#### Research Article

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# Effect of the manufacturing process on the equivalency qualification of glass fiber reinforced polymer

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Abstract: Glass Fiber Reinforced Polymer (GFRP) is widely used as aerospace material requiring high specific strength, specific stiffness, and excellent mechanical and chemical properties. To apply the already approved composite materials to other processes, an equivalency test that compares the mechanical properties of the composite materials based on the database is required. For the successful completion of the equivalency test, it is important to control the factors affecting the mechanical properties. The resin content and density of the specimens are manufactured differently according to the process. The effect of these factors on the change of mechanical properties required for equivalency qualification has not been sufficiently reported. In this study, an equivalency test was performed on the GFRP applied to the aircraft radome based on the procedure of the equivalency test and acceptance test proposed by the National Center for Advanced Materials Performance. The causes of problems occurring between equivalency tests were analyzed. It was confirmed that the resin content, density, and voids of the specimen affect the mechanical properties. As the resin content decreases, the density and voids were controlled, and it was confirmed that the average strength and modulus increase by 13.12 and 6.78%, respectively. The equivalency qualification was completed by applying an improved process in which these factors were controlled.

**Keywords:** glass fiber reinforced polymer, equivalency test, resin content, void, mechanical properties

## 1 Introduction

A composite is a material that has new properties by mixing two or more substances with different properties [1–3]. In general, it is composed of reinforcement and a matrix, and the properties vary depending on the type of reinforcement and the matrix. Composites in which strong fibers and resins are mixed, such as Glass Fiber Reinforced Polymer (GFRP) and Carbon Fiber Reinforced Polymer (CFRP), have high specific strength and specific stiffness. In addition, it has the advantages of lightweight, corrosion resistance, and design of mechanical properties of the structure. Due to these advantages, it is being applied to aircraft requiring excellent mechanical and chemical properties.

When manufacturing aviation parts using composite materials, re-qualification is required to utilize them for the manufacture of new aircraft parts, since the manufacturing process, material rot, and working environment are different even if they are the same as the previously certified materials. Aircraft re-qualification was performed by each company, and there was a disadvantage that it takes a lot of time and money. To supplement this, NASA/ FAA developed the Advanced General Aviation Transport Experiments (AGATE) program in 1995 to reduce the number of tests and applied a quality qualification process that satisfies statistical criteria and that can derive acceptable values based on the shared composite material property database [4]. Recently, it has evolved from the AGATE program to National Center for Advanced Materials Performance (NCAMP). Based on the database obtained from the AGATE program, it was commonly applied to the qualification of general-purpose aircraft, transport-class aircraft,

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and other aircraft parts. Therefore, material property tests were developed to present material qualification and acceptance procedures. In order to apply the already approved composite materials to other processes, it is an equivalency test to compare the properties of the composite materials based on the database [5].

The materials applied to the radome for aircraft should have stable electromagnetic properties for radar waves and should have excellent properties against environmental conditions and bird strike. Considering the electromagnetic and mechanical properties, it is known that GFRP is used [6,7]. In this study as well, an equivalency qualification test is necessary for material acceptance in a short period of time for the application of certified materials.

According to previous findings, the physical properties of fiber-reinforced composites are greatly affected by the properties of the reinforcement and matrix; therefore, many studies have been conducted on the effect of mechanical strength and voids [8-10]. It has been reported that the mechanical strength of composites increases with increasing fiber content and voids decrease with increasing fiber content [11-15]. However, several variables can occur even in the manufacturing process of a composite material of the same composition. Resin content varies depending on the manufacturing process and affects density and void formation. Specifically, the occurrence of voids affects the mechanical properties of the equivalency test, and the formation of voids in the composite manufactured through the bagging process may vary depending on the compaction holding time and moisture absorption during the hand layup process of the prepreg [16]. Although many studies have been done on the removal and moving of the formed voids [17-20], the effect of the change in the resin content and the removal of the voids according to the control of the bagging process in the case of composite materials is not addressed. Furthermore, there are only a few studies done about the effect of microstructural changes such as resin content and formation of voids, which may vary depending on the manufacturing process in composite produced by prepregs with the same fiber and resin content of the raw material, on mechanical properties of composites.

