工學碩士 學位論文

VARTM을 공법을 통한 JFRP의 계면처리 효과에 따른 기계적 특성에 대한 연구

Effect of coupling agent on the mechanical properties of jute fiber reinforced plastic made by VARTM

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2012年 8月

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材 料 工 學 科

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本 論文을 安熙範의 工學碩士 學位論文으로 認准함.

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Contents

Abstract

The jute is among the best of natural fibers in terms of tensile strength and availability. So jute fibers were used by VARTM (Vacuum Assisted Resin Transfer Molding) with silane coupling agent to make stronger JFRP(Jute Fiber Reinforced Plastic).

The purpose of this study was to determine the mechanical properties of epoxy resins and jute fiber as composites and to determine their validity. For this, the JFRP was made using VARTM and hand lay-up. This work evaluated the mechanical properties of the composites according to various manufacturing processes. The specimens made using the two methods were investigated using the tensile test and short-beam test. The specimen made by VARTM is stronger than that made by hand lay-up.

The introduction of a coupling agent distinctly influenced the mechanical properties of the composites. The strength of the specimen using a silane coupling agent eventually became stronger than the specimen without a silane coupling agent. Whether the JFRP was washed by alkali before use with coupling agent affected its mechanical properties, such as tensile strength and short-beam strength.

Using hand lay-up, VARTM, and a silane coupling agent, the jute fiber of volume fraction was checked by specimens. First, the jute fiber's weight was measured, and then the weight of the JFRP was confirmed. On the basis of the experiments, the hand lay-up, VARTM, and coupling agent were measured using the percentage formula. The specimens made using VARTM had a higher jute fiber percentage than those made by hand lay-up, but there was no difference regarding the existence of silane in the VARTM process.

The tensile fracture surface of each specimen was confirmed by SEM. The specimens made by hand lay-up, VARTM and washed and not by alkali before being used silane coupling agent were examined respectively for the observing interfacial adhesion from the covalent bond between the fiber and the matrix. Using the specimens observed by SEM, the difference of the strength of the specimens was investigated. In the case of the JFRP washed by alkali, the silane coupling agent improved interfacial adhesion from the covalent bond between the fiber and the matrix.

Chapter 1 Introduction

1-1 Background

In recent years, as a result of environmental and economic concerns, there has been a growing interest in the use of natural fiber reinforced composites by the academic sector and the industry [1]. Natural fibers have many significant advantages over synthetic fibers. Currently, many types of natural fibers have been investigated for use in plastics, including flax, hemp, jute straw, wood, rice husk, wheat, barley, oats, rye, cane (sugar and bamboo), grass, reeds kenaf, ramie, oil palm empty fruit bunch, sisal, coir, kapok, paper mulberry, banana fiber, pineapple leaf fiber, bamboo etc. Thermoplastics reinforced with special wood fillers are enjoying rapid market growth due to their many advantages, such as light-weight, reasonable strength, and stiffness [2]. Furthermore, natural fibers have been used for the reinforcement of polymeric matrices, to produce composites for low-cost applications. The jute is among the best of natural fibers in terms of tensile strength and flexural properties. The physical properties of fiber reinforced composite materials depend on various factors, such as the properties of the fiber and matrix polymer, the interface between the fiber matrix, and the fiber content.

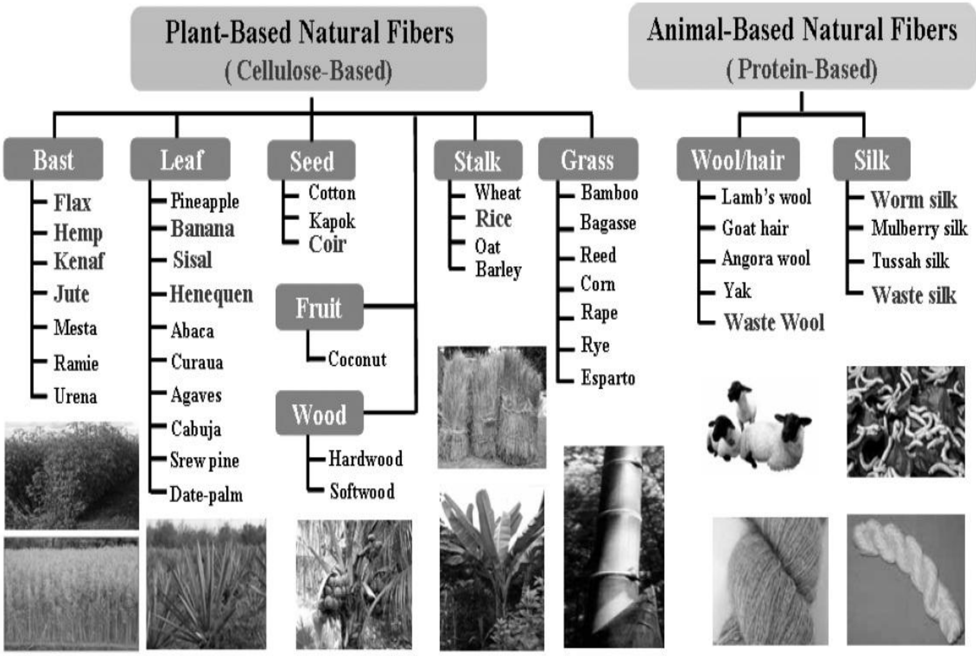


Fig. 1 Images of the natural fibers

1-2 Introduction of jute

Jute is a rain-fed crop with little need for fertilizer or pesticides. The production is concentrated in Bangladesh and some in India, mainly Bengal. The jute fiber comes from the stem and ribbon (outer skin) of the jute plant. The fibers are first extracted by retting. The retting process consists of bundling jute stems together and immersing them in low, running water. There are two types of retting: stem and ribbon. After the retting process, stripping begins. Women and children usually do this job. In the stripping process, non-fibrous matter is scraped off, then the workers dig in and grab the fibers from within the jute stem. India, Pakistan, China are the large buyers of local jute while Britain, Spain, Ivory Coast, Germany and Brazil also import raw jute from Bangladesh. India is the world's largest jute growing country. Jute is the second most important vegetable fiber after cotton; not only for cultivation, but also for various uses. Jute is used chiefly to make cloth for wrapping bales of raw cotton, and to make sacks and coarse cloth. The fibers are also woven into curtains, chair coverings, carpets, area rugs, hessian cloth, and backing for linoleum. While jute is being replaced by synthetic materials in many of these uses, some uses take advantage of jute's biodegradable nature, where synthetics would be unsuitable. Examples of such uses include containers for planting young trees, which can be planted directly with the container without disturbing the roots, and land restoration where jute cloth prevents erosion occurring while natural vegetation becomes established. The fibers are used alone or blended with other types of fiber to make twine and rope. Jute rope has long been popular in Japan for use in bondage. Jute butts, the coarse ends of the plants, are used to make inexpensive cloth. Conversely, very fine threads of jute can be separated out and made into imitation silk. As jute fibers are also being used to make pulp and paper, and with increasing concern over forest destruction for the wood pulp used to make most paper, the importance of jute for this purpose may increase. Jute has a long history of use in the sackings, carpets, wrapping fabrics (cotton bale), and construction fabric manufacturing industry. Traditionally jute was used in traditional textile machineries as textile fibers having cellulose (vegetable fiber content) and lignin (wood fiber content). But, the major breakthrough came when the automobile, pulp and paper, and the furniture and bedding industries started to use jute and its allied fibers with their non-woven and composite technology to manufacture nonwovens, technical textiles, and composites. Therefore, jute has changed its textile fiber outlook and steadily heading towards its newer identity, i.e., wood fiber. As a textile fiber, jute has reached its peak from where there is no hope of progress, but as a wood fiber jute has many promising features. Also, the importance of eco-friendly materials is recently emerging as alternative energy sources.

1-3 character of jute fiber

- Jute fiber is 100% bio-degradable and recyclable and thus environmentally friendly.
- It is a natural fiber with golden and silky shine and hence called 'The Golden fiber.'
- It is the cheapest vegetable fiber procured from the best or skin of the plant's stem.
- It is the second most important vegetable fiber after cotton, in terms of usage, global consumption, production, and availability.
- It has high tensile strength, low extensibility, and ensures better breathability of fabrics. Therefore, jute is very suitable in agricultural commodity bulk packaging.
- It helps to make best quality industrial yarn, fabric, net, and sacks. It is one of the most versatile natural fibers that has been used in raw materials for packaging, textiles, non-textile, construction, and agricultural sectors. Bulking of yarn results in a reduced breaking tenacity and an increased breaking extensibility when blended as a ternary blend.
- Advantages of jute include good insulating and antistatic properties, as well as having low thermal conductivity and a moderate moisture regain. Other advantages of jute include acoustic insulating properties and manufacture with no skin irritations.

