used in this study including $[0]_{24}$, $[0]_{16}$ and $[90]_{16}$ to evaluated thermal cycling effects on laminates with different fiber direction, each ply having a thickness of about 0.125 mm. The details of test specimens are listed in Table 1. The numbering rule is “Aged condition”-“Test temperature”-“Test type”-“Layer sequence”. “UA”, “A” and “TC” denote that aged conditions are unaged, aged at 200℃ and thermal cycling, respectively. “HT” and “RT” denote that test conditions are 200℃ and room temperature. “MT” and “CT” denote that test types are monotonic test and cycle test. “L” and “T” denote the layer sequence are longitudinal and transverse direction. Details are present as follows.

Number	Aged condition	Test condition	Dimensions	Layer sequence
UA-HT-MT	/	200℃	20×6×3	$[0]_{24}$
A-RT-MT	200℃ for 10min	RT	20×6×3	$[0]_{24}$
UA-RT-MT-L/T	/	RT	12×4×2	$[0]_{16}$ $[90]_{16}$
TC-RT-MT-L/T	Thermal cycling for 7 cycles	RT	12×4×2	$[0]_{16}$ $[90]_{16}$
UA-RT-CT	/	RT,Cyclic tests	12×4×2	$[0]_{16}$
TC-RT-CT	Thermal cycling for 7 cycles	RT,Cyclic tests	12×4×2	$[0]_{16}$

Table 1 Aged and test conditions and geometries of the test specimens.

2.2 Aged conditions

Some specimens were placed in chambers to age at 200℃ for 10min. First, specimens were placed in chamber. Then temperature began to raise with the maximum power. When the temperature reached to 200℃, it would be hold in 10min. Then the chamber was cooled with door closed to room temperature.

The thermal cycling tests were conducted by using two thermal chambers with cooling and heating function, respectively. Specimens were placed in the heating chamber for 120 min and then transferred to the cooling chamber for 120min. The procedure was repeated for seven times, which last 14 hours altogether as Figure 1 shown.

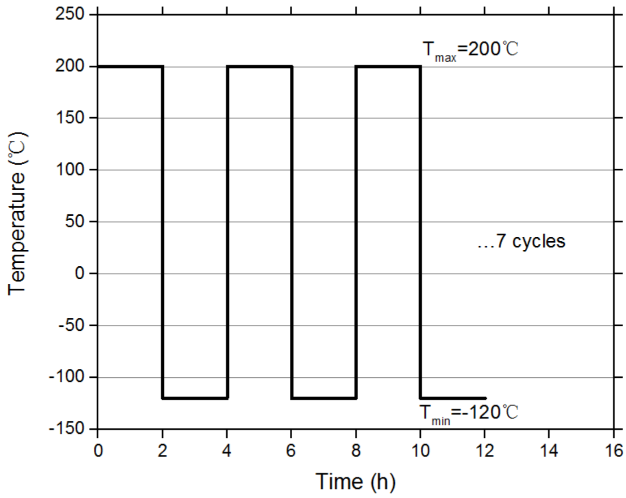

Figure 1 Thermal cycling temperature profiles.

2.3 Property evaluations

All specimens were cut to suitable dimensions from large panels in order to conduct interlaminar shear tests according to the standard [14] as show in Figure 2. All mechanical tests were conducted using a 30kN Instron 5567 electronic tensile machine (Instron, USA) with load rate equal to 1 mm/min until the load dropped to 30% of the maximum load. A temperature chamber consisting of heating system was designed and installed on the testing machine to control the environment temperature of specimens during the test. Specimen was placed in fixture, then the temperature in the chamber began to change as required. After reaching the specified temperature, temperature was held about 15 min before conducting the test to get temperature uniformity. Then the test began until the final failure occurred. Six repeats were carried out for each test configuration.

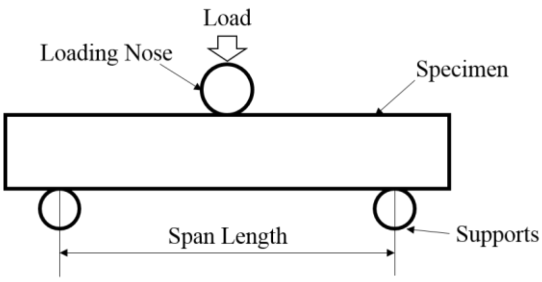

Figure 2 Schematic diagram for short beam strength test.

