INTERFACIAL EFFECT ON THE MECHANICAL PROPERTIES OF GLASS/PHENOLIC COMPOSITE

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SUMMARY: Interfacial effect on the mechanical properties of glass fiber/phenolic composites was discussed in this paper. Standard and silane modified resins were used as matrix, and a yarn and two kinds of rovings with different sizing were used as reinforcement. Effect of fiber on wetting were evaluated by Wilhelmy type dynamic wetting instrument, and it was clarified that sizing specially developed for phenolic resin was quite effective to improve resin impregnation into the fiber bundle. The mechanical properties were evaluated by using the resin impregnated unidirectional fiber bundle composite specimens. The effects of resin and fiber on strength and crack propagation were evaluated by the lateral compression test. Type of the fiber affected the dispersion of fibers in the matrix resin, and the roving developed for phenolic resin gave the well dispersion of the fiber. This led the high mechanical properties and high resistance to the crack initiation.

KEYWORDS: interface, phenolic resin, sizing, yarn, roving, fracture, silane modification, compression strength

INTRODUCTION

In recent years fiber reinforced polymer composites have been applied to the structural components in the transportation industries such as for automobiles, trains, ships, etc. In most of these applications, incombustibility is one of the most important required properties for the material. Phenolic resins can sustain temperatures of up to 200°C with very low toxicity and low smoke emission [1]. Moreover, they are considered fire-restricting materials, as they can fulfil function in an established fire (in contrast with many metals, even steel). Therefore phenolic resins are suitable as matrix resin for composites in transportation applications. However, phenolic resin produces H_2O during reaction process in curing and the nature of phenolic resins is brittle. These undesirable natures lead poor mechanical properties of phenolic composites, and therefore it is difficult to apply phenolic resin as matrix of composites in transportation industries. In general, load transfer from matrix to fiber plays an important role in order to improve the mechanical properties of composites. Therefore it is

considered that the interfacial adhesion between fiber and resin significantly affects the mechanical properties.

A lot of research has already carried out on interfacial adhesion in thermosetting composites. However, the data on interfacial adhesion in fiber reinforced phenolic resin composites are limited, and there is a strong need to clarify the interfacial effects on the mechanical properties. In order to evaluate these effects, the fragmentation test is generally performed, using a single filament embedded sample [2-4]. This method can be applied for material systems with ductile matrix because the fiber must be broken before the breakage of matrix resin. It is however difficult to apply this method to phenolic resin composites because of the brittle nature of the phenolic resin. In addition, conventional phenolic resin does not have inorganic reactive function against silane coupling agent in its chemical structure, and therefore it is generally considered that the phenolic resin does not have effective adhesion between fiber and matrix. From these backgrounds some papers have discussed the mechanical properties of phenolic composites [5,6], however, interfacial effects on the mechanical properties of phenolic composites have not studied in detail [7].

In this study, therefore, the effect of interfacial adhesion was evaluated by wetting measurement and mechanical testing of the resin impregnated unidirectional fiber bundle specimen. Effect of fiber bundles on the wettability against the phenolic resin was evaluated by Wilhelmy type dynamic wetting measurement. Three-point flexure and lateral compression tests were performed on resin impregnated unidirectional fiber bundle specimens, and the effects of sizing and resin on their properties were discussed.

EXPERIMENTAL PROCEDURE

Materials

Materials used as matrix and reinforcement are summarized in Table 1. Type-1 and -3 resins were normal phenolic resins delivered from different suppliers, and type-2 resin is a silane modified resin derived from type-1 resin. Three kinds of reinforcements are glass fiber bundles. Type-A bundle was a yarn, and type-B and -C were rovings. Type-C roving was specially developed for phenolic resin in order to obtain good interfacial adhesion.

Table 1: Resins and fibers used.

Resin	Type	Fiber	Type
Type-1	Cellobond J2027L (Blagden Chem.)	Type-A	300tex yarn (fiber diameter: 13μm)
Type-2	Cellobond J2053L (Blagden Chem.)	Type-B	320tex roving (fiber diameter: 15μm)
Type-3	Norsophen 1204 (Cray Valley)	Type-C	276tex roving (fiber diameter: 13μm)

Wetting Measurement

Dynamic wetting property was measured for all the fibers to type-2 resin by Wilhelmy type dynamic wetting instrument. Five fiber bundles were combined and the one end of the bundles was tightened by the thin filament in order to reduce the resisting force during advancing process in measurement. The tightened fiber bundles were advanced into the resin at a constant speed of 0.1mm/min. After reaching the dipped length of 9.9mm the advancing

was stopped and the fiber bundle kept dipping in the resin for 180sec. During these processes, the force change was measured.

