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Type of the Paper (Article)

Mechanical properties of thermoplastic composites made of commingled carbon fiber / nylon fiber

Mizuki Ono [1], **Masachika Yamane [2], Shuichi Tanoue[1,3], Hideyuki Uematsu[1,3] and Yoshihiro Yamashita [3,*]**

[1] Frontier Fiber Technology and Science, University of Fukui; mizuki_55@outlook.jp
[2] Headquarters for Innovative Society-Academia Cooperation, University of Fukui; myamane@u-fukui.ac.jp
[3] Research Center for Fibers and Materials, University of Fukui; yama-yo@u-fukui.ac.jp
* Correspondence: yama-yo@u-fukui.ac.jp; Tel.: +81-50-3633-8582

Abstract: Fiber-opening treatment of commingled yarns consisting of thermoplastic nylon fibers and carbon fibers could produce superior CFRTP, but few studies toward that end have been conducted. In this study, we investigated whether an open weave fabric consisting of commingled yarns made of carbon and nylon fibers could shorten the impregnation distance of resin to carbon fibers, and there are few reports on the design of fabrics by opening carbon fiber bundles consisting of commingled yarns. From this study, following are cleared. The impregnation speed of the nylon resin on the carbon fiber was very fast, less than 1 minute. As the molding time increased, the tensile strength and tensile fracture strain slightly decreased and the nylon resin deteriorated. The effects of molding time on flexural strength, flexural modulus, and flexural fracture strain were negligible. From the cross-sectional observation conducted to confirm the impregnation state of the matrix resin, no voids were observed in the molded products regardless of molding time or molding pressure, indicating that resin impregnation into the carbon fiber bundle of the open-fiber mixed yarn fabric was completed at a molding pressure of 5 MPa and a molding time of 5 min.

Keywords: commingle yarn; carbon fiber; opening yarn fabric; nylon fiber; composite

1. Introduction

With the introduction of global CO_2 regulations and rising fuel prices, the need for lighter cars has grown dramatically. Although there are options for reducing the weight of automobiles, such as the use of high-tensile steel plates and large quantities of aluminum alloy, the application of carbon fiber reinforced plastics (CFRP), which have excellent specific and modulus resistance, to structural elements is considered the most efficient one. CFRP is a composite material that uses plastic as a base material and carbon fiber as a reinforcement material. It should be used in a wide range of fields due to its characteristics of high strength, high elastic modulus, and light weight (low density). Depending on the matrixed resin used as the basic material, CFRP can be divided into two types: thermoset CFRP, which uses epoxy or unsaturated polyester, and thermoplastic CFRP, which uses polypropylene or nylon. Thermosetting CFRP is a flexible material prior to hardening due to the nature of the thermosetting resin, making it easy for the resin to impregnate the carbon fiber bundle and relatively easy to fabricate the prepreg sheet, which is the intermediate material for CFRP. However, thermosetting resins need more curing time because they undergo crosslinking reactions by chemical reactions. Therefore, if it is possible to give thermoplastic CFRP excellent properties such as high strength and a high elastic modulus comparable to those of thermoset CFRP, it will be expected to be a material with high productivity and excellent recyclability.

On the other hand, thermoplastics do not require a thermosetting process and can reversibly repeat the solid state at room temperature and the molten state at high temperature without any chemical reaction, which means that molding can be carried out in a very short time, thus requiring only the cooling of the material and leading to a short molding cycle. In addition, it can be remolded by reheating and has excellent recyclability. Therefore, if it is possible to give thermoplastic CFRP excellent properties such as high strength and a high elastic modulus comparable to those of thermoset CFRP, it will be expected to serve as a material with high productivity and excellent recyclability.

Demand for continuous fiber-reinforced thermoplastic composites, which have continuous fibers, is expected to increase significantly because the strength of the reinforcing fibers can be maximized for use in the structural materials of automobiles and aircraft. However, the melting viscosity of the thermoplastic resin is extremely high compared to that of the pre-hardening thermosetting resin. Therefore, the challenge is how to impregnate the reinforcing fiber bundle with thermoplastic resin in a short time. Preimpregnated thermoplastic carbon fiber is difficult to adapt to molded products with complex shapes unless the viscosity of the resin is reduced by preheating. The relationship between the resin/fiber distance and the resin penetration time was compared between the analytical model using the Darcy rule and the experimental results [1-6]. Several molding methods have been proposed for the manufacture of pre-preg thermoplastic resin and carbon fiber, including the powder method [7-9], the commingled fiber method [10-16], and the film stacking method[17,18], and the relationships between these molding methods and the degree of impregnation have been reported. The results show that the thermoplastic prepreg with the best impregnation properties is the one made of commingled yarn, which has the shortest distance between carbon and thermoplastic fibers. Consequently, it is necessary to establish a molding technology to produce CFRTP using commingled yarn.