For observing purposes, cross-section of specimens were grinded using sand papers between 800 and 2000 grit to polish surfaces followed by polishing using 1μm and 0.5μm diamond paste. Two different direction to observe cross-section are showed in Figure 3.

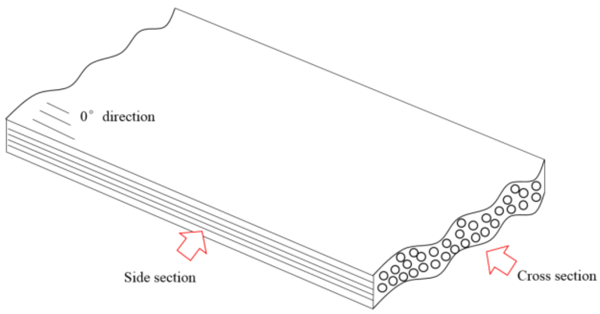

Figure 3 Microscopic observation of unidirectional laminates.

3 RESULTS AND DISCUSSION

3.1 Simulative temperature and pretreatment tests

Interlaminar shear responses of A-RT and UA-HT specimens are present as Figure 4 shown. Actually, aged in a short time has little effects on materials as studied earlier. Six repeats had highly repetitive in different conditions. The curves of A-RT specimens raised as a straight line until reached the maximum load values. Then curves began to drop as stepwise indicating that damage occurred progressively. But for UA-HT specimens, all damages generated at high temperature which showed different responses. Load rised with increasing displacement with a straight line trend. Curves turned to a more smooth trend when reached to the maxium points. The load dropped gradually which was totally different with that of A-RT responses. Polymer shows viscoelasticity properties at high temperature and interlaminar shear responses are affected by polymer behavior as well.

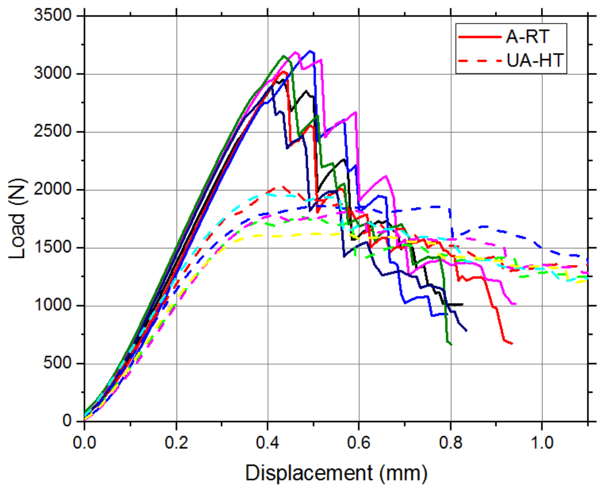

Figure 4 Interlaminar shear responses of A-RT and UA-HT specimens.

Fracture morphologies of side surfaces are present in Figure 5. For A-RT specimen, the fracture surfaces are smoother as Figure 5(b) shown. A straight fracture line runs through the whole thickness direction with some fragment included in cracks. At the part near to the fracture line, there are several unobvious delamination. But for UA-HT specimen, the fracture line is zigzag with larger fragments inserted in cracks. And it is obviously to found more delamination occurred in adjacent zones. From the detail view, fibers deboned with each other because of the high temperature effects on polymer's viscosity and interface's properties. More delamination and debonding in microscope occurred at high temperature so as to mechanical responses are different and the maximum load decreases as shown in Figure 5.

