Study on voids of epoxy matrix composites sandwich structure parts

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Abstract. Void is the most common tiny defect of composite materials. Porosity is closely related to composite structure property. The voids forming behaviour in the composites sandwich structural parts with the carbon fiber reinforced epoxy resin skins was researched by adjusting the manufacturing process parameters. The composites laminate with different porosities were prepared with the different process parameter. The ultrasonic non-destructive measurement method for the porosity was developed and verified through microscopic examination. The analysis results show that compaction pressure during the manufacturing process had influence on the porosity in the laminate area. Increasing the compaction pressure and compaction time will reduce the porosity of the laminates. The bond-line between honeycomb core and carbon fiber reinforced epoxy resin skins were also analyzed through microscopic examination. The mechanical properties of sandwich structure composites were studied. The optimization process parameters and porosity ultrasonic measurement method for composites sandwich structure have been applied to the production of the composite parts.

Keywords: Composites processing, Void, Microscopic analysis, Composites mechanical properties, Ultrasonic inspection

1. Introduction

Carbon fiber reinforced resin matrix composites (CFRP) is widely used in the aircraft because of its excellent specific strength, specific modulus and comprehensive properties, they can effectively reduce the weight of the aircraft, improve the load capacity of aircraft [1]. However, the void defects produced during the manufacturing in composites are receiving greater attention. Porosity is one of the most common defects in composite structures [2]. Voids are the critical problem for the polymer composites and are harmful to the mechanical properties of composites [3-4]. Therefore, it is significant to study the influence of process parameters on the microstructure and mechanical properties to analysis the void content and distribution for the application of composites. A lot of research have been done on improving the voids content by adjust the autoclave process [5]. Many researches have been focused on improving the voids content for composite laminate structure [6-7]. For the epoxy matrix composites honeycomb core sandwich structure can greatly reduce the weight of the composite structure and have good mechanical properties compare to the laminate, honeycomb core structure has already been widely used in civil aircraft structure [8]. This study by adjusting the lay-up manufacturing process parameters, analysis the voids formation and distribution in epoxy matrix composites honeycomb core sandwich structure. An optimization process parameters and porosity ultrasonic measurement method for composites sandwich structure have been established and applied to the production.

2. Experiment procedure

2.1. Materials and Composite preparation
HexPly® M21E/IMA carbon fiber reinforced epoxy prepreg, ECK4.0-96 Honeycomb core and adhesive film FM73 were used in the research. M21E/IMA was unidirectional tape.

The composite parts were fabricated by sandwich structure parts. The sandwich part studied in this work was co-cured with upper skin, adhesive film, honeycomb core, adhesive film and lower skin. The Upper skin stacking sequences studied in this work was:[45 135 (0)4 (45) 4 90 (135) 4 0 (45) 3 90 (135) 3 90 (45) 4 (0) 4 135 45]. The Lower skin stacking sequences studied in this work was:[45135(0)3 (45)3 90 (135) 3 (0)2 45(90) 3 (135) 3 0 (90) 3 (45) 2 (135) 2 90 0 (135) 2 (45) 2 (90) 2 0 135 (90) 3 (45) 3 (0) 3 (135) 3 90(45) 3 (0)3 13545]. The lower skin, upper skin and laminate area fiber volume content (FVC) tested in according to EN2564 type B should be around 59% ± 4%.

The lay-up process, the composite parts with different void contents were produced by using different compaction vacuum, the compaction vacuum were set as 30% and 100% of full vacuum respectively. The composite panels with different void contents were produced with a vacuum bag and autoclave using the same cure cycle. The extent of cure should be ＞95% determined by DSC. After machining, microscopic examination and metallographic microscopic analysis were done. Test samples were trimmed from each of those areas.

2.2. Microscopic analysis
For the microscopic analysis of CFRP, the micro-sections were previously embedded in a resin (cold mounting with an epoxy resin). Then embedded micro-sections were submitted to an adequate preparation by grinding & polishing. Grinding sequence begins with removal of material by disk grinding with SiC papers at high speed (300 rpm) using 180-grit silicon carbide paper and abundant running water. Subsequent grinding proceeds through 500, 1200 and 4000 grit papers. Polishing sequence begins with removal of scratches induced by grinding with clothes at low speeds (150 rpm), using 6μm diamond paste and abundant lubricant oil. Subsequent polishing proceeds through 3 μm and 1 μm diamond paste. Between different polishing clothes, all the samples have been cleaned using water, and observed under stereomicroscope(ZEISS “STEMI 2000-C”) to check good progression of polishing. Microscopic analysis on each field is analyzed under optical microscope in order to detect and characterize possible defects over micro-cut section, using a low magnification. When it is found a cracked area, this will be focused at higher magnification.