Mechanical Properties

Influence of resin and fiber on the mechanical properties was evaluated by using resin impregnated unidirectional fiber bundle composite specimen. In order to get the same fiber content in the composite, a certain number of fiber bundles were combined and they dipped into the resin bath with catalyst. After that, the resin was impregnated into the fiber bundles under vacuum condition. After finishing the impregnation process the resin impregnated fiber bundles were pulled into the PTFE tube mold with an inner diameter of 6mm covered by aluminum tube, and were cured at 80°C for 1hour. After complete curing, test specimens were cut from the impregnated fiber bundle rod.

Before the mechanical testing the impregnation of the resin into the fiber bundle and the fiber dispersion were evaluated by observing the cross-section of cured specimen. The mechanical properties were evaluated by the lateral compression test. The geometry of the specimen was 6mm in length and 6mm in diameter. The compressive load was applied to perpendicular to the long direction of the specimen. The compression strength (σ_c) was evaluated by

$$\sigma_c = \frac{P_c}{\ell \times d} \tag{1}$$

where P_c was the maximum compression load, ℓ and d were the length and diameter of the specimen. After the compression test the cross-section was observed in order to clarify the effect of fiber type on the crack propagation.

RESULTS AND DISCUSSION

Wetting Properties

Fig.1 shows the force-time curves during dynamic wetting measurement. In advancing process, the resin did not come into the fiber bundles, and the compression force appeared. The changes of force of type-A and -B in advancing process showed the same tendency, however, the compression force in type-C was higher than those in type-A and -B. This

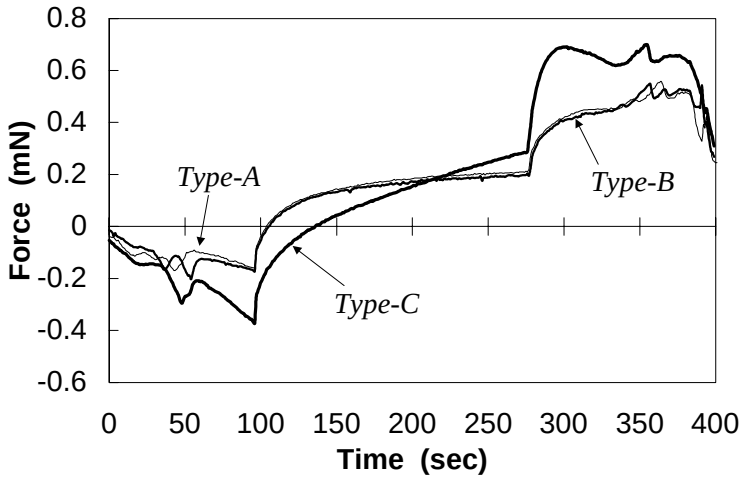


Fig.1: Changes of tension force in wettability measurement.

