Effect of Heating Rate on Interlaminar Shear Strength Property of Carbon Fiber-reinforced Composite with High-pressure Microwave Curing

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Abstract: Microwave curing has been deemed to be a rapid and cost-effective technology for curing carbon fiber-reinforced composite. In this study, a set of devices about high-pressure microwave curing were built with a real-time temperature measurement and control system of microwave input. Under a given high-pressure microwave curing process, different heating rates were applied to the carbon fiber-reinforced plastic (CFRP) laminates. The effect of heating rate on the curing quality was studied by interlaminar shear strength (ILSS), the void content of composite samples was observed by optical digital microscope(ODM) and the fracture surface of composite samples in these tests was inspected by scanning electron microscope (SEM). The results showed that the heating rate had a significant effect on the ILSS of CFRP laminates. The ILSS of CFRP laminates decreased and the void content increased with the increasing heating rate under high-pressure microwave curing. There were a large amount of hackles among carbon fibers when the heating rate was less than 6 °C/min. When the heating rate was 6 °C/min under high-pressure microwave curing process, the total time could be saved 28.85%, and the ILSS of sample was basically consistent with the value of sample in standard thermal curing.

1 INTRODUCTION

Carbon fiber reinforced plastics (CFRP) is widely used in various engineering application due to their characteristics of high mechanical properties, low density and flexible process ability. (Wisnom, 1999; Al-Saleh and Sundararaj, 2011; Gibson, 2010). Microwave processing can be considered as an alternative cost-effective route for curing CFRP composite due to its rapid and volumetric heating capabilities. Because of the specific heating mechanism, microwave curing offers many remarkable advantages over traditional thermal curing, including energy saving, low operating costs, high heating efficiency, increased throughput, shortened processing time and improved processing control. (Thostenson and Chou, 1999; Zhou et al., 2003; Mijovic and Wijaya, 1990). Therefore, a series of devicies about high-pressure microwave curing need to be built to realize relate heating rate. Meanwhile, it is important to understand the effect of heating rate on the interlaminar shear strength (ILSS) of CFRP laminates to choose the suitable heating rate.

There were many researchers who had great interest in microwave curing of composites because of its fast curing compared with the conventional thermal processing. (Thostenson and Chou, 1999; Bogdal et al., 2003; Thostenson and Chou, 2001; Fang and Scola, 1999). The main reason was that the electromagnetic radiation generated in microwave processing could penetrate the surface of the material and induce volumetric curing via rapid dielectric-related heat generation in the molecular level of the resin. (Jacob et al., 1995; Li et al., 2014; Li et al., 2018). For this different way of curing composites, some researchers studied the effect of temperature profiles of microwave curing on mechanical properties of CFRP lamanates. Xiang Hang *et al* studied the effects of different heating rate on the mechanical properties of CFRP laminates with microwave curing only at 0.1 MPa pressure. (Hang et al., 2017). However, curing pressure was one of the most significant factors in the process of

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curing forming of CFRP laminates. In previous works, the curing pressure was not less than 0.4MPa and if the curing pressure was too low, the composite could have plenty of voids that seriously affected mechanical propertise of CFRP in autoclave, which was consistent with microwave curing. (Li et al., 2014b; Li et al., 2015; Chen et al., 2017). Xiaoping Chen *et al* studied a novel method for curing CFRP laminate by high-pressure microwave. (Chen et al., 2016). Compared with traditional autoclave curing, the high-pressure microwave was able to save energy significantly, by which the mechanical properties of CFRP laminate specimens were enhanced in the same curing processing curve. Although a lot of scholars have done plenty of researches on the microwave curing of composite materials, there were few reports on effect of different heating rate on the ILSS about CFRP laminate with high-pressure microwave curing.

In this study, a set of devices about high-pressure microwave curing were built, and a real-time temperature measurement and real-time control system of the microwave input power were equipped. Furthermore, the effect of heating rate on the curing quality was studied by the ILSS and the fracture surface of composite samples in these tests was inspected by scanning electron microscope (SEM) in order to improve curing efficiency and save energy consume as well.

2 EXPERIMENTAL

2.1 Materials

The material for the experiment was the the T800/X850 carbon fiber reinforced plastics prepreg (purchased from Commercial Aircraft Co., Ltd., China). The CFRP prepreg was stored at -12 °C in a sealed contain, the physical properties of CFRP prepreg were shown in Table 1.