On the other hand, as an approach to fully demonstrate the strength of carbon fiber, one study has shortened the impregnation distance in the film stacking method by applying "open continuously reinforcing fiber tow," a technique to spread carbon fiber bundles thinly and widely [19]. It is also known that the "thin layer effect," which improves the mechanical properties of the composite material by reducing the thickness of the prepreg to an extremely low level, can also be obtained [20,21]. It is expected that a further fiber-opening treatment of commingled yarns consisting of thermoplastic and carbon fibers could produce superior CFRTP, but few studies toward that end have been conducted. In this study, we fabricated open-fiber blended yarns with an open-fiber treatment, wove fabrics using these yarns, and investigated the effects of forming time and forming pressure in press forming on the mechanical properties of the molded products.

2. Materials and Methods

Material

Commingled yarn consisting of PAN-based carbon fiber (CF) and low-water-absorbing nylon resin fiber was used as the material for the woven thermoplastic CFRP laminate. Table 1 shows the specifications of the commingled yarn. First, the yarns were opened to a width of 30 mm, and then the yarns were coated with 15wt% copolymerized polyamide powder to seal the opening commingled yarns. These were then woven with a rapier loom. Table 2 shows the properties of the opening commingled yarn fabrics. Figure 1 shows the procedure for opening and bonding commingled yarns. Figure 2 shows a photograph of the opening plain fabric using commingled yarn. The woven opening commingled yarn fabric was cut into 245 mm squares, and these fabrics were point-bonded to each other using a soldering iron with a temperature control function (RX-802AS manufactured by Taiyo Denki Sangyo), then laminated one by one (Figure 3). The temperature of the soldering iron was set to 270 ℃. The number of layers was set to 30 so that the thickness of the molded product would be approximately 2 mm. Table 3 shows the properties of the laminated fabrics made from opening commingled yarn.

Table 1. Specifications of commingled yarn.

Matrix resin	Low water-absorbing nylon MXD10 (LEXTER manufactured by Mitsubishi Gas Chemical Co. Ltd.) 1,532 filaments (3.125 d mono-filament)
Carbon fiber	PAN CF (Mitsubishi Gas Chemical Co. Ltd. TR50S12L) 12,000 filaments
Target value of fiber volume content (V_f)	50 %

Table 2. Specifications of woven fabric using commingled yarn.

Weave structure	Plain
basis weight	107.5 g/m²
Thickness	244.6 μm
Fiber volume content (V_f)	41.5 %

Table 3. Specifications of laminated materials.

Length	Width	Layer number
245 mm	245 mm	30 ply

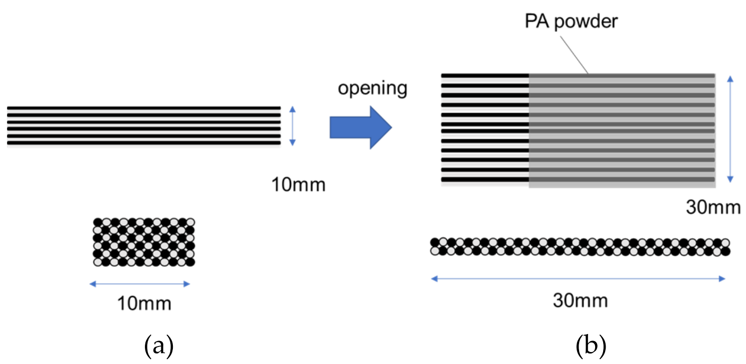


Figure 1. Image of opening of commingled yarn. (a)Standard commingled yarn; (b)Opening commingled yarn.

Figure 2. Woven fabric using opening commingled yarn.

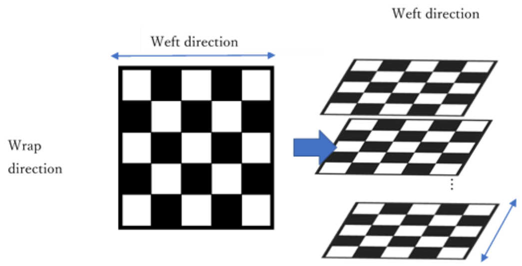
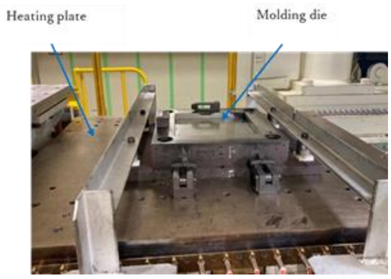


Figure 3. Laminated configuration of woven fabric using opening

Press molding

A heating and cooling press molding machine (Hitachi Zosen Fukui Corporation UFHS2500) was used as a press molding machine. Figure 4 shows a photograph of the machine. The heating plate and the cooling plate are lined up next to each other, and a die lifter is installed in front of and behind the hot plate. As a result, heated molds can be transported on a cooling plate using a mold lift for quick cooling. For the molding, a male−female mating type was used. The mold area dimensions were 250 mm square.