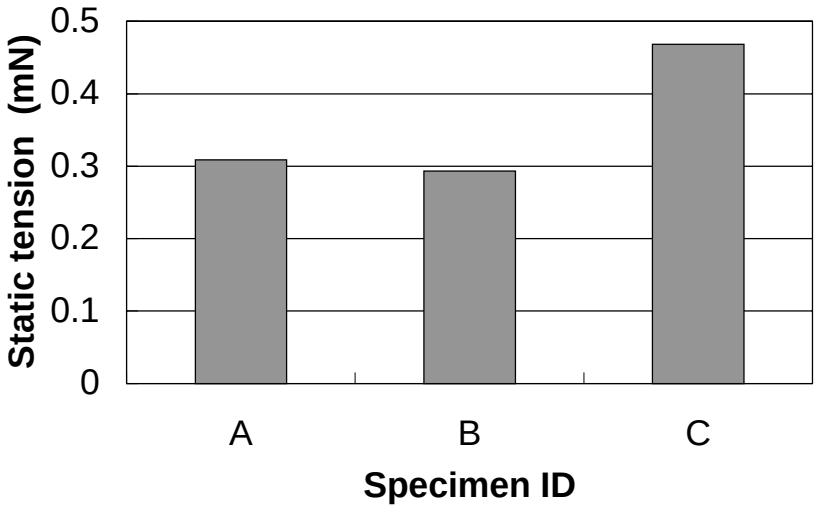
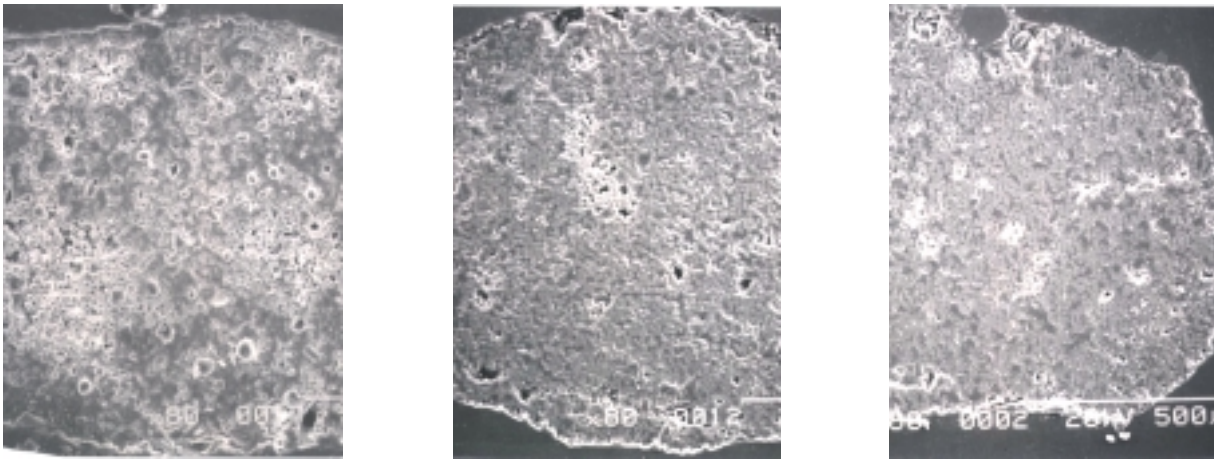


Fig.2: Comparison of Static tension force.

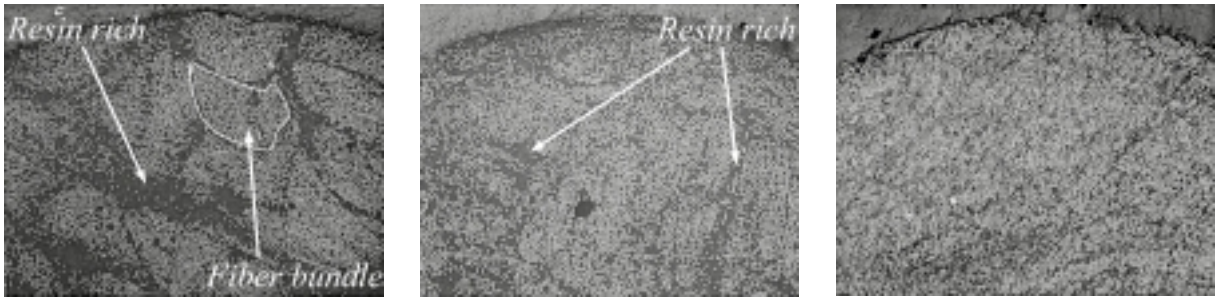


(a) (b) (c)

Fig.3: Cross-sectional micrographs of the cured fiber bundles after wetting measurement; (a) type-A, (b) type-B and (c) type-C.

phenomenon was related to the structure of the fiber bundle. Type-C fiber had poor binding and rough surface structure in bundle, and therefore the resisting force against the resin was higher than those in type-A and -B. In advancing process, the wetting to the resin could not be evaluated well. During static wetting process, the resin came into the fiber bundles and the force increased gradually. In type-A and -B the force increased rapidly and soon reached a constant value. On the contrary, the force continued to increase during static wetting process in type-C.

From the wetting measurement the static wetting tension was evaluated by the difference of the tension between the beginning and end of static wetting process. Fig.2 summarized the static tension force for each type of fibers. In comparison with type-A and -B the static tensions were almost the same independent of the bundle structure. These fibers were treated by the general procedure (not for phenolic resin) and major difference was only the bundle structure (yarn or roving). Therefore it is considered that the bundle structure affects a little the wetting property. On the other hand, the static tension in type-C was much higher than that in type-B. The difference between type-B and -C is the sizing of the fiber. Therefore it is



(a) (b) (c)

Fig.4: Cross-sectional micrographs of molded specimens with type-1 resin; (a) type-A, (b) type-B and (c) type-C fibers.

clear that the sizing developed for phenolic resin in type-C significantly improves the wetting property.