Table 1: The basic physical parameters of composite.

Name	Ply Thickness/mm	Density/Kg·m^{-3}	Fiber volume/%
Value	0.191~1.195	1570	65

The designed CFRP laminates dimension was 200 mm (length) ×200 mm (width) ×1.91 mm (thickness), and the thickness was approximately 1.82~1.85 mm after curing.

2.2 High-pressure Microwave Curing Equipment

In order to obtain the high-pressure microwave curing platform, the microwave feeding problem under high-pressure was solved by combining the fracture antenna with the pressure partition, and the high-pressure microwave curing was realized. Figure 1 showed the high-pressure microwave curing experimental platform which was mainly composed of two parts: the pressure system and the microwave generator. The uniform microwave was generated by the microwave generator (6) through confined glass (7) and the crack antenna (8) in the resonant cavity of the closed autoclave. According to preset curing process, the CFRP laminate on the top of transparent wave shelf (17) was heated under the action of microwave and the pressure was exerted through the vacuum pressure system in the inside of autoclave as well. A temperature measuring head was embedded in the CFRP laminate, one end of the temperature transmission line (9) was connected with the temperature measuring head, and the other end was led to the data acquisition instrument (1). The power of the microwave generator was linearly adjustable within the range of 0~4000 W and the frequency of the microwave generator was 2.45 GHz. Based on temperature feedback and timely adjustment of microwave power, the curing of the CFRP laminate according to the predetermined temperature curve could be realized. There were metal cavity in autoclave and iron oxide (Fe_2O_3) as good wave absorbing materials in device interface to make sure that microwave leakage was less than 1 mW. The microwave curing equipment was built inside the traditional autoclave. The highest pressure could reach 1.5 MPa and the maximum temperature could reach 300 °C. And the preset curing process could be realized by the automatic control system of microwave power.

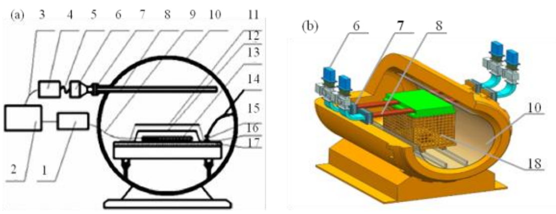

Figure 1: Schematic diagram of the structure of high-pressure microwave curing equipment (a) device module diagram (b) equipment structure diagram; (1) data acquisition instrument, (2) PLC control system, (3) connects cables, (4) microwave power control module, (5) power transmission cable , (6) microwave generator, (7) confined glass, (8) slot antenna, (9) temperature

measurement optical fiber, (10) autoclave, (11) vacuum bag, (12) air flat, (13) wave absorbing part, (14) vacuum tube, (15) quick connect, (16) sealing adhesive tape, (17) through wave shelf plate, (18) microwave cavity.

2.3 Preparation of CFRP Laminates

According to the requirements for the design size, hand layup of unidirectional composite laminates was conducted. A glass plate that could penetrate through the microwave was chosen as the mold plate. The specimens for interlaminar shear strength test were heated and cured in the microwave cavity, as shown in Figure 2. In order to study the influence of different heating rate on the ILSS of the CFRP laminate, five kinds of heating rate were chosen: 2.0, 4.0, 6.0, 8.0, 10.0 °C/min. The pressure of 0.1 MPa in the vacuum bag was kept and the pressure of 0.6 Mpa considered as high-pressure could be provided in the microwave curing cavity. Otherwise, the dwell time was 100 min at 180 °C and the cooling rate was 2 °C/min.