In this study, the equivalency test was performed on the GFRP utilized in the aircraft radome based on the procedure of the equivalency test and acceptance test proposed by NCAMP. Among the equivalency test conditions, tensile and compression tests were performed, and the first equivalency qualification was performed. The causes of problems occurring between the tests were analyzed, and the second equivalency qualification was completed by applying an improved manufacturing process. The resin content, density, and voids were analyzed for specimens manufactured by different processes, and the effect of the manufacturing process on the equivalency qualification test of composite materials was compared and observed.

# 2 Experimental procedure

The composite material used in this study is glass epoxy fabric prepreg, and the reinforcing agent is glass fiber with a 7781 style 8 harness satin structure. Before curing, the resin content and fiber volume fraction were 36 and 46%, respectively, and the CPT (Cured Ply Thickness) per ply was 0.24 mm.

The mechanical test conditions are shown in Table 1, and the specimen manufacture and quantity were

Table 1:	Equivale	ency test	conditions
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Test conditions	Environmental conditions	Test specification	Number of specimens	
			Case 1	Case 2
0° Tensile strength, modulus	RTD <sup>a</sup>	ASTM D 3039	2 × 4 <sup>c</sup>	_
<del>-</del>	ETW <sup>b</sup>		2 × 4	2 × 4
90° Tensile strength, modulus	RTD		2 × 4	_
-	ETW		2 × 4	2 × 4
0° Compressive strength, modulus	RTD	ASTM D 6641	2 × 4	2 × 4
	ETW		2 × 4	2 × 4
90° Compressive strength, modulus	RTD		2 × 4	2 × 4
	ETW		2 × 4	2 × 4

<sup>&</sup>lt;sup>a</sup> RTD: Room temperature dry (test temperature =  $25 \pm 5^{\circ}$ C, moisture content = as-fabricated).

<sup>&</sup>lt;sup>b</sup> ETW: Elevated temperature wet (test temperature =  $100 \pm 5$ °C, moisture content = ASTM D 5229).

 $<sup>^{\</sup>mbox{\tiny c}}$  Panel  $\times$  Specimens.

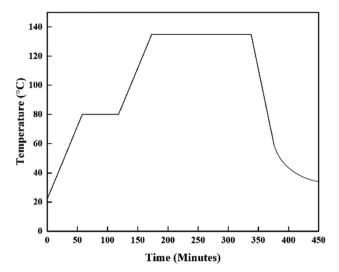


Figure 1: Curing cycles in the specimen manufacturing process for equivalency testing.

determined as a reduced sample used to calculate the allowable b-basis value in accordance with DOT/FAA/ AR-03-19 [5]. For the equivalency test, two panels were manufactured from 1 batch, and four specimens were extracted per panel, and a total of eight specimens per condition were tested. For the manufacturing process of the specimens, a vacuum bagging process through general hand lay-up was applied. In Case 1 process, a nonperforated release film was applied, and the cure cycle is shown in Figure 1. The heating rate and the cooling rate were 1 and 2 °C/min, respectively, and were holding at 80 and 135°C for 60 and 135 min, respectively. During the curing process, the vacuum pressure was held at -28 inHg. In Case 2 process, a perforated release film was applied, and the cure cycle was the same as in Case 1. During the curing process, vacuum pressure and autoclave pressure were held at -28 inHg and 15 Psi, respectively. The shape of the tensile and compression tests was manufactured according to ASTM D 3039 and ASTM D 6641 [21,22], respectively, as shown in Figure 2. The layup of specimens was designed as 8 ply. The moisture absorption test of the specimen to be applied to the Elevated Temperature Wet (ETW) test was performed in accordance with DOT/FAA/AR-03-19 and ASTM D 5229 [5,23]. For the moisture absorption test, the specimens were exposed in a chamber; temperature and humidity were controlled at  $62.5 \pm 5^{\circ}$ C and  $85 \pm 5^{\circ}$ K, respectively. The criterion for deciding the state in which absorption of the specimen is completed should be to ensure that the weight change is within 0.05%. The weight change was calculated as follows:

$$M \ (\%) = \frac{W_i - W_o}{W_o} \times 100,$$

where M is the moisture content (%),  $W_i$  is the current specimen mass (g),  $W_o$  is the oven-dry specimen mass (g).

According to the above formula, the weight change rate was calculated by measuring the weight of the specimen at 7-day intervals, and it was determined whether moisture absorption was completed. As shown in Figure 3, it was confirmed that the weight change rate of the specimen used in this study was secured within 0.05% after about 6 weeks (42 days) elapsed.