After the wetting measurement, the fiber bundles were cured in order to observe the impregnation of the resin into the fiber bundles. Fig.3 shows the cross-sectional micrographs of each fiber after the wetting measurement. In type-A and -B a lot of voids were remained inside the fiber bundle. This suggested that the impregnation of the resin is not well in these fibers. On the other hand, a few voids could be observed in type-C, and these results corresponded to the results of static wetting.

Mechanical Properties

Before the mechanical testing, the resin impregnation inside the compression test specimens was observed by microscope. Fig.4 shows the cross-sectional micrographs of the compression test specimens. In type-A specimen the fiber still existed as the bundle, and the resin rich region remained between the fiber bundles. Also in type-B specimen the resin rich region could be observed in spite of well dispersion of the fiber. On the other hand, the fiber distributed homogeneously and the resin rich region could not be observed. These tendencies were the same independent of the matrix resin. Therefore it is clearly seen that sizing developed for phenolic resin is effective to get the well dispersion of the fiber. In addition, the surface of the specimen was also observed by microscope. In type-1 and -3 specimens

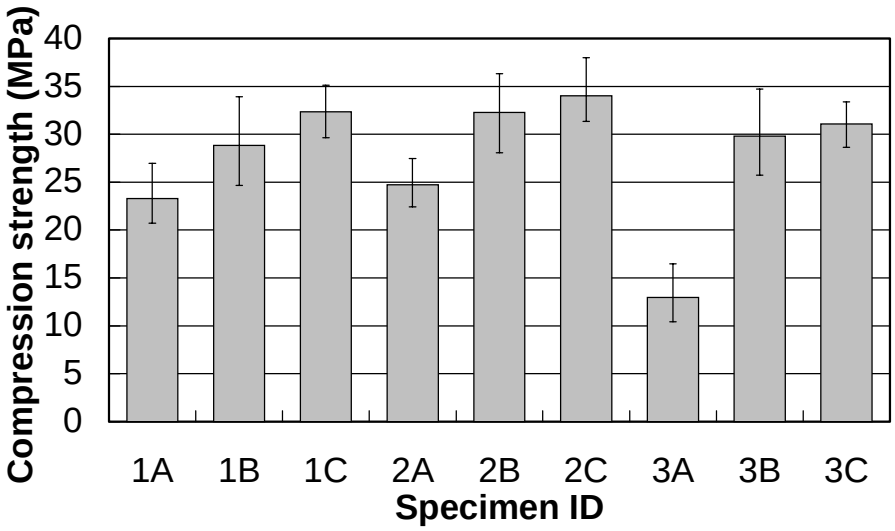
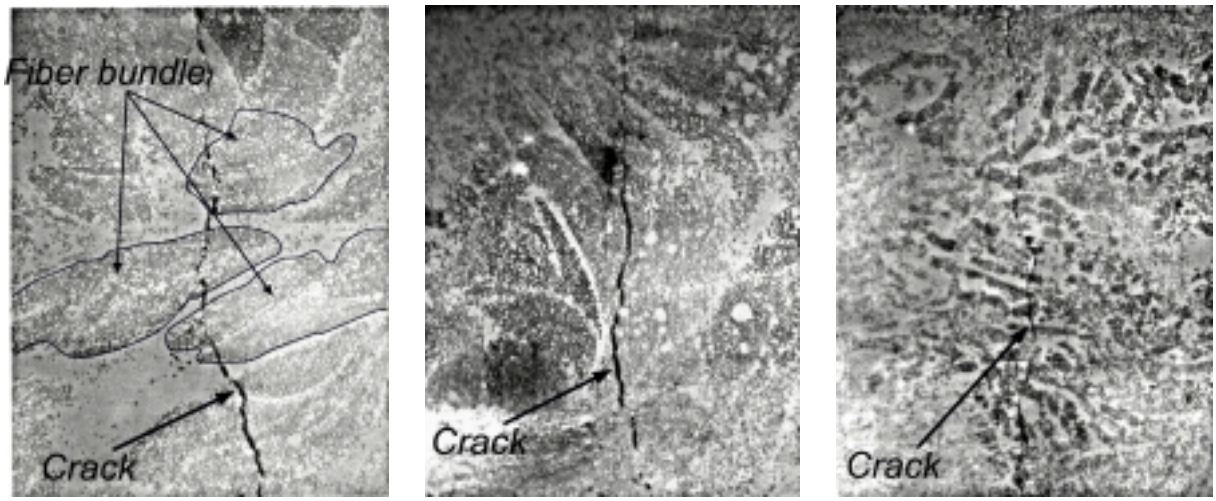


Fig.5: Comparison of lateral compression strength.