(**a**)



(**b**)

Figure 4. (**a**) Press molding machine; (**b**) □250 mm press molding die attached to heating plate.

Press forming conditions

The press forming process used in this experiment is described below.

(1) The opening commingled yarn fabric was laminated in advance using any number of layers and any lamination configuration, then dried in a vacuum dryer for at least 15 hours at 80℃.

(2) After it was confirmed that the mold had reached the desired molding temperature, the laminated opening commingled yarn fabric was removed from the vacuum dryer and fed into the mold.

(3) To melt the nylon fibers, a warm-up was performed under a pressure of 1 MPa for 5 minutes.

(4) The mold was maintained at a predetermined pressure (mold pressure) and for a predetermined time (mold time).

(5) After the heat molding was completed, the mold was unclamped, lifted off the hot plate by the mold lifter, transferred from the heating plate to the cooling plate at room temperature, and cooled again by applying the desired pressure (molding pressure).

(6) After the mold temperature was confirmed to be below 80℃, the mold was opened by the press machine and the molded product was removed.

To study the effects of formation pressure on the physical properties of molded products, molding was carried out at two pressure levels: 5 MPa and 10 MPa. The molding temperature was set to 270℃ and the molding time was set to four levels: 5, 10, 20, and 30 min (Table 4). Figure 5 shows an example of the temperature and pressure history during the molding, where the molding pressure is 5 MPa and the molding time is 30 minutes.

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Table 4. Press molding conditions

Molding temperature	Preheating	Molding time	Molding pressure
270 ℃	1 MPa, 5 min.	5 min., 10 min. 20 min., 30 min.	5 MPa, 10MPa(30min)

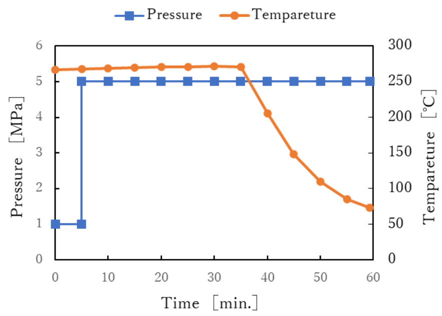

Figure 5. Profile of molding pressure and molding temperature.

Tensile test and four-point bend test

The tensile test was carried out as described in JIS K 7164. A CCD camera was used to measure the increase in the distance between the sample reference lines during the test. A four-point bending test was then performed according to JIS K 7074. Bending deflection was measured with a bending deflectometer placed directly under the center of the test specimen. Tables 5 and 6 present the test conditions of the tensile test, and the four-point bending test, respectively. For both tests, the fabric was cut out so that the warp direction of the fabric was in the longitudinal direction of the specimen. A precision universal testing machine (Shimadzu AG-IS100kN) was used for the tensile test. Figure 6 shows a photograph of the four-point bending test jig.

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Table 5. Conditions of tensile test.

Test specimen longitudinal direction		Warp direction
Test specimen dimensions	length	240 mm
	width	25 mm
	thickness	2+0.4 mm
distance between two lines of sight		50 mm
Distance between tabs		150 mm
Tab Length		45 mm
Tensile test speed		2 mm/min.
Sample number		5

Table 6. Conditions of four-point flexural test.

Test specimen longitudinal direction		Warp direction
Test specimen dimensions	length	100 mm
	width	15 mm
	thickness	2±0.4 mm
Distance between fulcrums		88 mm
Distance between indenters		29 mm
fulcrums radius		3 mm
indenters radius		3 mm
Bending speed		5 mm/min.
Sample number		6

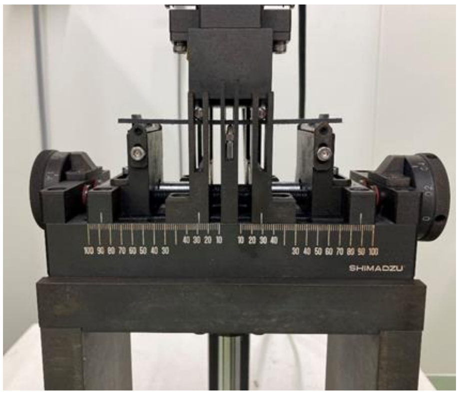

Figure 6. Four-point flexural test jig.

Measurement of fiber volume content

To measure the volume content of CF fiber V_f, the combustion method was adopted and the matrix resin was completely burned in an electric furnace at a combustion temperature of 400 ℃ for more than 15 hours. The densities of the carbon fiber and nylon were ρ_f=1.82×10³ kg/m³ and ρ_m=1.11×10³ kg/m³, respectively.