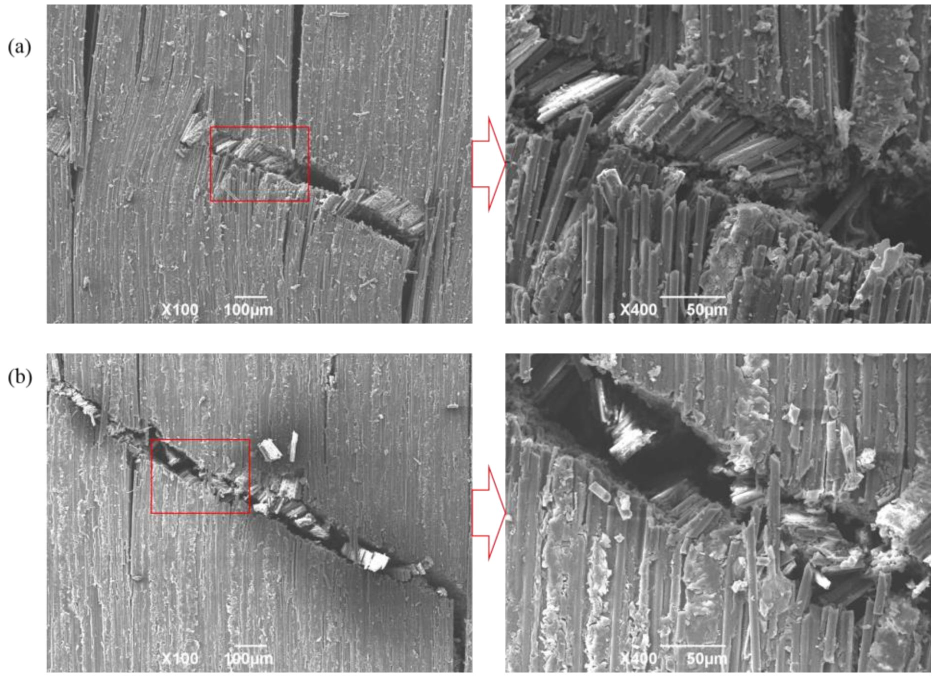

Figure 5 Side sufaces of (a) UA-HT and (b) A-RT specimens.

3.2 Thermal cycling

Interlaminar shear tests were conducted on $[0]_{16}$ and $[90]_{16}$ specimens to evaluate thermal cycling effects on interlaminar properties of composites. It is clearly seen from Figure 6 that the maximum loads decreased to 86% and 71% for $[0]_{16}$ and $[90]_{16}$ specimens, respectively. Interfaces are transition areas between resin and fibers, so thermal stress accumulate severely in these areas. Although no visible cracks occurred in laminates, micro cracks had generated in micro-interfaces as stated in following part.

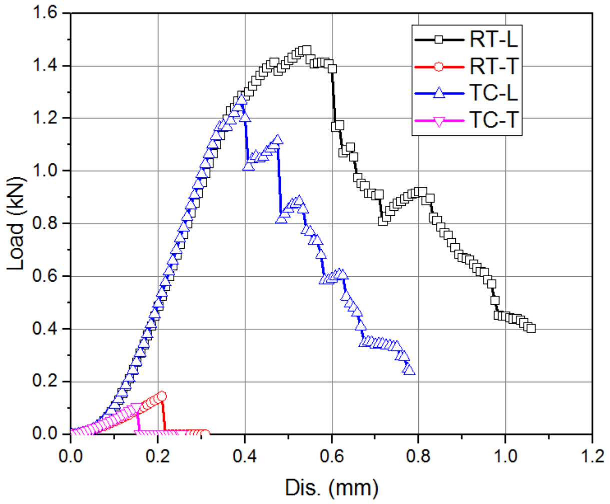

Figure 6 Interlaminar shear responses of aged and reference specimens.

Electron micrographs showing micro-cracks in cross section are present in Figure 7. Actually, there is nothing different between the two specimens in macro scale. In microscope, fibers and matrix bond well for UA-RT specimen. It can be seen smooth transition zone between fibers and matrix. But for TC-RT specimen, it is obviously to found interfaces around fibers are eroded seriously even some fragments exist in the interface. Fibers and polymer deboned due to differences between their coefficients of thermal expansion during the dramatic temperature changes. Under the cyclic thermal stress erosion, some fragments generate due to the original defects existing in material. Electron micrographs showing micro-cracks in side section are present in Figure 8 to show matrix changes at different aged conditions. Unbroken side section is present in Figure 8(a) to show UA-RT specimen. No obvious cracks are found in polymer except for some voids produced in manufacturing process. Fibers and polymer have a good bond in this condition. But aged during seven thermal cycles, more cracks in matrix can be seen in Figure 8(b) to present thermal cycling effects on microscope morphology. It is concluded that thermal stress introduced by thermal cycling is seriously enough to generate cracks in matrix. The effects have great influences in CFRPs' mechanical properties especially the matrix-dominant properties.

