Effect of Heating Process on Flexural Strength and Toughness of CFRTP Molded by Multi-layer Press -Investigation on Effect of Residual Stress-

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Abstract. This study investigated the effect of differences in heating process of each mold due to the molding with multi-layer press on changing in mechanical properties of CFRTP. CFRTP specimens molded with 10 pages multi-layer were prepared for 3 point bending tests and DCB tests, ENF tests in order to investigate the different of mechanical properties. The change in the mechanical properties of CFRTP molded by multi-layer press were investigated considering residual stress occurred by differences in heating process of each mold. Average of flexural strength, mode-I interlaminar fracture toughness and mode-II interlaminar fracture toughness of the specimen molded at 5th page were decreased compared with that of 1st page. Assuming the fiber as a rigid and considering the thermal shrinkage of resin, compressive residual stress should be occurred in the fiber since free shrinkage of the resin was constrained. It was considered that the reduction of flexural strength with the failure close to the compression surface was caused due to this additional compressive residual stress. The tensile residual stress also should be occurred in the resin due to constraint of free shrinkage of the resin. The observed reduction of toughness in the resin was also occurred for the procedure of crack propagation. The reduction rate of mode-I interlaminar fracture toughness was much remarkable than that of mode-II interlaminar fracture toughness. Tensile residual stress in the resin was dominant since the mode-I fracture is a crack opening mode.

1. Introduction

Carbon fiber reinforced thermoplastics (CFRTP) have been attracting attention and widely used in various fields (for example, aerospace engineering, transportation industries, sporting goods and building materials etc.) where weight saving is desired due to their excellence in specific strength and specific modulus as compared with metal materials [1-7]. In order to meet this demand, in recent years, there are strong requirements to establish molding methods that achieves both improved productivity and low cost. In this study, multi-layer press method was applied to molding CFRTP in order to greatly reduce molding time and cost by piling up some molds. Generally, mechanical properties of CFRTP are depended on heating process. In molding CFRTP molded by multi-layer press, since distance of molds from hot plate of the press machine is not constant, heating process of each mold is depended on the set positions in multi-layer. However, the effect of differences in heating process of each mold due to the
molding with multi-layer press on changing in mechanical properties of CFRTP has not been clarified. The purpose is to reveal the effect of differences in heating process of each mold due to the molding with multi-layer press on changing in mechanical properties of CFRTP.

In this study, CFRTP specimens molded with 10 pages multi-layer were prepared for 3 points bending tests and DCB tests, ENF tests in order to investigate the different of mechanical properties. The change in the mechanical properties of CFRTP molded by multi-layer press were investigated considering residual stress occurred by differences in heating process of each mold.

2. Experimental program

2.1. Materials
Prepreg sheets of unidirectional continuous carbon fiber fabricated with MXD10 (m-xylylenediamine10: melting point 215 degree Celsius, Young’s modulus 3.08GPa, shrinkage strain function 1.1%) as matrix were prepared. The prepreg sheets were supplied by Mitsubishi Engineering-Plastics Corporation, Japan. The volume fraction of carbon fibers was approximately 40% in the prepreg sheets.

2.2. Molding method of multi-layer press
Figure 1 shows the schematic views of multi-layer press. Each page was defined as the unit of molding of multi-layer pressing. The number of pages was 10 for multi-layer press. In this study, each page was called with sequence number in order from the top one with "p" text, so that top and bottom were called (p.1 mold) and (p.10 mold), respectively, as shown in the figure. Prepreg sheets were cut into 750 x 750mm and set into each mold. Figure 2 shows examples of heating process of the 1st, 3rd, 5th and 10th pages during multi-layer pressing. The temperature was elevated at 266 degree Celsius keeping 0.1MPa of pressure. After reaching to the maximum temperature, the temperature was maintained for 60 minutes. After starting cooling, the pressure was increased to 0.5 MPa.

Figure 1. Schematic views of multi-layer press.

Figure 2. Illustration of molding process diagram.
2.3. Mechanical testing methods

2.3.1. Quasi-static 3 point bending tests
Flexural strength of CFRTP molded by multi-layer press was measured by quasi-static 3 point bending tests according to ASTM D7264 at 5 mm/min (cross head speed) by using a universal material testing machine. For this test, stacking sequence of the specimens was [0°/45°/90°/-45°], of quasi-isotropic 8 layers. Dimensions of the specimens were 60 (length) x 15 (width) x 1.2 (thickness) mm. Test data were obtained with at least 5 samples. The states of cross section of specimens after 3 point bending tests were observed by digital microscope.

2.3.2. Double cantilever beam tests
Mode-I interlaminar fracture toughness of CFRTP molded by multi-layer press was measured by double cantilever beam (DCB) tests according to ASTM D5528 at 1 mm/min (cross head speed) by using a universal material testing machine. Figure 3-(a) shows schematic view for DCB tests. For this test, stacking sequence of the specimens was [(0°/90°)_n/0°], of orthotropic 26 layers. Aluminum foil with approximately 15μm thickness was previously inserted between 13th and 14th ply of the laminate. Dimensions of the specimens were 250 (length) x 25 (width) x 4.0 (thickness) mm. The length of pre-crack was 50 mm. Test data were obtained with at least 5 samples. Mode-I interlaminar fracture toughness was calculated by following equation (1) and (2) [8].

\[
G_{IC} = \frac{3}{2(2H)} \left( \frac{P_c}{B} \right)^2 \frac{C}{a} \quad (1)
\]

\[
a = a_1 (BC)^{1/3} + a_0 \quad (2)
\]

where \(G_{IC}\), \(a\), \(2H\), \(B\), \(P_c\), \(C\), \(a_1\) and \(a_0\) denote mode-I interlaminar fracture toughness, crack length, specimen thickness, specimen width, initial critical load, loading point compliance of the initial elastic part, the slope of the approximate straight line of plotting \(a/2H\) (the dimensionless crack length) on the vertical axis and \((BC)^{1/3}\) (the cubing root of the crack opening displacement compliance per unit width) on the horizontal axis and the intercept of the above approximate straight line, respectively.