Confirmation of resin impregnation to the interior of the carbon fiber

To observe the condition of the impregnated resin in the carbon fiber bundle, a cross section of the molded product was observed. The center of the flat molded plate was cut into a 15 mm square using a composite cutting machine, and the cut sample was

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encapsulated in unsaturated polyester resin. The cross section with a mirror finish was observed using a microscope (Keyence VHX-2000).

3. Results

Fiber volume content

The fiber volume content V_f of each molded product measured by the combustion method is shown in Table 7. The measured results of V_f were 38.3%~38.9% for the products molded at 5 MPa for 5 min. On the other hand, V_f 48.7% of the molded products were formed at 10 MPa. The value of V_f for each molded product formed at 5 MPa reflects the design V_f of 41.5% for the opening commingled yarn fabric shown in Table 2 and is almost the same regardless of the forming time. On the contrary, the value of V_f for the molded product formed at a molding pressure of 10 MPa was 48.7%, which was much larger than the design V_f value. This is probably because there is more leakage during molding at the higher molding pressure of 10 MPa.

Table 7. Test condition of four-point flexural test.

Molding time	5 min.	10 min.	20 min.	30 min.	
Molding time	5 MPa				10 MPa
Fiber volume content (V_f)	38.9 %	38.7 %	38.3 %	38.6 %	48.7 %

Tensile test

The stress–strain (S–S) curve obtained from the tensile test is shown in Fig. 7. Each sample shows a test result close to the average among the five tests. It can be seen that the S–S curve of the product molded at 5 MPa is linear up to the maximum tensile stress regardless of the molding time. On the other hand, the S–S curve of the product molded at 10 MPa is lower. Figure 8 shows the relationships among tensile strength, tensile strain, tensile modulus, and molding time. The tensile strength and breaking strain decreased slightly with increasing molding time, but the elastic modulus did not change. The longer the molding time, the lower the elongation at break and consequently the lower the breaking stress. This may be due to thermal degradation of the nylon matrix. Figure 9 shows the results of varying the molding pressure while the molding time was fixed at 30 minutes. As shown in Table 7, the higher the molding pressure, the more the nylon matrix resin is pushed out of the molded product and the volume fraction of fibers increases, which is thought to increase the rupture strength and elongation at break. Moreover, when the molding pressure is high, the force that pushes the nylon matrix resin out of the molded product disturbs the arrangement of carbon fibers, and the number of carbon fibers that are not straight in the tensile direction increases, which is thought to reduce the modulus.

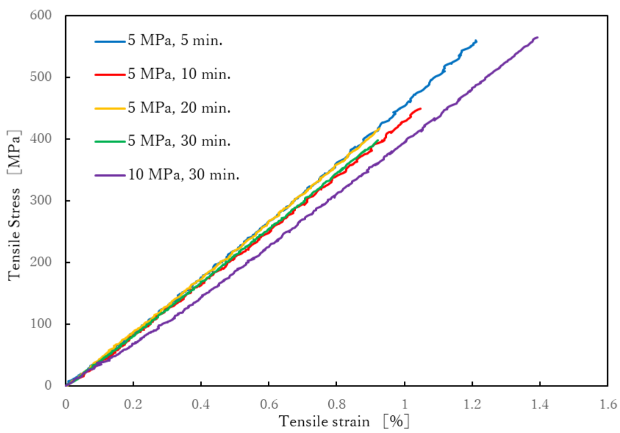
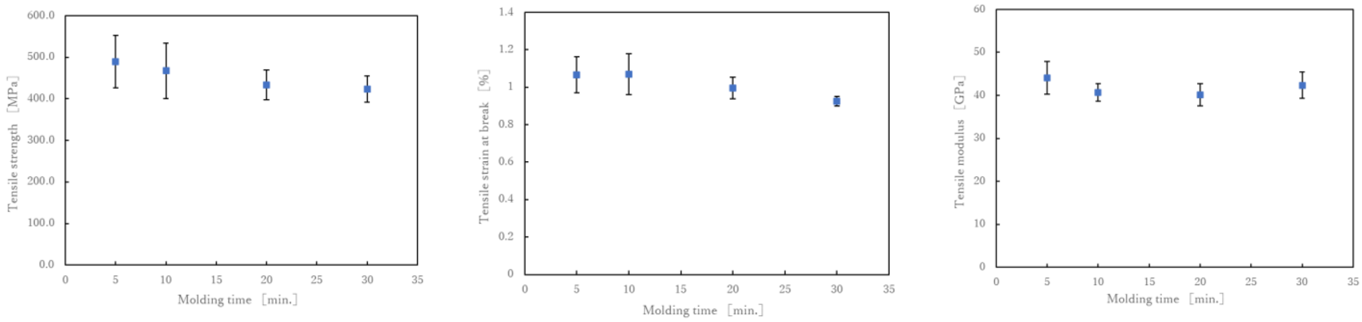
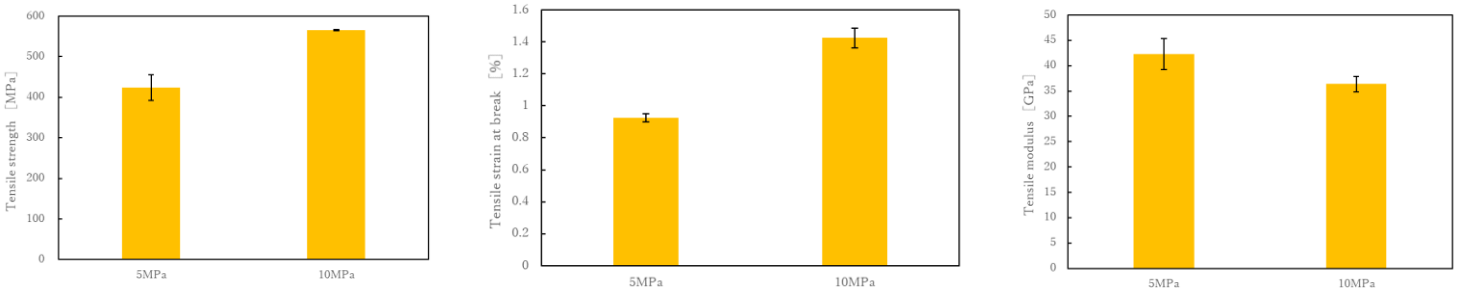