Table 1 Chemical component of natural fibers

Type of Fiber	Cellulose	Lignin	Hemicellulose	Pectin	Wax	Moisture Content
	wt%	wt%	wt%	wt%	wt%	wt%
Bast						
Jute	61~71.5	12~13	13.6~20.4	0.2	0.5	12.6
Flax	71	2.2	18.6~20.6	2.3	1.7	10.0
Hemp	70.2~74.4	3.7~5.7	17.9~22.4	0.9	0.8	10.8
Ramie	68.6~76.2	0.6~0.7	13.1~16.7	1.9	0.3	8.0
Kenaf	31~39	15~19	21.5	-	-	-
Leaf						
Sisal	67~78	8.0~11	10.0~14.2	10.0	2.0	11.0
PALF	70~82	5~12	-	-	-	11.8
Henequen	77.6	13.1	4~8	-	-	-
Seed						
Cotton	82.7		5.7	-	0.6	-
Fruit						
Coir	36~43	41~45	0.15~0.25	3~4		8.0

Table 2 Properties of natural fibers

Type of Fiber	Density	Diameter	Tensile Strength	Young's Modulus	Elongation Break
	g/cm3	μm	MPa	GPa	%
Cotton	1.5~1.6	-	287~800	5.5~12.6	7.0~8.0
Jute	1.3~1.4	25~200	393~773	13~26.5	1.16~1.5
Flax	1.50	-	345~1100	27.6	2.7~3.2
Hemp	-	-	690	-	1.6
Ramie	1.50	-	400~938	61.4~128	1.2~3.8
Sisal	1.45	50~200	468~640	9.4~22.0	3~7
PALF	-	20~80	413~1627	34.5~82.51	1.6
Coir	1.15	100~450	131~175	4~6	15~40
E-glass	2.5		2000~3500	70	2.5
S-glass	2.5	-	4570	86	2.8
Aramid	1.4	-	3000~3150	63~67	3.3~3.7
Carbon	1.7	-	4000	230~240	1.4~1.8

(b) Products of jute fiber

Fig. 2 Images of the jute fiber and products

Chapter 2 Method of experiments

Various types of materials in many forms have been used for the molding of composites. Depending on the type of material used, both the molding form and conditions vary, and there is a substantial difference in the product's properties. The criteria for the selection of a more efficient process, other than the cost of any process, are symmetry of size and shape, specific strength and surface treatment. Figure 3 shows the selection criteria considering production efficiency.

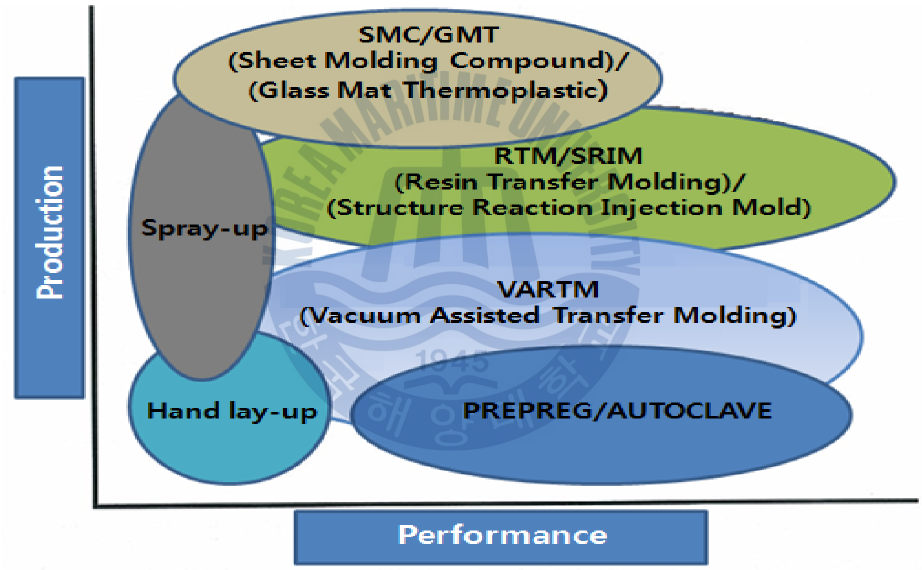
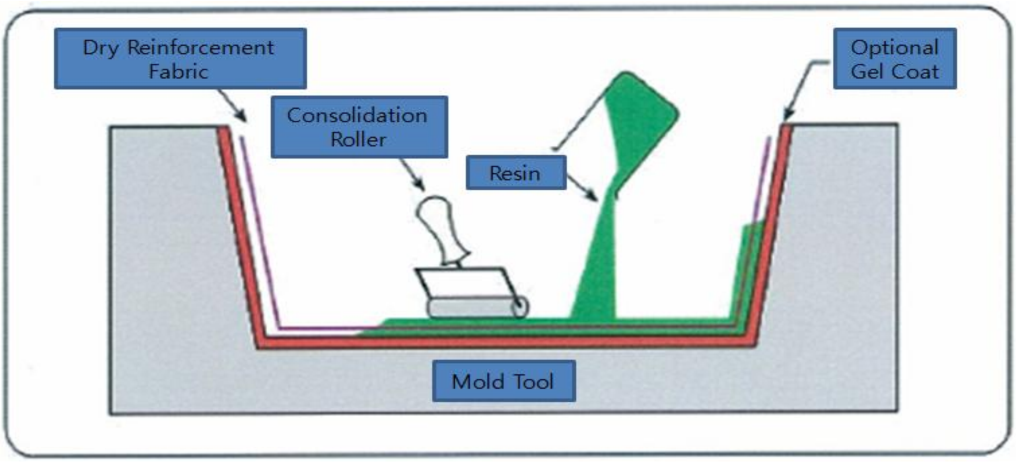


Fig. 3 Selection of effective process

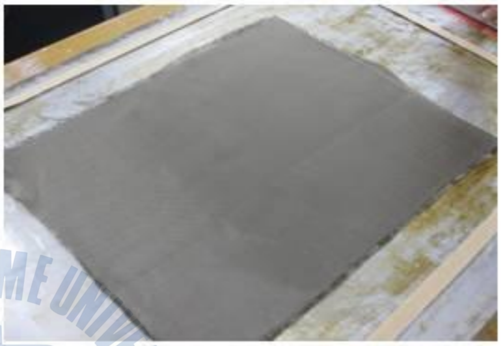
2-1 Hand lay-up

Hand lay-up is a simple method for composite production. A mold must be used for hand lay-up parts unless the composite is to be joined directly to another structure. The mold can be as simple as a flat sheet or have infinite curves and edges. For some shapes, molds must be joined in sections so they can be taken apart for part removal after curing. Before lay-up, the mold is prepared with a release agent to insure that the part will not adhere to the mold.

Reinforcement fibers can be cut and laid in the mold. It is up to the designer to organize the type, amount and direction of the fibers being used. Resin must then be catalyzed and added to the fibers. A brush, roller or squeegee can be used to impregnate the fibers with the resin. The lay-up technician is responsible for controlling the amount of resin and the quality of saturation.



(a) Release Coating



(b) Stacking Reinforcements

(c) Impregnation of Resin

(d) Cure

Fig. 4 Image of the hand lay-up procedure

2-2 VARTM

Vacuum bagging follows similar steps to the hand lay-up process. When the resin is adequately impregnated into the fibers with excess, the part is sealed with vacuum bagging materials. A layer of peel ply is laid down on the part because the resin will not stick to peel ply. Then a layer of breather cloth is laid down to soak up excess resin and insure an adequate path for the vacuum pressure. Then a layer of plastic sheeting is laid down and sealed around the part to isolate the part from the atmosphere. A hose connected to a vacuum is attached to the sealed part. A vacuum is then applied to the enclosed part. The part will be compacted by the vacuum and the breather cloth will collect the excess resin. Vacuum resin transfer molding follows the first steps of hand lay-up. The mold is prepared with a release agent, the fibers are cut and placed into the mold, and then new steps are taken. The next steps are as follows: a layer of peel ply is laid down on the fibers, a layer of mesh netting is laid on the part, special air tubes are fixed at desired entrance and exit points, vacuum tubes are connected to the air tubes, the part is sealed with plastic sheeting and a vacuum is applied.