UTM (Universal Testing Machine, Instron 5985) was used as the test equipment, and room temperature and ETW conditions were performed at 100°C. The test speed was 2 mm/min, and the strain was measured by attaching a uniaxial strain gauge to the center of the specimens in the direction of loading. Density and resin content of the specimens were measured according to ASTM D 792 and ASTM D 3171 [24,25], respectively.

Equivalency test in reference to the standard test was performed for the mechanical properties of 16 conditions; thus, Pass/Fail was identified based on statistical analysis using Hypothesis Testing for Equivalence provided by NAIR's NCAMP. The equivalence test is performed statistically with a confidence level of 95%. The main acceptance qualification of the equivalency test is that the strength compares the average value with the minimum

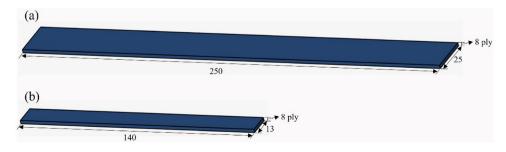


Figure 2: Specimen design: (a) tensile and (b) compressive test (Si unit: mm).

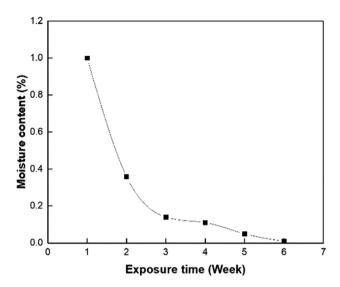


Figure 3: Moisture absorption behavior of the ETW specimens.

value, and the modulus only considers the average value. The pass standard must have a statistically 95% confidence level in strength and young's modulus compared to the standard test.

## 3 Results and discussion

The first and second equivalency tests were performed by applying the specimens manufactured in Case 1 and Case 2 processes, respectively, and the representative failure modes identified in each test condition are shown in Figure 4. The failure modes of the tensile and compression

**Table 2:** Results of the first equivalency test applying the Case 1 process

Test conditions		RTD	ETW
0° Tensile	Strength	Pass	Fail
	Modulus	Pass	Pass
90° Tensile	Strength	Pass	Fail
	Modulus	Pass	Fail
0° Compression	Strength	Pass	Fail
	Modulus	Fail	Pass
90° Compression	Strength	Pass	Fail
	Modulus	Fail	Fail

tests were confirmed with reference to ASTM 3039 and ASTM 6641, respectively. As a result, the tensile test of the 0° specimens confirmed the failure modes of Angled Gage Middle fractured at the intermediate strain gage, and Lateral Gage Middle fractured at a region slightly deviating from the strain gage. In the tensile test of 90°, the strength of the fiber to withstand stress is lower than that in the 0°; thus, two failure modes were confirmed such as Lateral At grip/tab top fractured in the grip area and long Splitting Gage Middle fractured in the stress direction. In the case of the compression test, the failure mode of the fractured through-thickness at grip/tab top was confirmed since the stress was transmitted in the thickness direction due to buckling of the fiber regardless of the direction of the specimens. These are all common acceptable failure modes; therefore, it was confirmed that no problems occurred during the test.

Table 2 shows the results of the first equivalency test of the specimens manufactured by applying the Case 1 process. Figure 5 is the result of comparing the standard

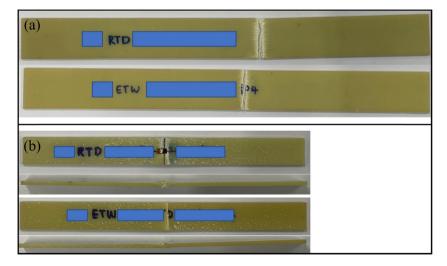


Figure 4: Failure mode of (a) tensile and (b) compression test specimens.