Figure 7. Stress-strain curves of the tensile test.



(**a**) (**b**) (**c**)

Figure 8. (a) Effect of molding time on tensile strength under molding pressure of 5 MPa; (**b**) Effect of molding time on tensile strain under molding pressure of 5 MPa; (c) Effect of molding time on tensile modulus under molding pressure of 5 MPa.



(**a**) (**b**) (**c**)

Figure 9. (a) Effect of molding pressure on tensile strength under molding time of 30 minutes; (**b**) Effect of molding time on tensile strain at break under molding time of 30 minutes; (c) Effect of molding pressure on tensile modulus under molding time of 30 minutes.

Four-point bending test

Figure 10 shows the S–S curve obtained from the four-point bending test. Each sample shows the most average results of the six samples tested. It can be seen that the S–S curve is linear up to the maximum breaking bending stress under any of the molding conditions. Figure 11 shows the relationship between flexural strength, flexural modulus, flexural strain, and molding time. The results showed that the molding time had no effect on the bending strength, bending modulus, or bending strain. Kawabe et al. [19] applied a bending test to a molded body with a thickness of 1 mm, consisting of 19 layers each made of alternating 12K carbon fiber unidirectional sheets opened to a width of 5 mm and nylon 6 film with a thickness of 25 μm. As a result, the bending strength increased as the molding time increased, but the increase after 5 minutes was slight. The tensile properties in Figure 6 decreased with increasing molding time, but the bending properties in Figure 9 were not affected by molding time. Considering that the impregnation of the nylon film on the opened 12K carbon fiber bundle is almost complete in 5 minutes [19], the resin impregnation in the commingled yarn consisting of nylon and 12K carbon fiber in this study was sufficiently complete in less than 5 minutes. In the flexural test, we did not observe any decrease in strength due to nylon resin degradation over the long molding time observed in the tensile test. This is presumably because an opened weave fabric was used. All fractures in the flexural tests were on the tensile side, and the composite failed without interlaminar shear failure or buckling failure. This may be because the strength retention effect of the weft yarn due to the woven structure is greater than the degradation of the resin due to the long molding time, and the effect could not be observed within the error range. On the other hand, the higher the molding pressure, the slightly better the bending properties became, but these were within the margin of error (Figure 12).