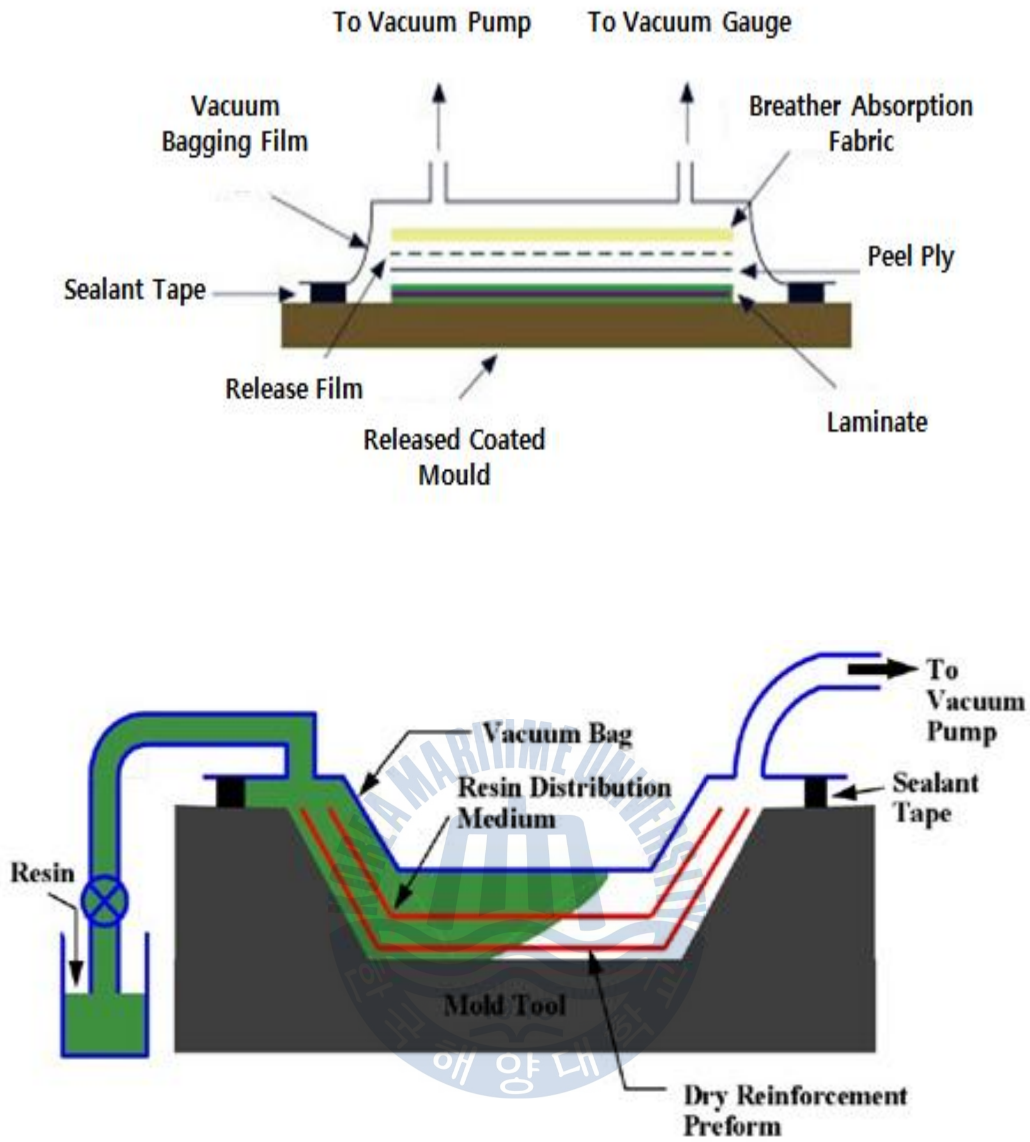
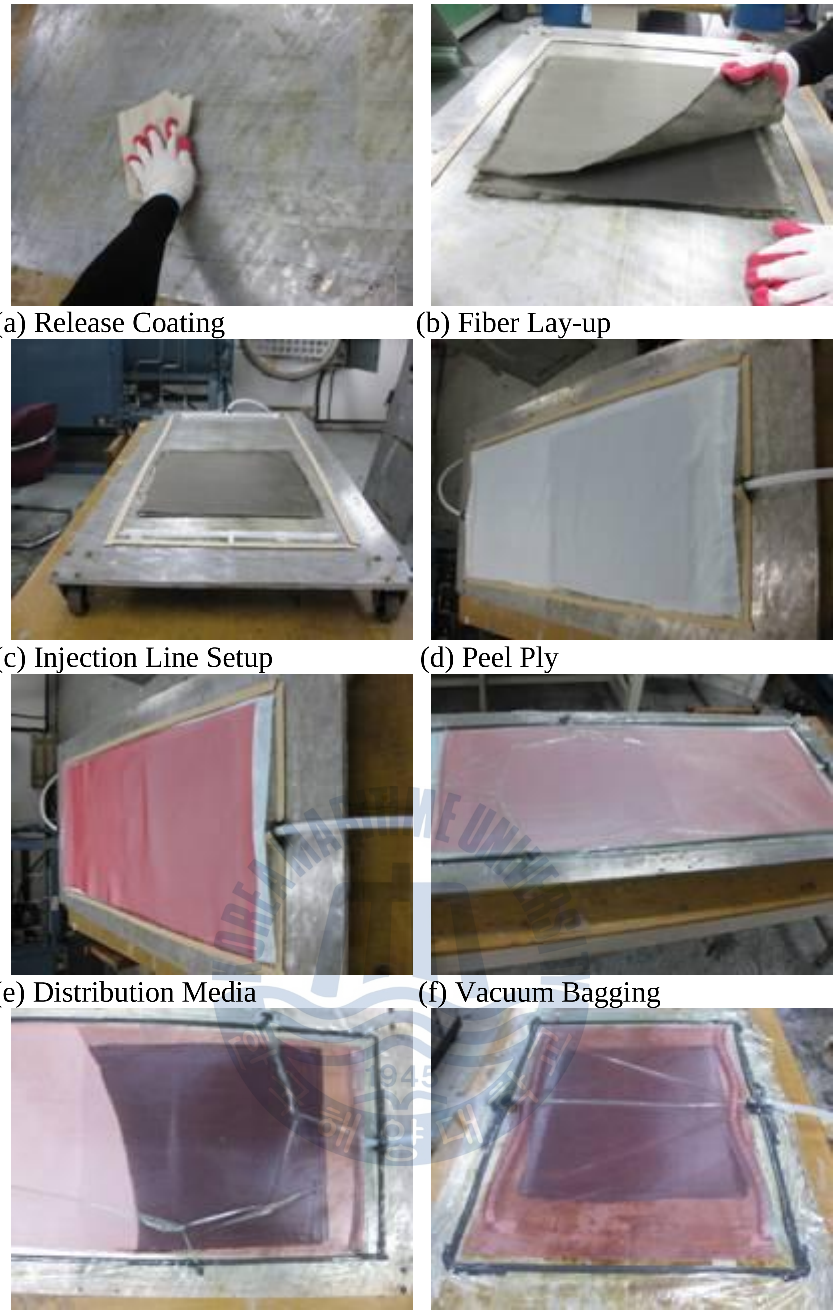


Fig. 5 Image of the VARTM process



(a) Release Coating (b) Fiber Lay-up

(c) Injection Line Setup (d) Peel Ply

(e) Distribution Media (f) Vacuum Bagging

(g) Resin Infusion (h) Cure

Fig. 6 Image of the VARTM procedures

2-3 Coupling agent

Treatments of alkali solution and silane coupling agent are typical way for a improved adhesion between the fiber and the matrix. The alkali solution treatment can make the fiber of the crystallizability, because of exiting the Hemicelluloses and Lignin within the fiber and eliminating the weak surface layer. Alkali solution by processing the fibers within the Hemicelluloses, lignin fibers exit while removing weak surface layer has a more deterministic. The silane coupling agent can improve the interfacial adhesion from covalent bond between the fiber and the matrix. The hygroscopicity can be shown from the characteristics of a good interfacial adhesion. The interfacial adhesion depends on the interfacial contact area of the acid-base between the fiber and matrix in a lot of effectors. The combination of acid-base form the fiber interface is an important factor which is to the control of interfacial adhesion strength in the matrix. So, when the combination of the acid-base in the matrix combination is known, the best combination can be possible between the fiber and the matrix.