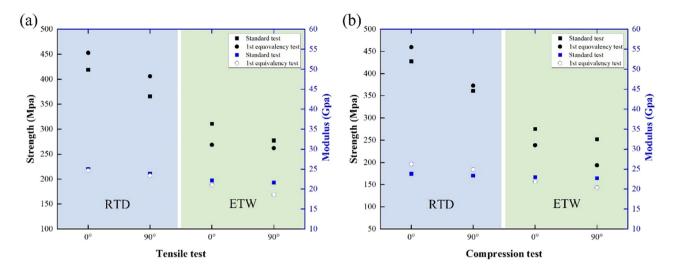


Figure 5: The result of the first equivalency test: (a) tensile and (b) compression test.

test and the equivalence test result, and it was confirmed that the tensile test and compressive strength under the Room Temperature Dry (RTD) condition passed the equivalency qualification standard. If the RTD area in Figure 5(a) and (b) is considered, it has passed by securing properties higher than the allowable value of the physical properties of the standard test. If the RTD area in Figure 5(a) and (b) is checked, it has passed by securing a statistically 95% confidence level compared with the physical properties value of the standard test. The cause of the failure of the compressive young's modulus of the RTD condition was that it was confirmed as a result of deviating from the standard value by securing a value that was 6 to 10% higher than that of the standard test (Figure 5(b)). It is assumed that this deviation occurred due to the change in the epoxy properties according to the material rot. A reproducibility experiment was conducted to prove this point. On the other hand, it was confirmed that most of the results in the ETW condition test failed. As in the ETW area of Figure 5(a) and (b), it is the result of not securing a statistical confidence level when

compared with the physical properties value of the standard test.

Figures 6 and 7 show the effect of the specimen according to the type of release film and different cure pressures. Figure 6(a) shows a cross-section of the first equivalency test specimens to which a non-perforated release film was applied. Based on the observations, when a non-perforated release film is applied, the flow of the resin does not pass through the film layer during the curing process but flows inside causing voids in the specimens. As the voids occur, the thickness and crosssectional area of the specimen increase. The voids are generated in the matrix and the volume of the specimens increases. In general, the internal voids reduce the bonding force between the reinforcement and the matrix and, consequently, affect the transfer of stress, resulting in a lower strength [26,27]. Furthermore, it is known that the amount of moisture absorption increases due to the generated voids [28], which affects the mechanical properties during the ETW condition test, thus has a high probability of failure.

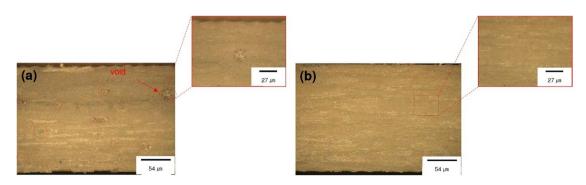


Figure 6: Comparison of specimen cross-sections according to (a) Case 1 and (b) Case 2.

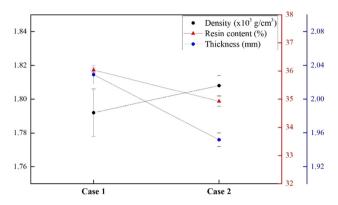


Figure 7: Specimen density, resin content, and thickness according to the process.

To control the internal voids, a perforated release film was applied, and the cure pressure was increased to manufacture the specimens. The cross-section of the specimen produced by the improved manufacturing process is shown in Figure 6(b). They confirmed that no voids were observed overall in the internal image of the improved specimens.

Figure 7 shows the results of measured density, resin content, and thickness of the specimens manufactured in each process. It was confirmed that the Case 1 specimen had increased resin content as well as thickness and decreased density compared to Case 2. The thickness of the specimen is 1.92 mm when calculated based on CPT. Based on this, the thickness increased by 5.37% in Case 1 and 1.63% in Case 2. Resin content and thickness have a proportional relationship, which affects the existence of voids and density of the specimens. This shows a similar trend with the effect of voids as illustrated in Figure 6(a). Based on the results of Figures 5–7 and Table 2, the

analysis showed that the cause of the failure of the first equivalency test was the impact of the resin content, voids, and density of the specimens. Even if the raw material of the specimen is the same, the flowability of the resin is affected according to the selection of subsidiary materials in the bagging process, and the state of the specimen may be different depending on the pressure applied to the specimen in the curing process [29–32]. The selection of subsidiary materials can be attributed to the resin content, and an assumption was made that cure pressure affects the voids and density of the specimens [14,27].