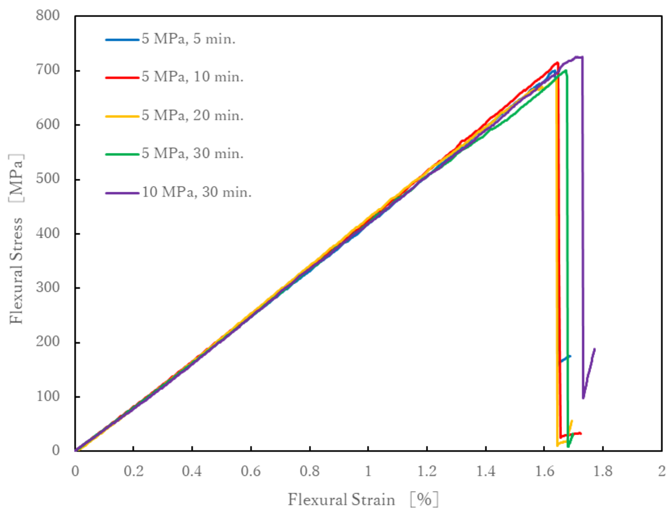
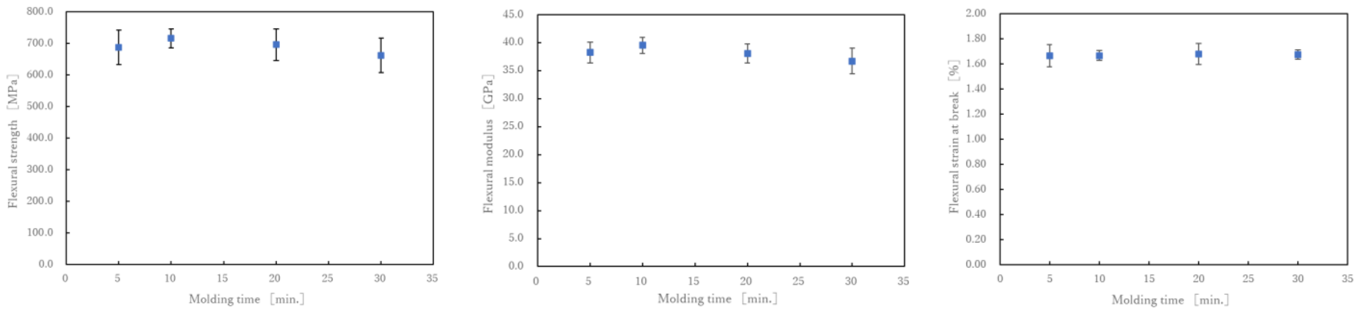
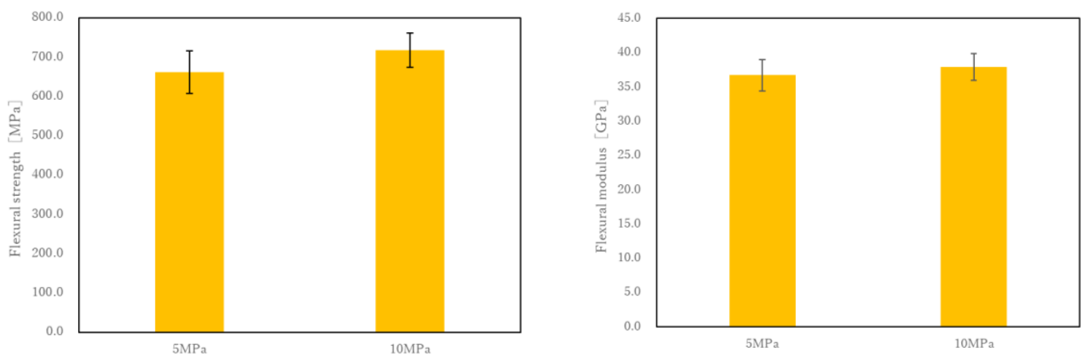


Figure 10. Stress-strain curves of the four-point flexural test.



(**a**) (**b**) (**c**)

Figure 11. (a) Effect of molding time on flexural strength under molding pressure of 5 MPa.; (**b**) Effect of molding time on flexural modulus under molding pressure of 5 MPa; (c) Effect of molding time on flexural strain at break under molding pressure of 5 MPa.



(**a**) (**b**)

Figure 12. (a) Effect of molding pressure on flexural strength under molding time of 30minutes; (b) Effect of molding pressure on flexural modulus under molding time of 30 minutes.

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4. Discussion

Figures 13 and 14 show cross-sectional images of the products molded at 5 MPa and 10 MPa, respectively. The white horizontal stretch in each image shows the weft yarn of the carbon fiber, the white dot shows the warp yarn of the carbon fiber, and the gray area shows the matrix resin, Nylon-MXD10. The matrix resin was sufficiently impregnated into the carbon fiber bundle, and no voids were observed. Figure 15 shows the effects of molding temperature and pressure on the thickness of the commingled yarn after molding. Even with a molding time of 5 minutes, the nylon fibers are completely melted whereas the carbon fiber bundle is still somewhat swollen. Therefore, 10 minutes is the optimum molding time even for an opened fabric.

The impregnation rates of resin into the carbon fiber of the carbon fabric / film laminate shown in Figure 16(a) and of the commingled yarn fabric shown in Figure 16(b) were investigated. Ueda et al. [3] used the Darcy rule to evaluate the impregnation rate of resin on carbon fiber. Using Ueda et al.'s formula of Equation (1)[3] and calculating the distance between the commingled nylon and carbon fibers l0 as 10 μm, we found that the resin was completely impregnated into carbon fibers in 20 seconds at a molding pressure of 1 MPa preheating (Figure 15). This is example 1 of an equation:

$$I = \left(\frac{2k(P_m - P_0)}{\eta l_0^2}\right)^{\frac{1}{2}} \times t^{\frac{1}{2}} \tag{1}$$

t: molding time, η: resin viscosity (120 Pa-s), l_0: distance from the resin flow front to the center of the fiber bundle (10 μm commingled yarn, 100 μm film), P_m: press pressure (1~10 MPa), P_0: atmospheric pressure (0.101 MPa), *k*: constant (1.83×10^{-14}).On the other hand, if the distance l0 is set to 100 μm, assuming the lamination of the opened carbon fiber and resin film, it took 3 minutes and 20 seconds for the resin to completely impregnate the carbon fiber under a molding pressure of 10 MPa (Figure 15).