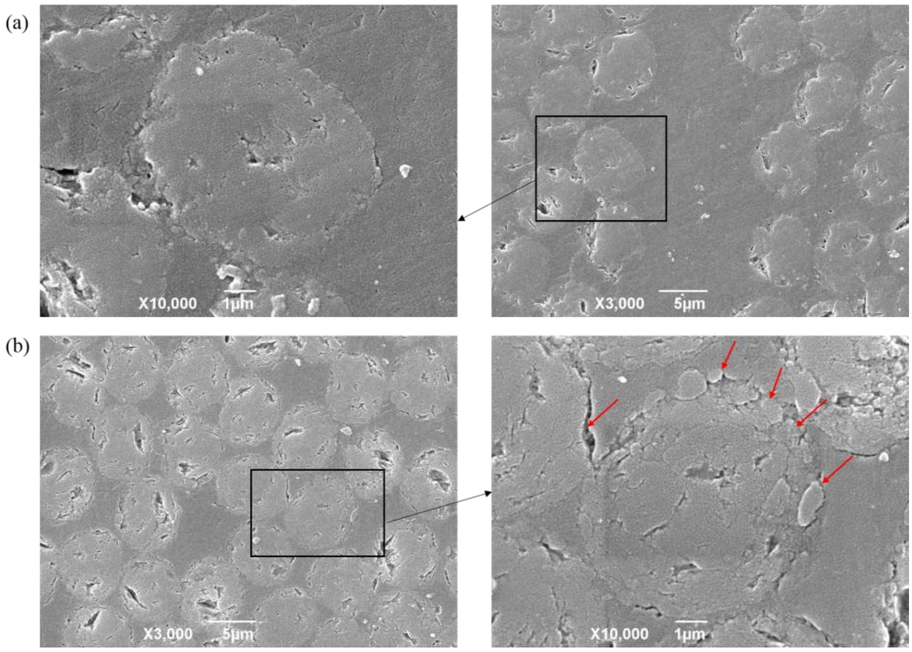

Figure 7 Electron micrographs showing micro-cracks in cross section (a)UA-RT (b) TC-RT

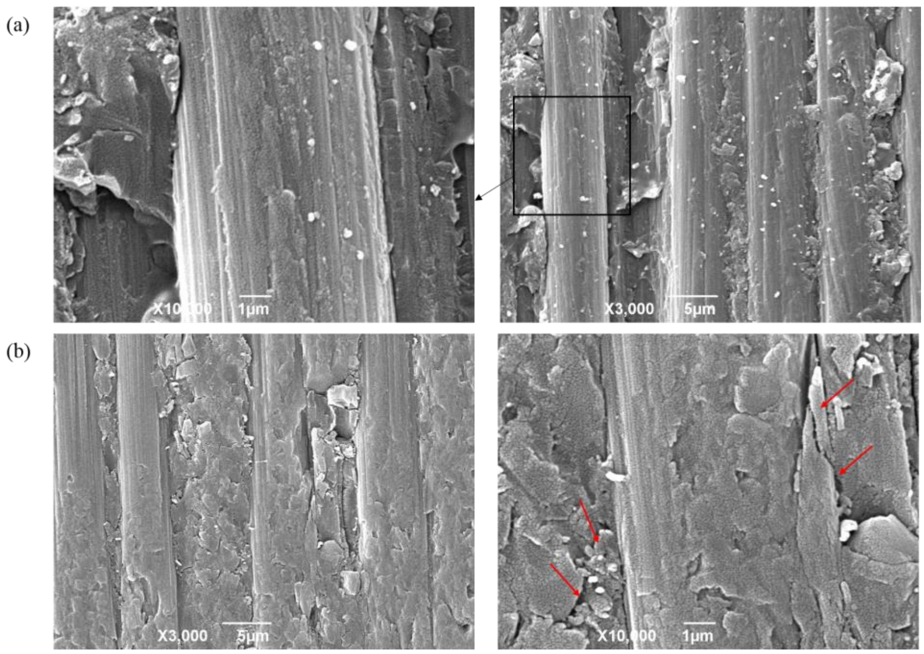

Figure 8 Electron micropraphs showing micro-cracks in side section (a)UA-RT (b) TC-RT

3.3 Damage accumulation

In this section, specimens were subjected to cyclic load to access damage accumulation in laminates. This processes were achieved by increasing the displacement step by step until final break occurred as shown in Figure 9(a).