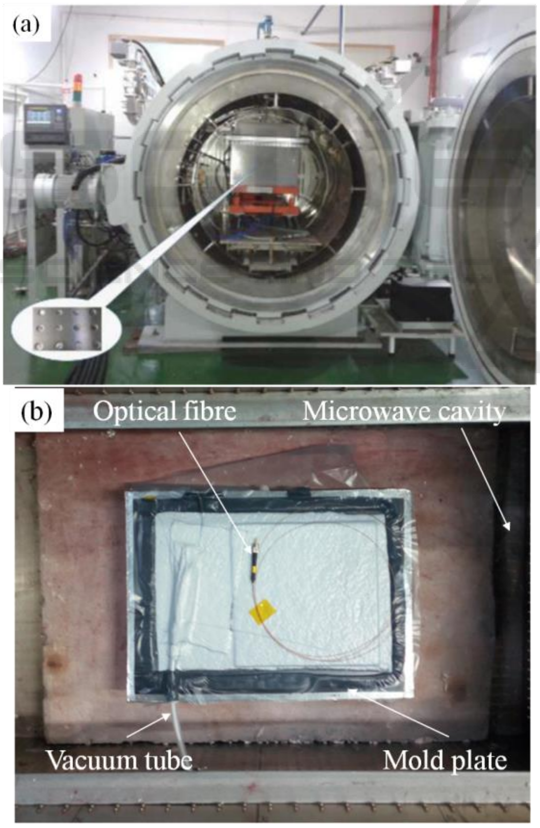

Figure 2: Photograph of (a) high-pressure microwave curing equipment and (b) the curing specimen of CFRP laminates.

2.4 Characterization Techniques

2.4.1 Void Content

The void content was observed using the optical digital microscope (ODM, model: OLYMPUS DS×500). To calculate the void content, the samples from different location of laminate were prepared and then ground and polished to get a scratch-free surface. The micrographs of samples were obtained under the optical digital microscope. The void content was calculated as an average value from at least 20 micrographs.

2.4.2 Mechanical Property Test

The ILSS of the CFRP laminate was the most important, the most common and the most concerned indicator after curing. To assess the effects of microwave on the mechanical property of the composite laminates, the inter-laminar shear strength test (ASTM JC/T 773-2010) was employed for mechanical property evaluation. These tests were carried out on the CMT5105 tensile testing apparatus (Sansi Taijie Co., Ltd., China) at room temperature. The size of the sample was 20 mm (length) ×10 mm (width) ×2 mm (thickness). Each group of parallel specimens was not less than 5. The inter-laminar shear strength τ_m (MPa) of the composite samples was determined according to Equation (1):

$$\tau_{ILSS} = \frac{3}{4} \times \frac{F}{bh} \quad (1)$$

Where F was the failure load or maximum load (N), b was the sample width (mm) and h was the specimen thickness (mm).

2.4.3 SEM Characterization

To study the bonding condition between the fiber and the resin in each specimen after interlaminar shear strength test, the SEM (Model: TESCAN MIRA3 LUM, USA) was employed to study the surface microstructure of the specimens.

3 RESULTS AND DISCUSSION

3.1 Void Content Analysis

Figure 3 shows the quantitative relationship between the heating rate and the void content of specimens in high-pressure microwave curing. It can be seen that the void content of sample increases from 0.28% to 0.89% as the value of heating rate rises from 2 °C/min up to 10 °C/min, which can illustrate that the void content of sample has been increased by 68.54%. Especially, when the heating rate is less than 6 °C/min, the value of the void content changes slightly, while the–void content increases obviously when the heating rate is more than 6 °C/min. From the concrete value of view, the value of the void content is 0.44% at the heating rate of 6 °C/min. Contrastively, the value of the void content is 0.75% at 8 °C/min, which has been increased by 41.3%. Hence, it can illustrate that the void content of CFRP laminates increases with increasing heating rate under high-pressure microwave curing.

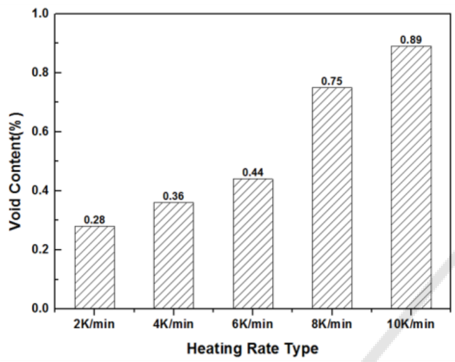

Figure 3: The void content of CFRP laminates at different heating rate.