Motochika et al.[22] studied the resin impregnation rate of just the commingled carbon fiber yarn and nylon fiber yarn used in this study. They found that the resin impregnation rate after 1 minute was 98.5% (not impregnated rate: 1.5%). It can be deduced that the formation of an opening weaving fabric made of commingled yarns can be completed in no time.

In terms of the impregnation rate of the resin in the carbon fiber, the rate is assumed to be the same whether the fabric of commingled yarns is manufactured from opening yarns or not. On the other hand, as shown in Fig. 16, in the case of CFRTP, which is not a commingled yarn but a carbon fiber bundle with resin layers above and below, it is useful to reduce the thickness of the fiber bundle and opening the fiber bundle in order to increase the rate of thermoplastic resin penetration into the carbon fiber. The woven sheet in Fig. 16(a) has great rigidity because the resin is in the shape of film. However, fabrics made of commingled yarns are flexible. In addition, the flexibility is more pronounced for opening the fabrics. The woven fabric made by opening carbon fiber bundles has the merit of maximizing the original strength and modulus of elasticity of carbon fibers because it can reduce the curvature of fibers at the intersection of warp and weft yarns.

Takahashi et al. [23] compared the transverse tensile strengths of carbon fiber and PEEK commingled yarn fabrics, carbon fiber yarn and PEEK yarn fabrics, and even carbon fiber-impregnated PEEK prepreg. The results showed that the order of elastic moduli was CF/PEEK prepreg > CF/epoxy prepreg > CF/PEEK commingle > CF/PEEK fabric > PEEK resin. The rupture stress was CF/PEEK prepreg > CF/PEEK commingle > CF/epoxy prepreg > PEEK resin > CF/PEEK fabric.

In the present study, CF/Nylon commingled yarn was used and was woven with opening fiber. In the future, it will be necessary to develop a loom suitable for fiber opening and to improve the commingled yarn manufacturing method so that we can compare the results with those of woven fabrics of commingled yarn without fiber opening, the effect of sagging of carbon fiber by fiber opening, and the optimum width of fiber opening.

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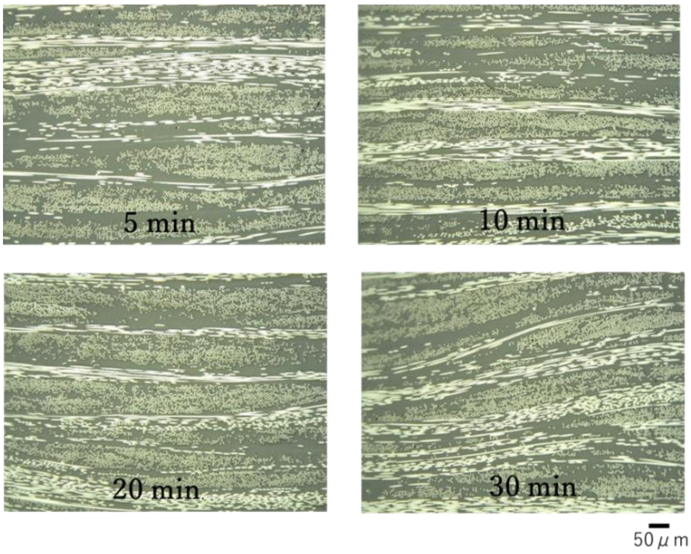


Figure 13. Cross-sectional photographs of composite materials made at different molding times at a molding pressure of 5 MPa.

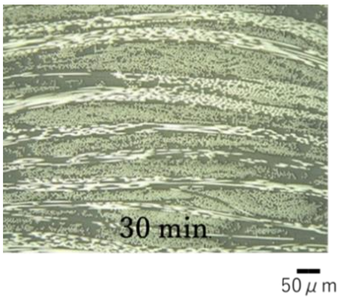


Figure 14. Cross-sectional photographs of composite materials made at different molding times at a molding pressure of 10 MPa.