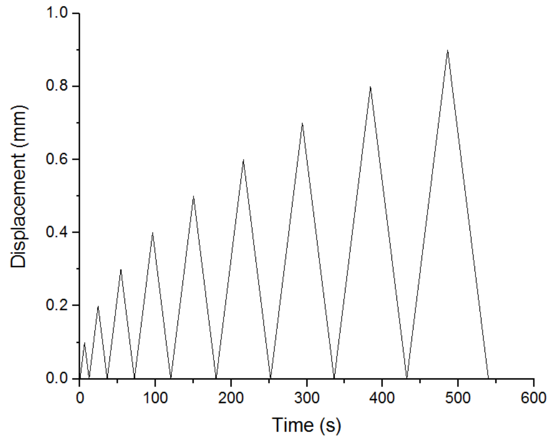

Figure 9 Displacement-time curves.

Load-displacement curves resulting from the previous processed are shown in Figure 10. The load changed periodically from a maximum value to zero, each cycle taking laminates closer to failure. The envelope of cyclic curves is similar to the monotonic curve presented in the previous section. Before the displacement reaches to 0.3mm, the cyclic curves are coincide with the monotonic curves. When the load dropped to zero, the displacement fall back to the initial state. It denotes that elastic deformation occurs in this stage. After that, the stiffness begins to drop and the maximum loads decrease after every loop until the final failure. Cyclic load can bring in interlaminar damage in FRPCs to reduce stiffness and ILSS.

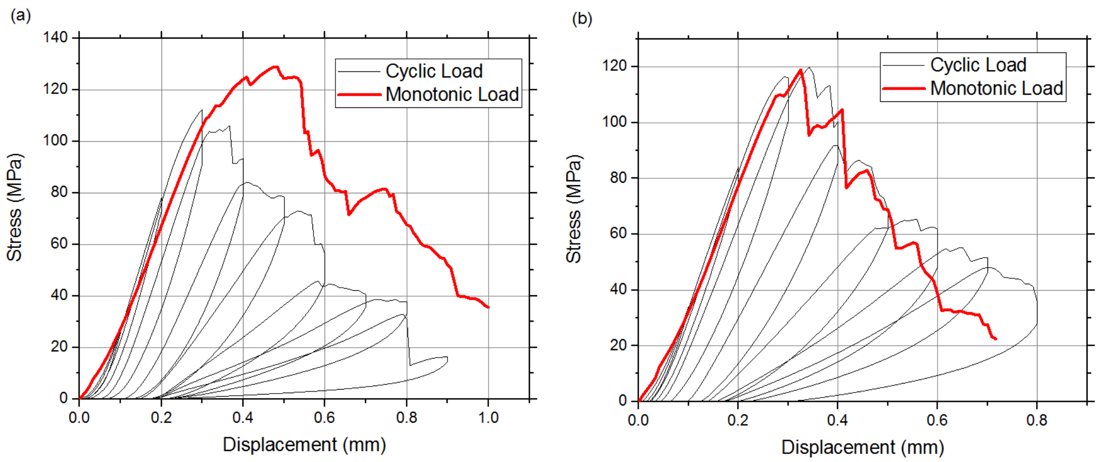

Figure 10 Cycild load and monotonic load responses of (a) UA-RT-CT (b)TC-RT-CT

4 CONCLUSIONS

In this work, micro-cracks and mechanical properties changes of composites at high temperature and aged with thermal cycling were studied compared with that at room temperature. The following conclusions can be drawn:

(1) ILSS of UA-HT specimens is about 30% off compared to A-RT specimens. Polymer shows viscoelasticity properties at high temperature and interlaminar shear responses are affected by polymer

behaviour which is different with the step by step failure mode at room temperature. And more delamination are found in fracture faces of UA-HT specimens.

(2) Although no visible cracks can be found in aged specimens, ILSS of TC-RT-MT-L specimens was 86% of that of the unaged specimens. It is obviously to found interfaces around fibers are eroded seriously even some fragments exist in the interface, which is due to coefficients of thermal expansion differences between fibers and polymer during the dramatic temperature changes.

(3) Cyclic load is applied to specimens to simulate damage accumulation. After the elastic stage, damage occurred and stiffness decreased, which denotes the damage degree in FRPCs.

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