To understand the effect mechanisam of the heating rate on void content of composites under high-pressure microwave curing, assuming that the composite pregreg is a kind of porous grid structure, which is composed together by reinforcing carbon-fibers and resin fluid with the saturation wetting viscosity, (Amico and Lekakou, 2000). Darcy's law Equation (2) can be used to describe the phenomenon:

$$V = \frac{K}{\mu} \Delta P \qquad (2)$$

where V is the velocity vector of resin flow, K is the fiber permeability, μ is the resin viscosity, ΔP is a pressure gradient that along the thickness of the resin. It can be seen that the velocity of resin flow was proportion to the pressure gradient and fiber permeability, and inversely proportion to the viscosity of resin. There are pressure gradient of 0.6 MPa in CFRP laminates under high-pressure microwave curing, which can almost ensure that the excess resin and internal gas were excluded out of the laminate and the compactness degree of fibers had been improved. However, the difference of value of void content still exists in CFRP laminates. This is probably due to the change of the resin flow in the initial stage of curing process. Firstly, a higher heating rate can result in a more uneven temperature distribution in CFRP laminates in microwave curing, which can lead to an uneven level of viscosity distribution in CFRP laminates. Hence, this phenomenon can cause a hindrance on the resin flow and resin impregnation of carbon fiber surfaces. Secondly, when the heating rate is too high under high-pressure microwave curing, there are no enough time to ensure the resin impregnating carbon fiber surfaces. Therefore, a higher heating rate bring a negative effect on interfacial adhesion of carbon fiber and resin matrix, and further result in difference in void content of sample.

3.2 Interlaminar Shear Strength Analysis

Autoclave curing is one of the main curing techenology of carbon fiber-reinforced composites. The recommended satandard autoclave curing process is that the heating rate is 1.5 °C/min, the dwell time is 150 min at 180 °C, the cooling rate is 2.0 °C/min, and the curing pressure is 0.6 MPa. The mean value of the ILSS of CFRP laminates is 98.47 MPa, as shown in Table 2.

Microwave curing can be seen a rapid curing technology, but the effect of heating rate on the ILSS of CFRP laminates under high-pressure microwave curing was not clear.

Table 2: The value of interlaminar shear strength in satandard autoclave curing.

Sample number	Test piece 1	Test piece 2	Test piece 3	Test piece 4	Test piece 5	Test piece 6	Average value
Sample result(MPa)	94.67	99.29	100.5	97.31	100.89	98.15	98.47

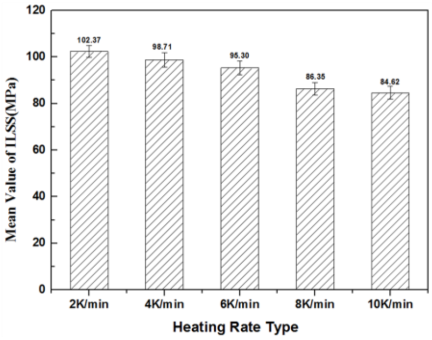

Figure 4: The mean value of ILSS of CFRP laminates at different heating rate under high-pressure microwave curing.

Figure 4 shows the mean value of the ILSS of CFRP laminates at different heating rate under high-pressure microwave curing. It can be seen that the ILSS of the CFRP decreases from 102.37 MPa to 84.62 MPa decreases by 17.34% as the value of heating rate rises from 2 °C/min up to 10 °C/min. which shows that the heating rate has a significant influence on the ILSS of CFRP laminates. What's more, the ILSS of sample is almost basically consistent with satandard autoclave curing when the value of the heating rate increases from 2 °C/min up to 6 °C/min. However, the mean value of the ILSS of CFRP laminates sharply decline when the heating rate is more than 6 °C/min. So it means that the ramp rate of microwave curing cannot be too high since the ILSS of the CFRP laminates may be influenced. In fact, when the heating rate is 6 °C/min, the ILSS of CFRP laminates is 95.30 MPa, which is slightly reduced by 3.21% compared with standard thermal curing. Contrastively, when the heating rate is 8 °C/min, the ILSS of CFRP laminates is only 86.35 MPa which is reduced by 12.3% compared with standard thermal curing. Therefore, it can be seen that the heating rate has a significant effect on the ILSS of CFRP laminates under high-pressure microwave curing. Furthermore, the value of ILSS of CFRP laminates decline and the void content of specimen rises with inrease of heating rate in high-pressure microwave curing.

To further investigate the effect mechanism of heating rate on the ILSS of the specimens, the SEM micrographs of the fracture surface of tested sample at different heating rate are shown in Figure 5. Delamination usually occurs under shear load, resulting in the formation of overlapping white patches on the surface of the fracture, which is called hackles. (Xie and Sun, 2003). The enhancement effect of the ILSS of CFRP laminates in microwave curing may be well attributed to the improvement effect on interfacial bonding of the carbon fibre–polymer matrix. But a higher heating rate has a negative effect on the ILSS of CFRP laminates.