2-3-1 Coupling agent experiment

The fiber was treated in room temperature for 2 hours, and then the jute was washed by alkali until pH 7 and dried completely from room temperature to 80℃ for a period of time. Also, the untreated jute fiber was completely dried at 80℃. Meanwhile, the surface treatment method used by silane coupling agent is to dry completely in an oven at 50℃ after being immersed in 0.5 wt% silane coupling aqueous at a room temperature for a minute. The specifications of coupling agent are as following

Table 3 Properties of the silan coupling agent

XIAMETER OFS-6030 Silane		
Test	Unit	Value
Appearance	Cst	Colorless to very pale yellow liquid
Viscosity at 25C (77F)		2.5
Specific Gravity at 25C (77F)		1.04
APHA Color	℃(˚F)	138 (280)
Purity by GC	%	>98.9
Molecular weight		248.35
CAS#		2530-85-0

XIAMETER OFS-0611 Silane		
Test	Unit	Value
Appearance	Cst	Colorless to very pale yellow liquid
Viscosity at 25C (77F)		1.6
Specific Gravity at 25C (77F)		0.946
APHA Color	℃(˚F)	96 (205)
Purity by GC	g/mol	>98.5
Molecular weight		221.37
CAS#		919-30-2

Fig. 7 Image of the silane coupling agent and chemical structure

Chapter 3 Experiment

In this section, the equipment used to measure the mechanical properties of the JFRP is detailed. Three kinds of specimens were made, by hand lay-up, VARTM and coupling agent, respectively. These three specimens types were measured by a tensile test, a short-beam test, optical microscopy and SEM.

3-1 Experiment equipments

3-1-1 Universal testing machine

A universal testing machine, also known as a universal tester, materials testing machine or materials test frame, is used to test the tensile stress and compressive strength of materials. It is named after the fact that it can perform many standard tensile and compression tests on materials, components, and structures. The set-up and usage are detailed in a test method, often published by a standards organization. This specifies the sample preparation, gauge length (the length which is under study or observation), analysis, etc. The specimen is placed in the machine between the grips and an extensometer if required can automatically record the change in gauge length during the test. If an extensometer is not fitted, the machine itself can record the displacement between its cross heads on which the specimen is held. However, this method not only records the change in length of the specimen but also all other extending / elastic components of the testing machine and its drive systems including any slipping of the specimen in the grips.

Fig. 8 Image of the universal test machine

3-1-2 Optical microscope

An optical microscope is a type of microscope that uses a beam of electrons to illuminate the specimen and produce a magnified image. The optical microscope uses electrostatic and electromagnetic "lenses" to control the electron beam and focus it to form an image. These lenses are analogous to, but different from the glass lenses of an optical microscope that forms a magnified image by focusing light on or through the specimen. So, the surrounding of the fracture surface was observed by the optical microscope, after being measured by tensile test.

Fig. 9 Image of the optical microscope

3-1-3 SEM

Normally, thin film deposited with sputtering process is composed of minute target metal particle produced by plasma collision. Unit size of the deposited target metal particle is nm or less. Thus, high magnification and resolution analysis device is needed in order to observe distribution structure of this deposited particle on thin film. In this research, SEM is used to observe the distribution structure of metal thin film particle deposited on transparent conductive multilayer thin film. SEM uses field emission electron gun. Field emission electron gun process cathode tip as minute needle form, which is below nm, and apply high voltage to lead electron (tunneling effect) and by using the contact lenses, make electron as a prove structure to observe surface structure. It is usually used in field where high magnification and resolution is required, and it can observe the surface with 1000000 time high magnification. So, each specimen is conducted by an SEM, after tested materials.

Fig. 10 Image of the SEM

3-2 Characteristic evaluation

3-2-1 Mechanical test

The tensile and the three-point short-beam test were conducted to identify the mechanical properties of the JFRP. The tensile test was conducted in accordance with ASTM D 638. The test conditions and the specimens' dimensions are presented in Fig. 11. Seven specimens for each experiment were made by hand lay-up and VARTM. Of these, five specimens, excluding those with the highest and the lowest results, were taken to represent the average. The equipment used was Kyung-Do's universal test machine (Model: KDMT-156). In accordance with the relevant specifications, the test method of steps for Cross head speed was 5mm/mm of tensile test and 1mm/mm of short-beam test. The shape of each equipment item is shown in Fig. 11.

Accordingly, the specimens (Fig. 11 (b)) underwent ASTM D 2344, to test short-beam strength prior to their short-beam test. The short beam strength results follow.

$$F^{sbs} = 0.75 \times \frac{P_m}{(b \times s)} \quad (1)$$

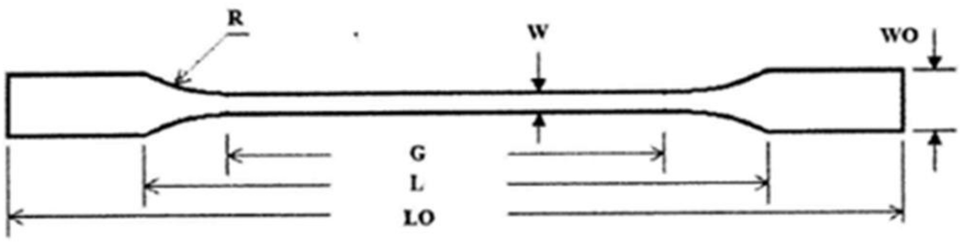
F^{sbs} : Short-beam strength [MPa]

Pm : Max load observed during the test [N]

b : Measured specimen width [mm]

h : Measured specimen thickness [mm]

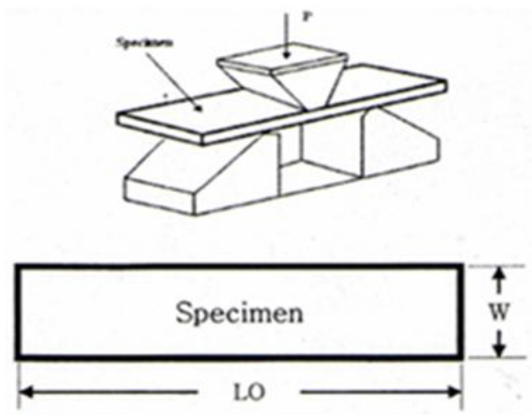
Stacking direction	[0 °, 90 °]
Spec.	ASTM D 638
Temperature	Room temperature, dry
Test speed	5 mm/min



Unit : mm	T	L	WO	W	LO
Dimension (ASTM)	3.0	57	19	13	165

(a) Condition of tensile test and dimension of tensile specimen.

Stacking direction	[0 °, 90 °]
Spec.	ASTM D 2344
Temperature	Room temperature, dry
Test speed	1 mm/min



Unit : mm	T	W	LO
Dimension (ASTM)	3.0	6.0	18.0

(b) Condition of short-beam test and dimension of short-beam specimen.

Fig. 11 Condition and dimension of each test specimen

(a) Tensile test

(b) Short-beam test

Fig. 12 Images of method for each test

3-2-2 Measurement of the volume fraction

For glass fibers, the volume fraction of jute fiber is measured by combustion to identify the affects of its mechanical properties. But, using combustion for JFRP, the jute fibers would burn with the resin. For this reason, the volume fraction of the jute was measured as a proportion of weight. The method was to compare the weight of the JFRP before and after the addition of jute fiber.

$$\textbf{Jute fiber percentage} = \frac{\textbf{Jute fiber weight}}{\textbf{JFRP weight}} \times \mathbf{100}$$

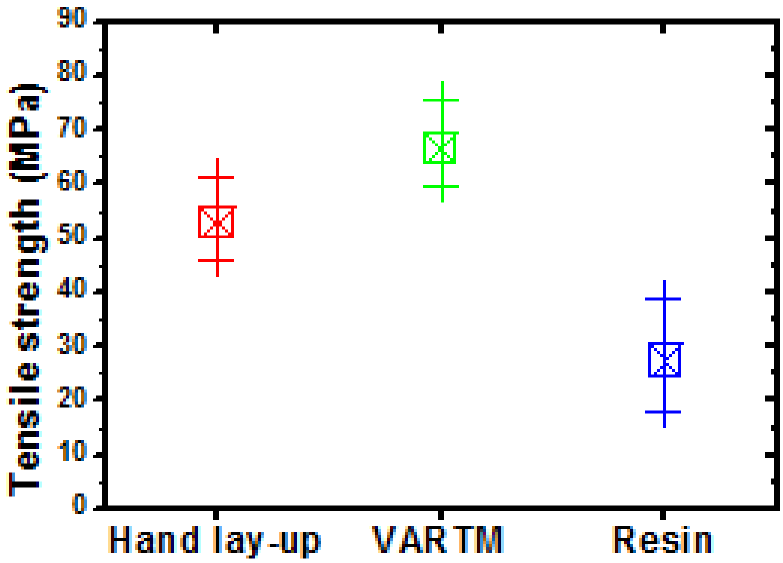
Fig. 13 Images of specimens for fiber content