The second equivalency test was performed except for the tensile test under the RTD condition since successful results were obtained in the first equivalency test. Specimens for second equivalency qualification were produced following the improved Case 2 process. The results of the second equivalency test are shown in Figure 8 and Table 3. As shown in Figure 8, the second equivalency test

**Table 3:** Results of second equivalency test applying the Case 2 process

Test conditions		RTD	ETW
0° Tensile	Strength	Pass	Pass
	Modulus	Pass	Pass
90° Tensile	Strength	Pass	Pass
	Modulus	Pass	Pass
0° Compression	Strength	Pass	Pass
	Modulus	Pass	Pass
90° Compression	Strength	Pass	Fail
	Modulus	Pass	Pass

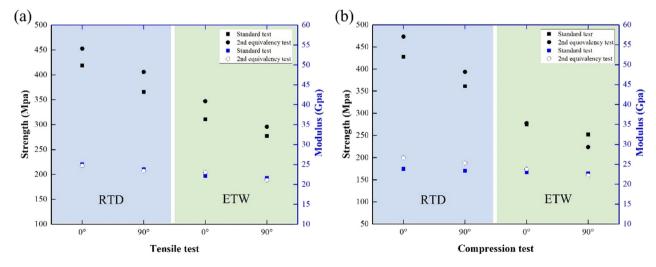


Figure 8: The result of second equivalency test: (a) tensile and (b) compression test.

Table 4: Comparison of physical properties of the results of the first and second equivalency tests

Test conditions		Comparison of physical properties under RTD condition (%)	Comparison of physical properties under ETW condition (%)
0° Tensile	Strength	_	25.09
	Modulus	_	9.08
90° Tensile	Strength	_	11.96
	Modulus	_	11.58
0° Compression	Strength	3.24	14.05
	Modulus	1.56	8.28
90° Compression	Strength	12.43	11.95
	Modulus	1.72	8.44

secured a statistically 95% confidence level compared to the physical properties value of the standard test, and it was confirmed that most of the results of the second equivalency test, based on the improved process, were successful. The compressive young's modulus reproducibility test results under the RTD conditions presented in Figure 5 and Table 2 exhibited the same trend as the results of the first equivalency test. As a result, it passed through a readjustment of the allowable value. The 90° compressive strength of the ETW condition failed, and as shown in Figure 8(b), it was confirmed that the physical property value was secured by ~11% lower than the allowable values of the physical properties of the standard test. The failure is because the moisture absorption condition is different from that used for standard specimens. The moisture absorption conditions in this study were carried out in the method of ASTM D 5229 for 42 days with reference to the DOT/FAA/AR-03/19 [5] document, and the moisture absorption conditions of the standard test were conducted for 10 days in the method of section 507.5 of MIL-STD-810G [33]. As the period of exposure to moisture of the specimens increases, the difference in physical properties appears, and the period of exposure to moisture in this study is a poor condition [34]. In addition, the 90° specimens have a lower fiber volume fraction compared to the 0° specimens [35], indicating that it is more sensitive to moisture absorption conditions. As a result, due to the difference in moisture absorption conditions of the specimens, the standard test and the equivalency test of the produced specimens were data that cannot be compared, and re-adjustment of the allowable value was required based on the result of the manufactured specimens. If the moisture absorption condition of the ETW specimens was settled according to MIL-STD-810G [33] rather than the ASTM standard, it is expected that satisfactory results will be obtained for equivalency qualification.

The results of the second equivalency test conducted by applying the improved process were confirmed to

secure high physical property values under all test conditions compared with the first equivalency results as demonstrated in Table 4. As a result, the application of the Case 2 process controlled the internal voids better compared to the Case 1 process; also, it can be estimated that the controlled voids increase the mechanical properties. In addition, it was analyzed that the effect of moisture absorption conditions was less deteriorated than that of the specimens with voids by controlling the internal voids. During the manufacturing process, the flow of resin determines the resin content of the composite material; hence, the voids, thickness, and density of the specimens are manufactured differently. This may cause differences in mechanical properties and may be an important factor in deciding whether the equivalency is obtained or not.

### 4 Conclusion

For GFRP applications in aircraft radome, the results of the equivalency test and acceptance test suggested by NCAMP were presented, and the causes of problems occurring between tests were analyzed. The equivalency test was completed by applying the improved manufacturing process; as a result, the values of average strength and young's modulus were increased by 13.12 and 6.78%, respectively. It was confirmed that the control of the resin content, density, and voids generated during the specimens manufacturing process affects the mechanical properties, and the existence of voids increased the amount of moisture absorbed even during the moisture absorption process. Based on these results, the fine control of the process or the selection of subsidiary materials affects the state of the material; hence, the mechanical properties of the specimens can be greatly improved by the results.

**Conflict of interest:** The authors state no conflict of interest.

**Data availability statement:** All data generated or analyzed during this study are included in this published article.

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