(a) (b) (c)

Fig.6: Cross-sectional micrographs of fractured specimens in compression; (a) type-A, (b) type-B and (c) type-C.

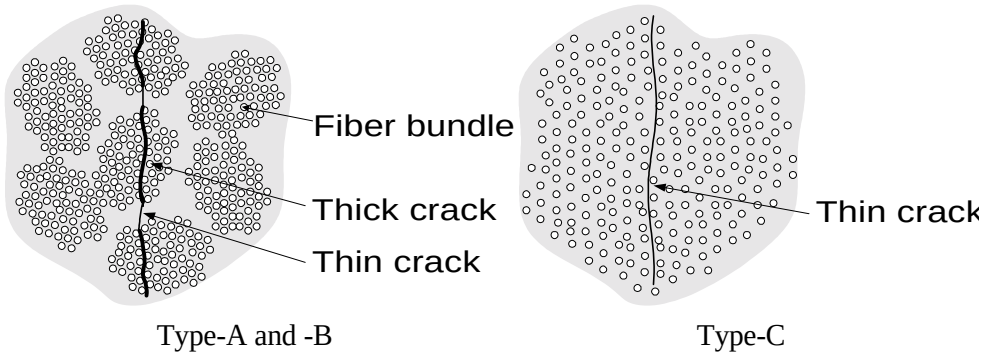


Fig.7: Schematic representation of crack propagation in compression test.

many voids also remained on the surface of the specimen and the size of each void was millimeter order. However, the void size in type-2 specimen was reduced.

Fig.5 summarizes the compression strength of each specimen. Independent of the matrix resin the strength of type-A specimen showed the lowest value among the series of specimens. Among roving specimens (type-B and -C, which were different in sizing) the strength of type-C was higher than that of type-B. In general, it is considered that mechanical properties of FRP are enhanced by improvement of wetting. In phenolic composites tested here, however, the improvement of strength by sizing was not so effective compared with the improvement of wetting shown in Fig.2. Therefore it seems that the strength is affected by the other effect in addition to the wetting. The silane modification of phenolic resin also improved the strength, especially in type-B fiber. In order to discuss the effect of fiber and resin on the strength the cross-section of the fractured specimens was observed by microscope. Fig.6 shows the cross-sectional micrographs of the fractured specimens in compression. In all the specimens, single crack propagated parallel to the loading direction. This means that crack initiated from the center of the specimen due to the tensile stress perpendicular to the loading direction. In type-A and -B specimens thick crack occurred and progressed rapidly. In particular, the width of crack opening was much wider in the fiber bundle. On the other hand, only thin crack could be observed in type-C specimen. These results may be related to the fiber volume content at local area. Fig.7 illustrated the crack propagation mechanism in the lateral compression test.

In type-A and -B the shape of the fiber bundle was still remained in the cured specimen and resin rich region existed between the fiber bundle. This induced the heterogeneity of fiber content in the specimen. At the fiber concentrated area the local fiber volume content was high and it induced the significant stress concentration. Therefore the crack initiated at lower stress and eventually it led the lower compression strength. In type-C specimen, however, the well dispersion of the fiber was obtained shown in Fig.4, and as a result, the local stress concentration did not caused. From this discussion, it is considered that the compression strength in the impregnated fiber bundle phenolic composite is significantly affected by the fiber dispersion. The difference in strength due to the silane modification was produced by the influence of void size. According to the detailed microscopic observation, the void size on the surface was reduced by the silane modification of the resin. Therefore it is considered that the void on the surface is also one of the sensitive factor to the compression strength in phenolic composite.

CONCLUSION

This paper dealt with the effect of fiber and resin on wetting and mechanical properties of glass fiber/phenolic composite. Three kinds of fibers with different structure and sizing were used as reinforcement and three kinds of resins with different composition were used as matrix. Modification of sizing remarkably improved the static wetting and it also improved the mechanical strength. Improvement of strength related to the fiber depended on fiber dispersion. Well fiber dispersion did not induce the local stress concentration and it led the higher compression strength. Silane modification of resin also improved the mechanical strength. Silane modification of resin reduced the void size and it improved the compression strength.

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