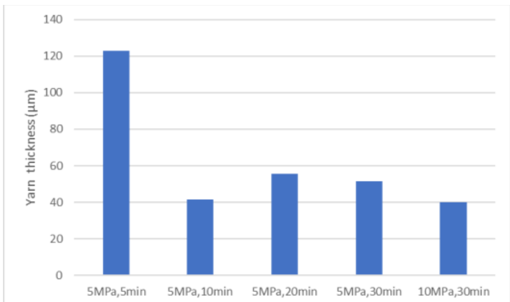
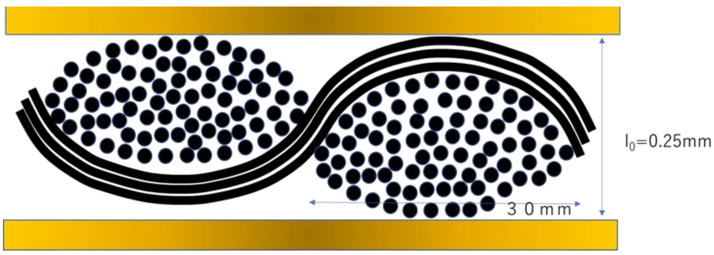


Figure 15. Effects of molding temperature and pressure on the thickness of the commingled yarn after molding.



(a)

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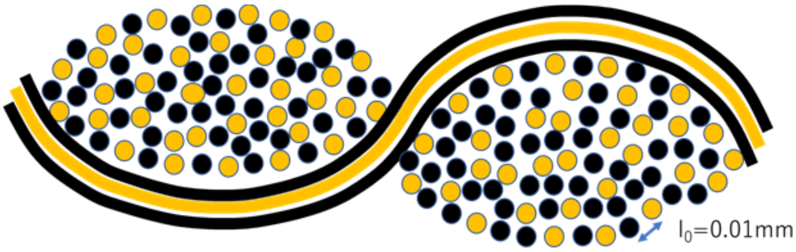

(b)

Figure 16. (a)Molding of composite materials from carbon fiber fabric and thermoplastic film; (b) Molding of composite materials from commingled yarns.

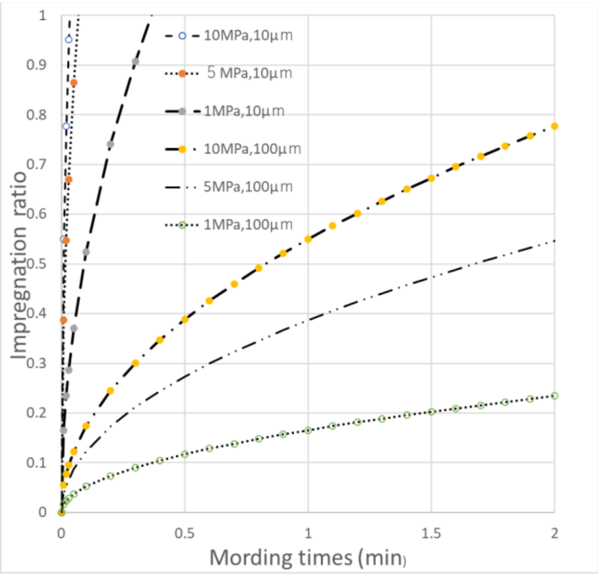

Figure 17. Impregnation ratio as a function of molding pressure and l_0.

5. Conclusions

We investigated whether an open weave fabric consisting of commingled yarns made of carbon and nylon fibers could shorten the impregnation distance of resin to carbon fibers and thus shorten the molding time in CFRTP molding. Woven fabrics made of nylon fiber and carbon fiber commingled yarns are easy to handle, and the decrease in the elastic modulus of carbon fiber due to crimping, which is characteristic of woven fabrics, can be minimized. There are few reports on the design of fabrics by opening carbon fiber bundles consisting of commingled yarns.

(1) The impregnation speed of the nylon resin on the carbon fiber was very fast, less than 1 minute. As the molding time increased, the tensile strength and tensile fracture strain slightly decreased and the nylon resin deteriorated. The tensile strength and tensile strain at break increased with increasing molding pressure due to the increased outflow of matrix resin and the increased fiber volume content in the molded product.

(2) The effects of molding time on flexural strength, flexural modulus, and flexural fracture strain were negligible.

(3) As a result of the cross-sectional observation conducted to confirm the impregnation state of the matrix resin, no voids were observed in the molded products regardless of molding time or molding pressure, indicating that resin impregnation into the carbon fiber bundle of the open-fiber mixed yarn fabric was completed at a molding pressure of 5 MPa and a molding time of 5 min.

(4) The combination of commingled yarn, open fiber, and woven fabric suggested the possibility of a flexible, easy-to-handle thermoplastic CFRTP prepreg.

Author Contributions: Data analysis, M.Ono.; Experimental and investigation, M.Ono.; Research method, M.Yamane.; Experimental advice, M.Yamane.; Writing—original draft preparation, H.Uematsu.; writing—review and editing, M.Yamane.; Project administration, S.Tanoue.; Funding acquisition, Y.Yamashita. All authors have read and agreed to the published version of the manuscript.

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