Chapter 4 Results and Discussion

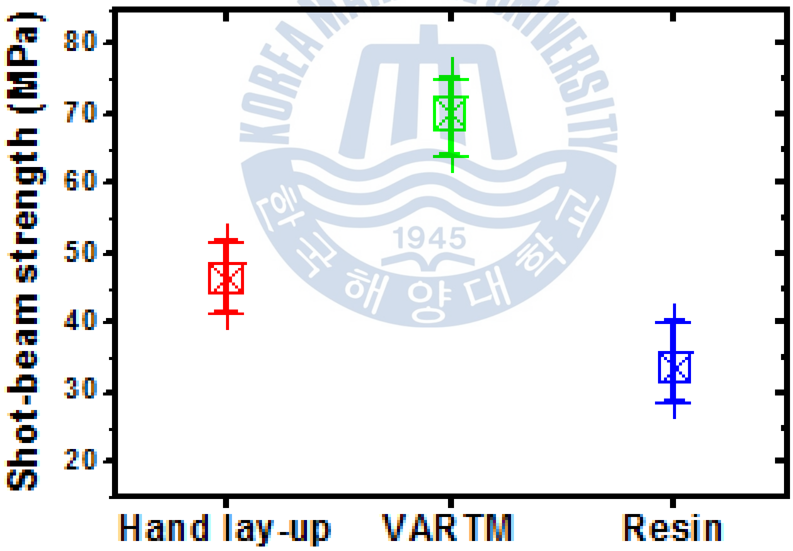
4-1 Comparison hand lay-up and VARTM

When the mechanical properties resulting from each molding process were analyzed, the VARTM process proved superior in all aspects to the hand lay-up process based on the strength of JFRP specimens. The mechanical properties of hand lay-up and VARTM resin, as shown in the results of the tensile test and the short-beam test are presented in Fig. 14. Both tensile strength and short-beam strength is higher in the VARTM specimens. Although the strength of hand lay-up specimens is lower than that of the VARTM specimens, it is higher than that of pure resin. Strength of resin in specimens was not consistent with tensile strength. However, the short-beam test results matched the tensile strength results.

To further analyze these results, the optical microscope and the SEM were used. As shown in Fig. 15, when observed by optical microscope, the occurring inner void in the hand lay-up specimens remained greater than the inner void in the VARTM specimens. This micro-void affected the mechanical properties of hand lay-up specimens. The results shown in Fig.16 and 17 were observed by SEM after the tensile test. When the impregnation of resin was observed, specimens made by VARTM were seen to be better impregnated than specimens made by hand lay-up.



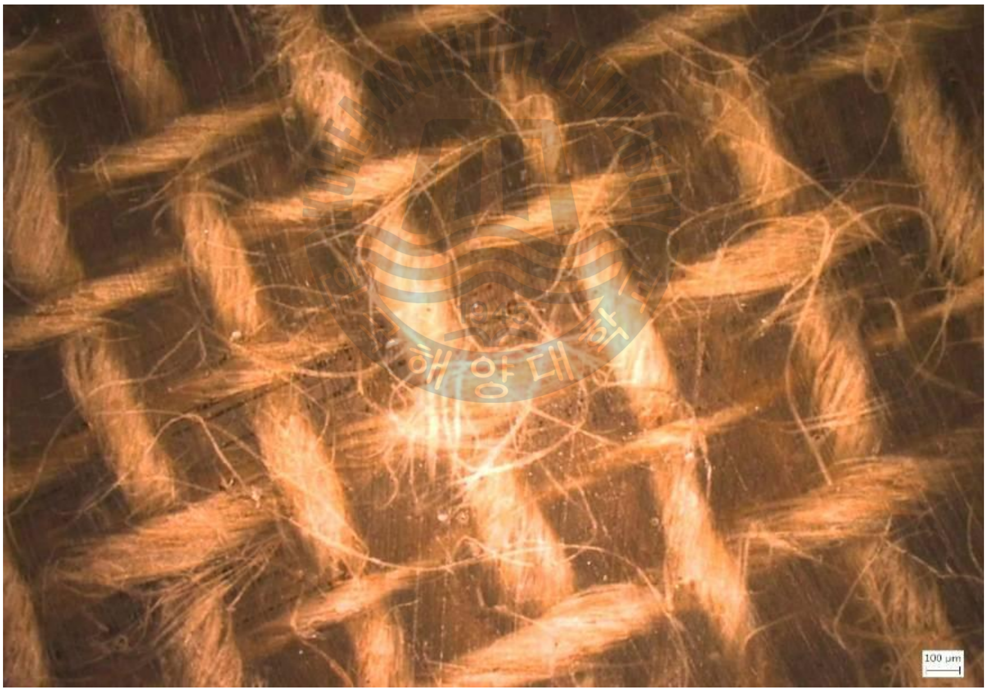
(a) Results of the tensile test



(a) Results of the short-beam test

Fig. 14 Results of each test

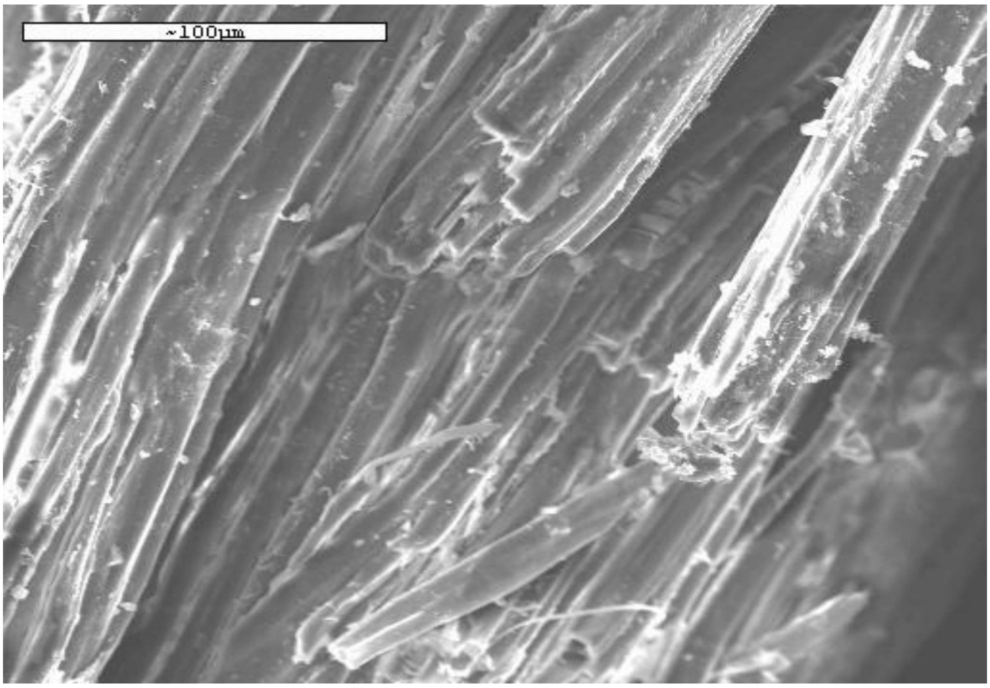
(a) Surface made by hand lay-up



(a) Surface made by VARTM

Fig. 15 Images of the surface made by hand lay-up and VARTM

(optical microscope)