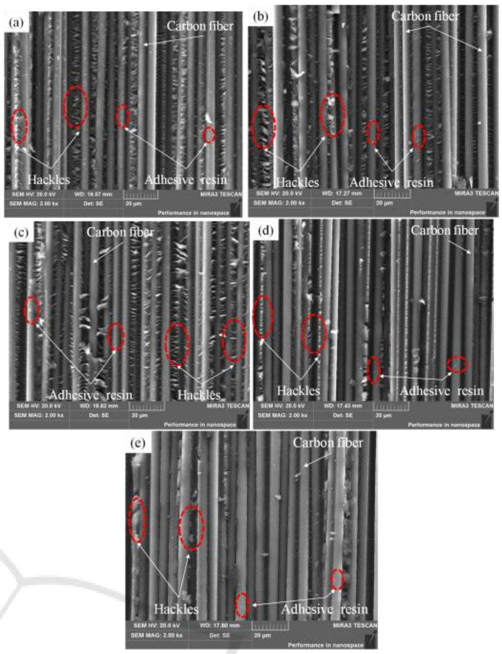

Figure 5: SEM micrographs of (a) heating rate of 2 °C/min (b) heating rate of 4 °C/min (c) heating rate of 6 °C/min (d) heating rate of 8 °C/min (e) heating rate of 10 °C/min.

Figure 5 shows the sample micrograph after interlaminar shear strength test at different heating rates. When the heating rate is low, the carbon fiber is full filled with hackles. Moreover, it can be seen that adhesive resin and hackles show a downward trend with the increase of heating rate under high-pressure microwave curing. This is because a higher heating rate cause a hindrance on the resin flow and resin impregnation of carbon fiber surface, and then make interfacial adhesion of carbon fiber and resin matrix weaken, which can explain the value of ILSS of CFRP laminates reduces with the increase of heating rate. Figure 5(a) ~ Figure5(c) show a large amount of hackles among fibers, accordingly, the value of the ILSS of CFRP laminates is relatively high. However, Figure 5(d) and Figure 5(e) show a small amount of hackles among fibers, accordingly, the value of the ILSS of CFRP laminates is low. What's more, when the heating rate is less than 6 °C/min, there is a large amount of hackles among carbon fibers. Contrastively, there is a small amount of hackles among carbon fibers when the heating rate is more than 6 °C/min. In other words, hackles distributing among adjacent fibers decrease with heating rate increases under high-pressure microwave curing, which is consistent with the results of macroscopic mechanical tests. Therefore, with increase of heating rate under high-pressure microwave curing, the resin flow and resin impregnation of carbon fiber surface were hindered in a certain extent. This phenomenon causes the void contet decrease and brings a negative effect on interfacial adhesion of carbon fiber and resin, which causes difference of the ILSS of CFRP laminates in macroscopic mechanical property and difference of hacles in microcosmic fracture surface.

It can be seen that when the heating rate is 6°C/min under high-pressure microwave curing process, the ILSS of CFRP laminates is 95.30 MPa and the void content of samples is 0.44%, whose value is basically consistent with value of sample in standard thermal curing, which can meet the need of engineering application of composites. In addition, the way of microwave curing is different with thermal curing because of selective heating of microwave heating, which can reduce energy consumption compared with thermal curing. Furthermore, the heating rate of 6 °C/min is recommended under high-pressure microwave curing, the heating time is only 25 min and the total time could be saved by 28.85%, so this heating rate can improve curing efficiency and reduce energy consumption.

4 CONCLUSIONS

According to the analysis results obtained by ODM and SEM technologies, the heating rate had a significant influence on the void content of sample and the ILSS of CFRP laminates under high-pressure microwave curing. The void content of sample increased with the increasing heating rate under high-pressure microwave curing. The ILSS of CFRP laminates tended to decrease and hackles distributing among adjacent fibers decreased with increase of the heating rate. Specially, when the heating rate was more than 6 °C/min, the ILSS of CFRP laminates decreased sharply. In the heating rate of 6 °C/min process under high-pressure microwave curing, the total time could be saved by 28.85%, and the ILSS of sample was basically consistent with the value of sample in standard thermal curing, which could be provided in reference for the later microwave curing application of composites.

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