2.3.3. End notched flexure tests
Mode-II interlaminar fracture toughness of CFRTP molded by multi-layer press was measured by end notched flexure (ENF) tests according to ASTM D7905 at 0.5 mm/min (cross head speed) by using a universal material testing machine. Figure 3-(b) shows schematic view for ENF tests. For this test, stacking sequence of the specimens was [(0°/90°)_n/0°], of orthotropic 26 layers. Aluminum foil was also previously inserted between 13th and 14th ply of the laminate. Test data were obtained with at least 5 samples. Dimensions of the specimens were 250 (length) x 25 (width) x 4.0 (thickness) mm. The length of pre-crack was 50 mm. Mode-II interlaminar fracture toughness was calculated by following equation (3) and (4) [9, 10].

\[
G_{IIc} = \frac{qa_1^2 C_1}{2B(2L^3+3a_1^3)} \quad (3)
\]

\[
a_1 = \left( \frac{C_1}{C_0} a_0^3 + \frac{2}{3} \left( \frac{C_1}{C_0} - 1 \right) L \right)^{\frac{1}{3}} \quad (4)
\]

where \(G_{IIc}\), \(a_0\), \(P_c\), \(C_0\), \(C_1\), \(a_1\), \(L\) and \(B\) denote mode-II interlaminar fracture toughness, initial crack length, initial critical load, loading point compliance of the initial elastic part, loading point compliance at initial critical load, estimated crack growth length at initial critical load, supports span and specimen width, respectively.
2.4. Estimation of residual stress in carbon fiber
Compressive residual stress should be occurred in the fiber due to the thermal shrinkage of resin was calculated considering linear expansion coefficient of resin. Compressive residual stress was calculated by following Equation (5)

$$\sigma_R = \alpha \Delta t E (1 - V_f)$$  \hspace{1cm} (5)

where $\sigma_R$, $\alpha$, $\Delta t$, $E$ and $V_f$ denote compressive residual stress should be occurred in the fiber, linear expansion coefficient of resin, temperature difference, Young’s modulus of resin and fiber volume fraction, respectively.

3. Results and discussions

3.1. Flexural strength
Figure 4 shows differences of the flexural strengths of the specimens molded at 1st and 5th page. The states of cross sections of specimens after 3 point bending tests are shown in figure 5. Average of flexural strength of the specimen molded at 5th page was decreased approximately 10% compared with that of 1st page. Specimens on each page were failed between layers close to the compression surface and finally broken at the tensile part.

3.2. Mode-I interlaminar fracture toughness
Figure 6 shows differences of the mode-I interlaminar fracture toughness of the specimens molded at 1st and 5th page. Average of mode-I interlaminar fracture toughness of the specimens molded at 5th page was decreased approximately 20% compared with that of 1st page.

3.3. Mode-II interlaminar fracture toughness
Figure 7 shows differences of the mode-II interlaminar fracture toughneces of the specimens molded at 1st and 5th page. Average of mode-II interlaminar fracture toughness of the specimens molded at 5th page was decreased approximately 13% compared with that of 1st page.
3.4. Effect of residual stress

The temperature in the mold of the 1st page was lower than the melting temperature of the resin, while it was higher than that in the 5th page, during cooling process after molding pressure was respectively reached to 0.5MPa. Figure 8 shows illustrations of the models of residual stress. Assuming the fiber as a rigid and considering the thermal shrinkage of resin, compressive residual stress should be occurred in the fiber since free shrinkage of the resin was constrained. Approximate 20 [MPa] of compressive residual stress of the fiber was estimated according to the differences in heating process, where linear expansion coefficient of resin was assumed to be 55 \(10^{-6}/\text{degree Celsius}\). It was considered that the reduction of flexural strength with the failure close to the compression surface was caused due to this additional compressive residual stress.

![Figure 8. Models of residual stress occurred in fiber.](image)

4. Conclusion

In this study, the effects of heating process on the change of flexural strength and interlaminar fracture toughness of CFRTP molded by multi-layer press in terms of residual stress was discussed. The following conclusions were given.

1. Average of flexural strength, mode-I interlaminar fracture toughness and mode-II interlaminar fracture toughness of specimens molded at 5th page were decreased compared with those of 1st page.
2. Approximate 20 [MPa] of compressive residual stress of the fiber was estimated according to the differences in heating process. It was considered that the reduction of flexural strength with the failure close to the compression surface was caused due to this additional compressive residual stress.
(3) The tensile residual stress also should be occurred in the resin due to constraint of free shrinkage of the resin. The observed reduction of the toughness in the resin was also occurred for the procedure of crack propagation. The reduction rate of mode-I interlaminar fracture toughness was much remarkable than that of mode-II interlaminar fracture toughness. Tensile residual stress in the resin was dominant since the mode-I fracture is a crack opening mode.

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