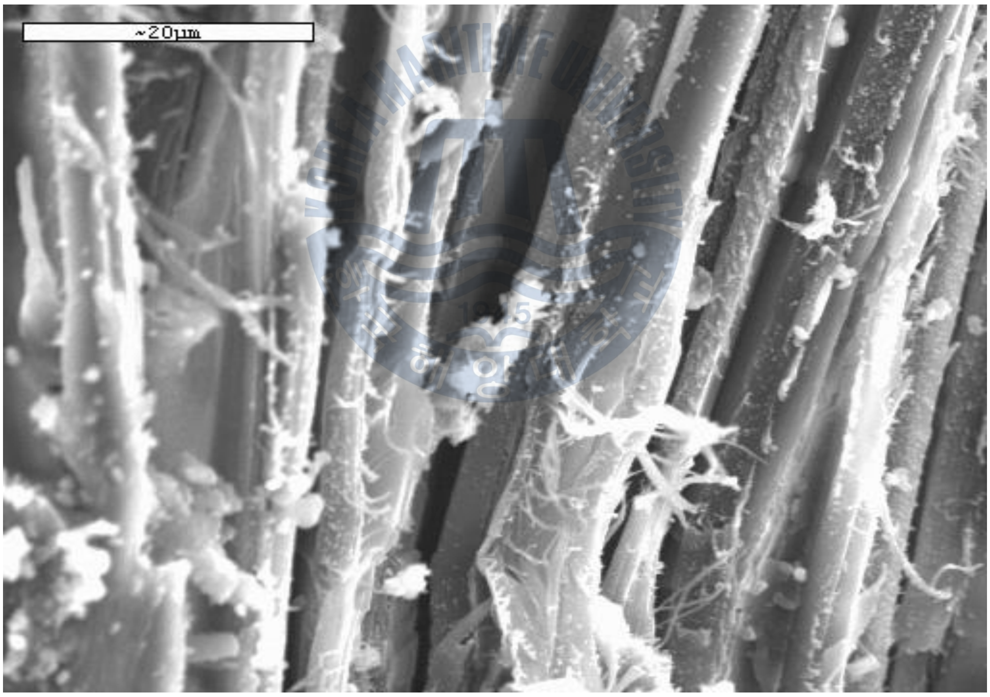
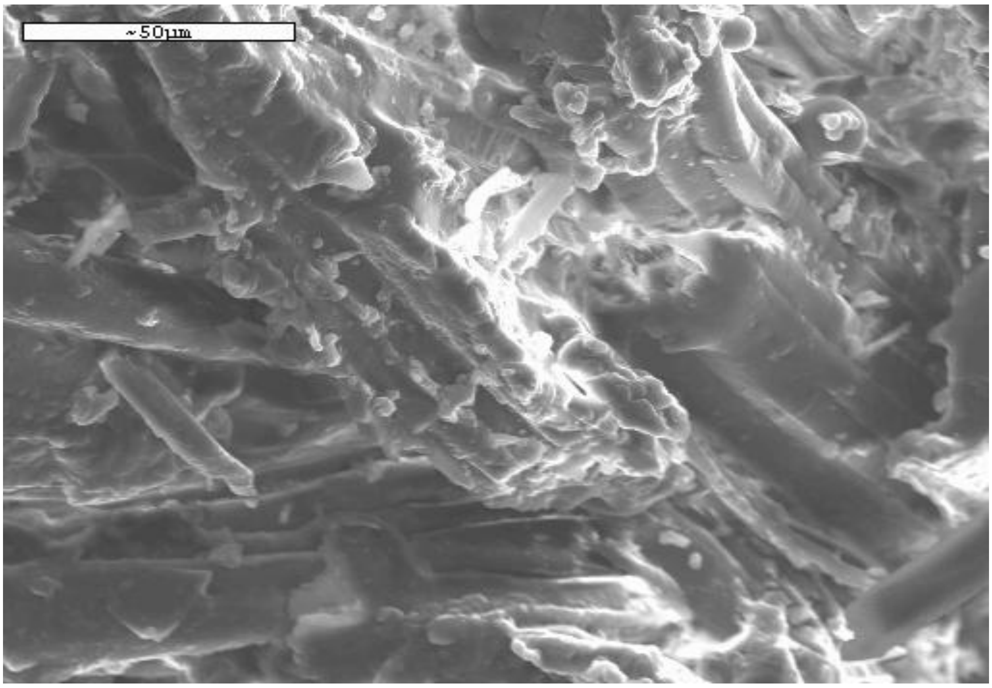


Fig. 16 Images of specimens made by hand lay-up (SEM)



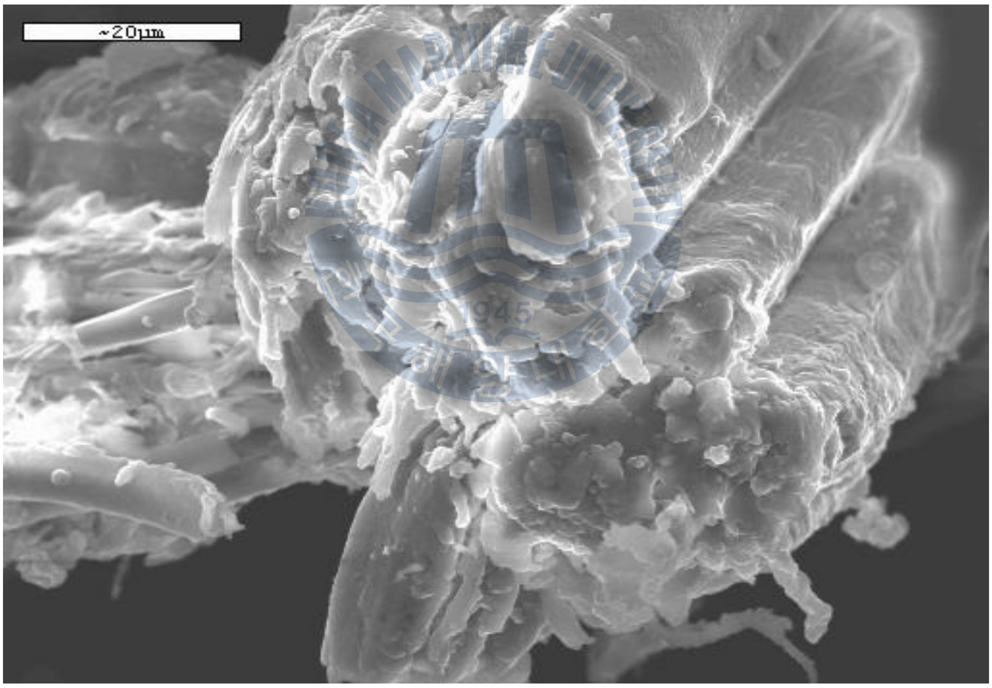
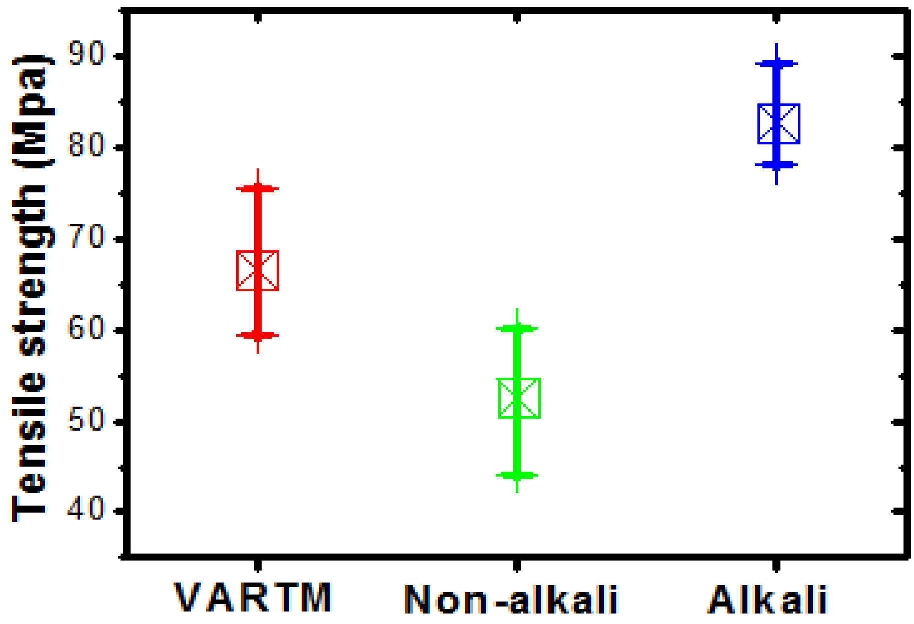


Fig. 17 Images of specimens made by VARTM (SEM)

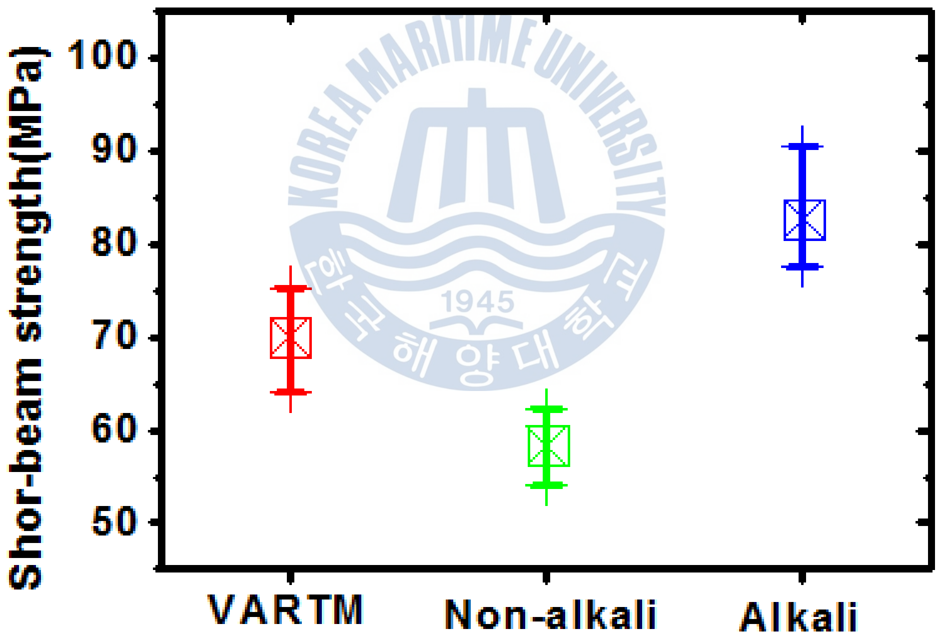
4-2 Effect of coupling agent

This comparison between the hand lay-up and VARTM process shows that the VARTM process is superior. The JFRP was tested to identify the affect of the jute being washed in alkali before the silane coupling agent was used. The results showed that JFRP washed in alkali before the silane coupling agent was added was stronger.

First, the mechanical properties of alkali-washed JFRP against those of non-alkali washed JFRP, were examined. Fig. 18 shows that the tensile strength of JFRP washed in alkali was higher than that of JFRP not washed in alkali. The tensile strength of JFRP with no coupling agent was higher than that of non-alkali-washed JFRP with a silane coupling agent. The short-beam tests gave the same values as the tensile strength tests. The alkali-washed JFRP had the highest tensile strength and the highest short-beam strength of all the specimens (see Fig. 19). The tensile and short-beam strengths of hand lay-up specimens were similar to those of non-alkali-washed. Comparing Fig. 20 and 21, we see that the alkali-washed JFRP was especially well impregnated. For alkali-washed JFRP the silane coupling agent can improve the interfacial adhesion from covalent bond between the fiber and the matrix.

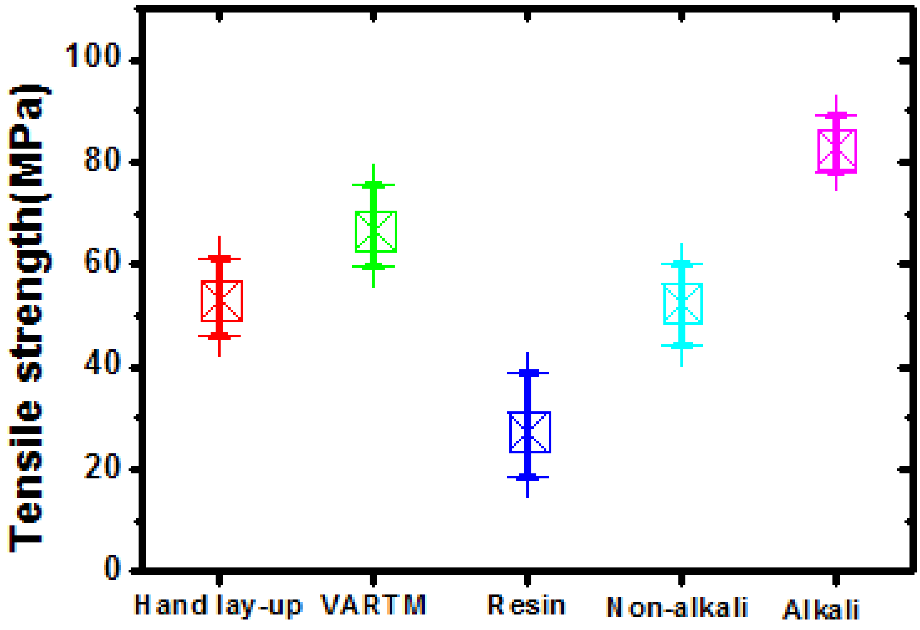


(a) Results of tensile test

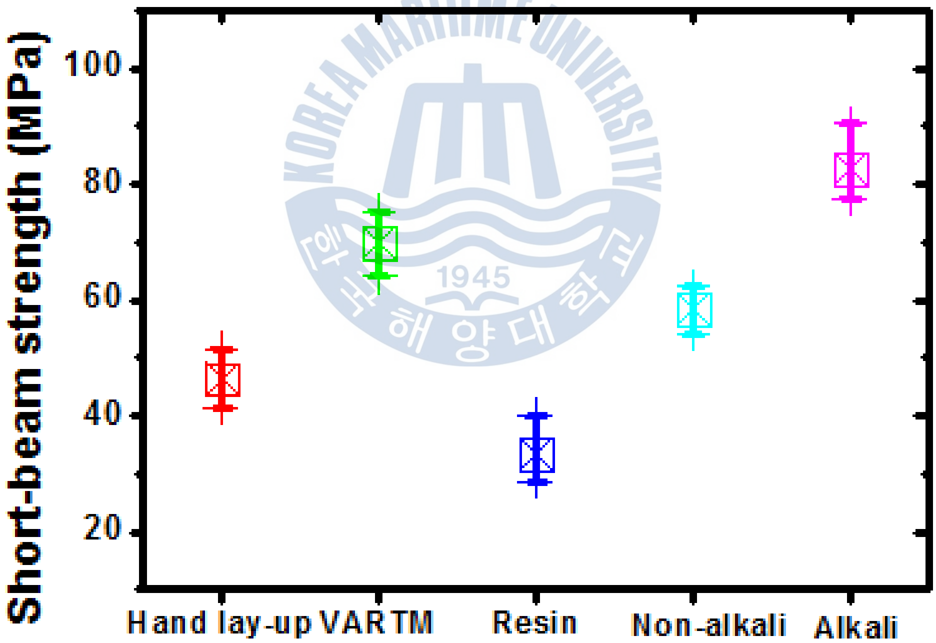


(b) Results of short-beam test

Fig. 18 Images of the result of each test



(a) Results of tensile test



(b) Results of short-beam test

Fig. 19 Comparison of the strength for each process

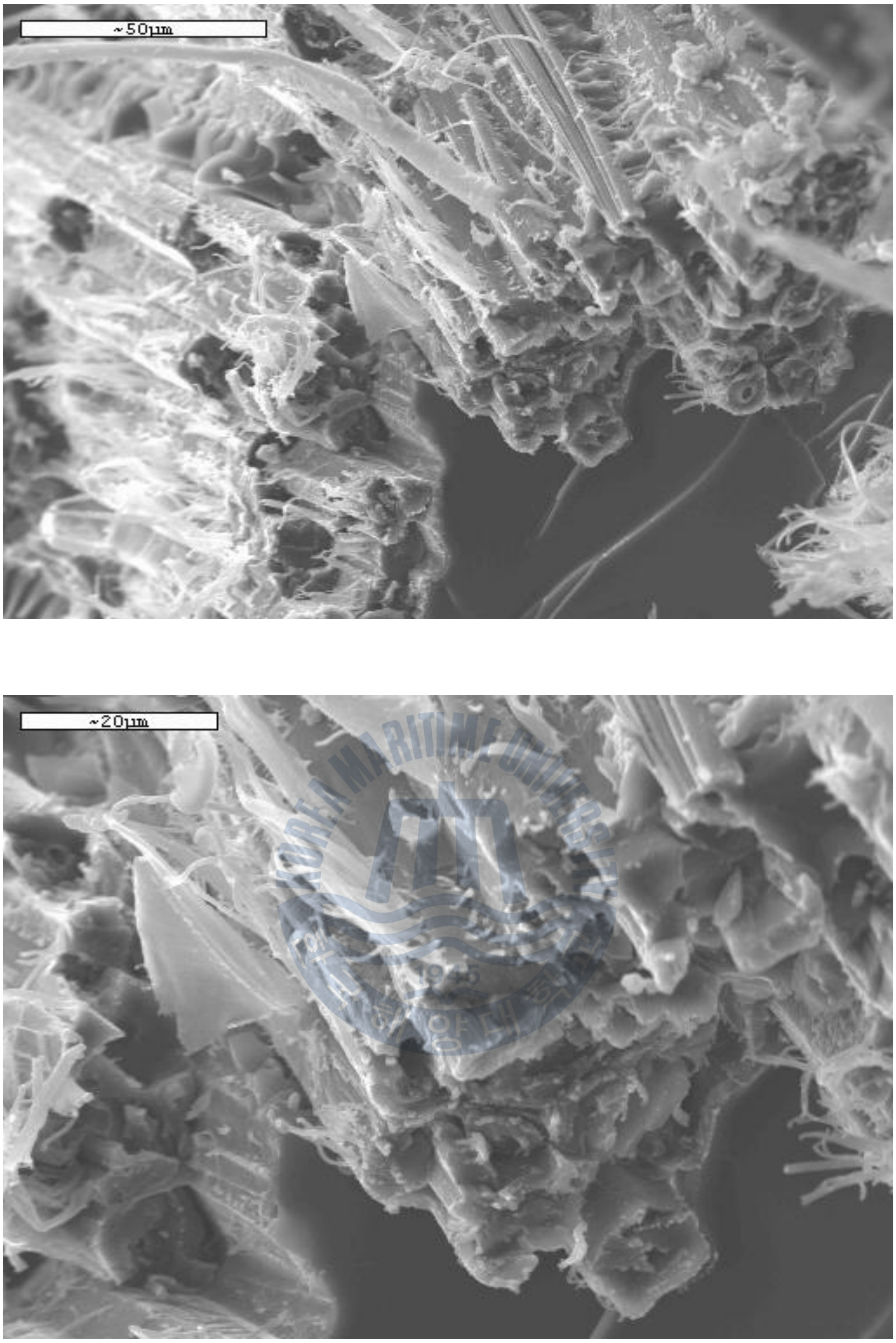
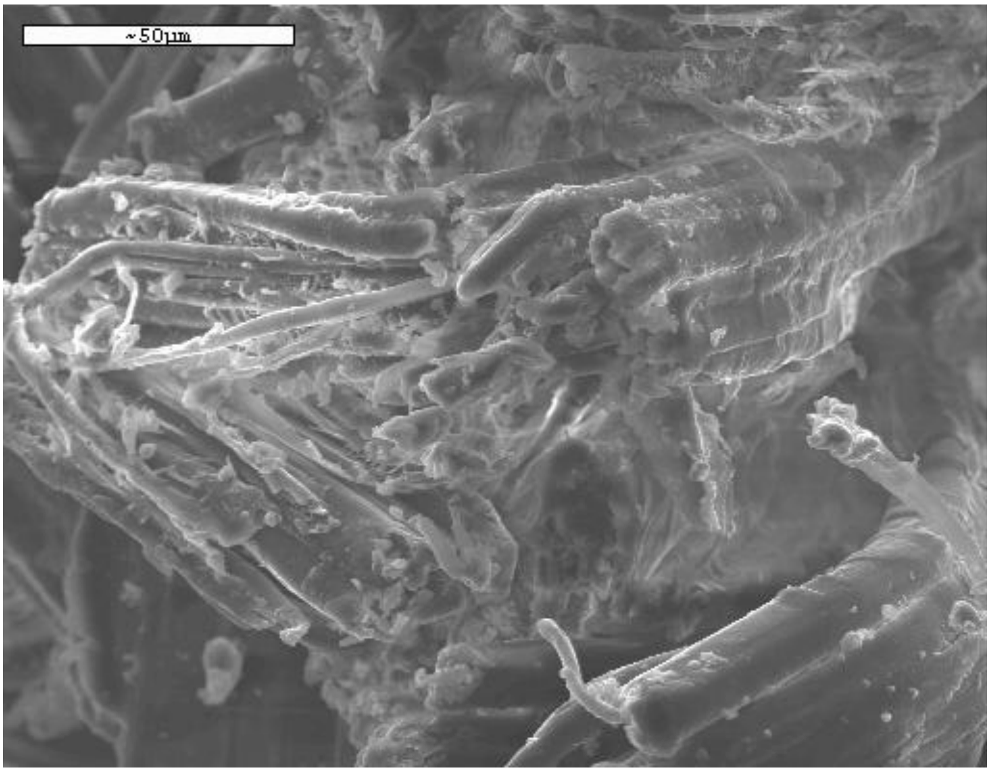


Fig. 20 Images of non-alkali-washed JFRP before the silane coupling agent



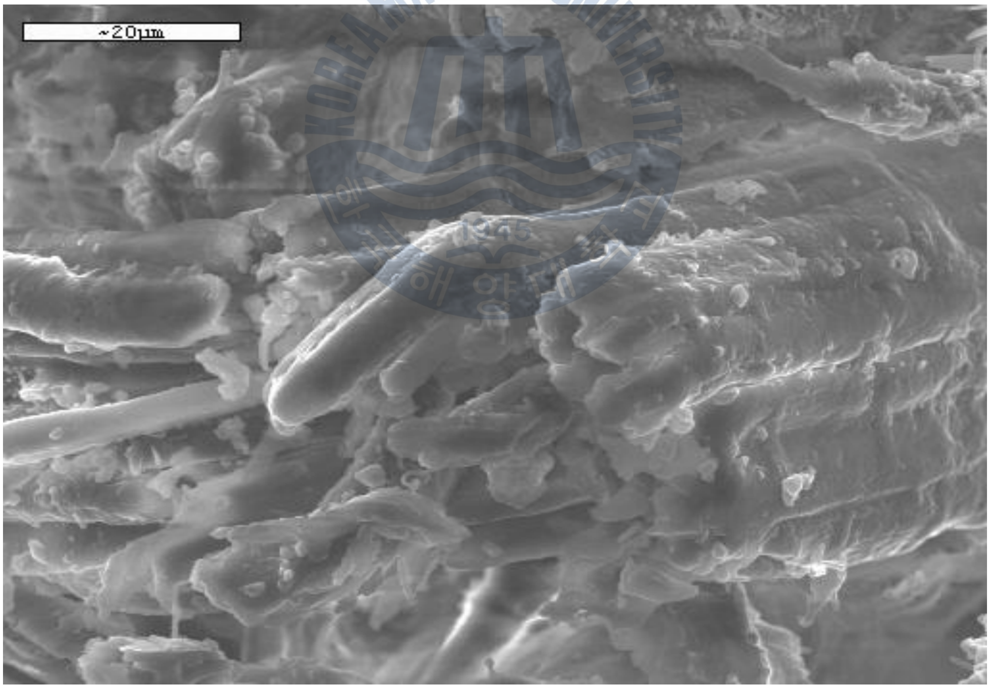


Fig. 21 Images of alkali-washed JFRP before using silane coupling agent

4-3 The volume fraction of jute fiber

Table 4 shows the volume-fraction results measured by each method. The volume fraction values differ depending on the methods used.

The volume fraction of VARTM is higher than that of hand lay-up, because of the removal of air in the former production process. Non-alkali-washed specimens had a lower volume fraction than alkali-washed specimens, but the difference was slight. In conclusion, the volume fraction of jute in alkali-washed samples with silane coupling was the highest. This confirmed that alkali-washed JFRP with silane coupling agent had the strongest mechanical properties.

Table 4 The volume fraction of the jute fiber

Methods	The volume fraction of jute fiber
Hand lay-up	48.58%
VARTM	56.63%
Non-alkali-washed before using silane coupling agent	56.16
Alkali-washed before using silane coupling agent	57.12

Chapter 5 Conclusion

Jute composites were successfully developed in this research. The mechanical properties (tensile, short-beam) of composites produced according to various methods were studied and are discussed here. Variation depending on the alkali treatment with the coupling agent of jute fiber was also studied.

[1] Through comparison hand lay-up and VARTM production methods were respectively evaluated. On average, the two methods showed a 15 MPa difference in tensile strength and 24 MPa difference in short-beam strength. Economically, jute fiber can be made by VARTM more efficiently than by hand lay-up. In short, the comparison of VARTM with hand lay-up for jute composite processing showed that the tensile and short–beam strength of VARTM specimens was greater than that of hand lay-up specimens.

[2] Alkali-washed JFRP using a coupling agent showed a higher strength than non-alkali-washed samples. In this comparison, the alkali-washed JFRP showed a 27 MPa difference in tensile test and a 15 MPa difference in short–beam test. Non-alkali-washed JFRP shows results lower than jute-only samples made by VARTM, with a 28 MPa difference. In short, specimens made by VARTM process and washed in alkali before using a coupling agent, were the strongest of all.

[3] Observed by the SEM and the optical microscope, JFRP made by hand lay-up showed more defects than VARTM. Put simply, VARTM was shown to be superior to hand lay-up as a manufacturing process. The specimens made by hand lay-up, VARTM and with alkali-washed versus non-alkali-washed before using silane coupling agent, were examined for the interfacial adhesion from the covalent bond between the fiber and the matrix. It was found that specimens washed in alkali before using a coupling agent, had stronger mechanical properties than the others.

[4] The tensile and short-beam strengths of jute composites were affected by resin weight percentage. The lower resin weight jute composites processed by VARTM was stronger than those processed by hand lay-up. The concentration of jute fiber made by hand lay-up was also lower than that made by VARTM. Although the volume fraction of the alkali-washed JFRP showed few changes, it was a little higher than samples with

no coupling agent. High fiber loading was possible with a strong material. In this way, advanced JFRP can be made.

Overall, good quality composites with very acceptable specific properties can be formed from jute and polyester resins. Thus, this valuable fiber can have considerable use in this country. And as a future development from the paper, we should aim to make higher strength materials.

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