The micro sections of CFRP were prepared in accordance with ASTM E3-11.The microscopic examination was carried out with small magnification, and the void content was determined using Automatic Image Analysis with a larger magnification.

The automatic image analysis system consisting of the following components must be used: A high quality reflected light microscope equipped with bright-field objectives. A programmable automatic stage control the movement of the specimen in x and y direction. An automatic focus system can control the z-direction of the stage. A camera with adequate resolution and sensitivity, and a computer with an automatic image analyzer were used to analyze and store the measurement data.

2.3. Mechanical Test and Ultrasonic Inspection
The samples corresponding to Filled Hole Compression (FHC) test results were also investigated. The layup for the FHC is $[135/45/90/135/0/45/135/0/45/90]_s$. The specimens were produced separating to the part but on the same tool and under the same vacuum bagging. FHC test specimens were manufactured with extra plate manufactured with the part in the same bagging as shown in figure 1. The specimen dimensions should be width = 36 ± 0.2 mm, length = 160 ± 1.0 mm and thickness =

2.54 mm. Bond10 glass fabric plies M21 / 120 at 45°/-45° for tabs (thickness=1.08 mm) onto the specimens with FM300. The length must be 60 mm on each side.

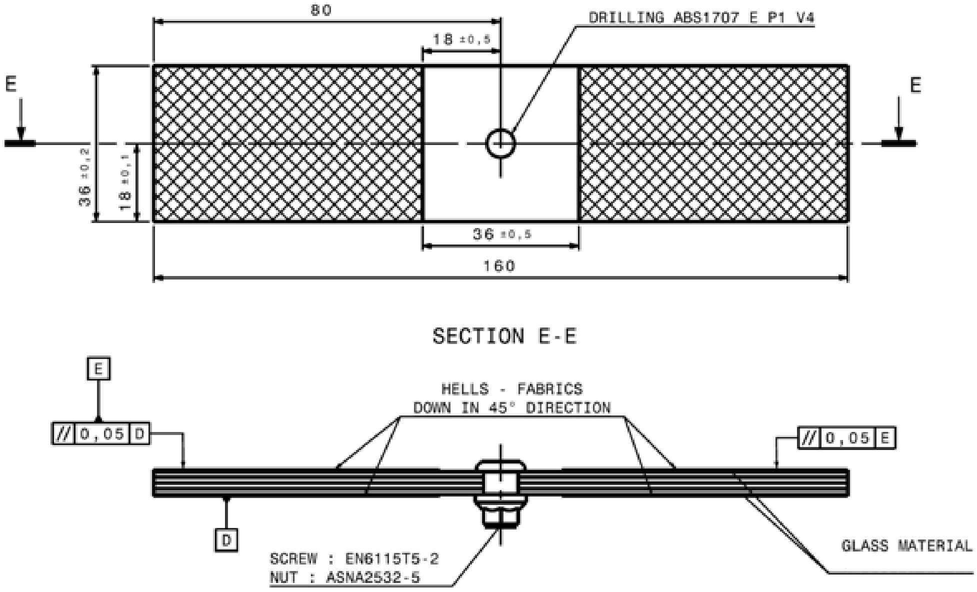

Figure 1. Filled Hole Compression specimens.

All panels were inspected with ultrasonic detector (Masterscan 380 and OminiScan MX2).

3. Results and discussion

3.1. Microscopic analysis

Figure 2 shows the photo of typical CFRP sandwich structure cross section manufactured with 30% vacuum compaction, we selected 2 area: MS-B-F-1 Verification of laminate bonding on trailing edge area, MS-B-F-2 and MS-B-F-3Verification of bonding between laminate and core ramp.

Figure 2. CFRP sandwich structure section photo.

Figure 3. Low magnification picture of MS-B-F-1.

Figure 3 shows the laminate bonding on trailing edge area cross section MS-B-F-1. In the figure1, 2, 3, 4, 5, 6 were selected and marked for the higher magnification observation in figure 4.

Figure 4(a) shows the microstructure of the laminate voids in area 1, figure 4(b) shows the detail of figure 4(a) with higher magnification. Figure 4(c) shows the laminate voids of area 3, figure 4(d) shows the detail of figure 4(c) with higher magnification. Figure 4(e) shows the detailed morphology of laminate 5. Figure 4(f) shows the detailed morphology of laminate 6.We can see the laminate presents voids in all the length of the interested area.

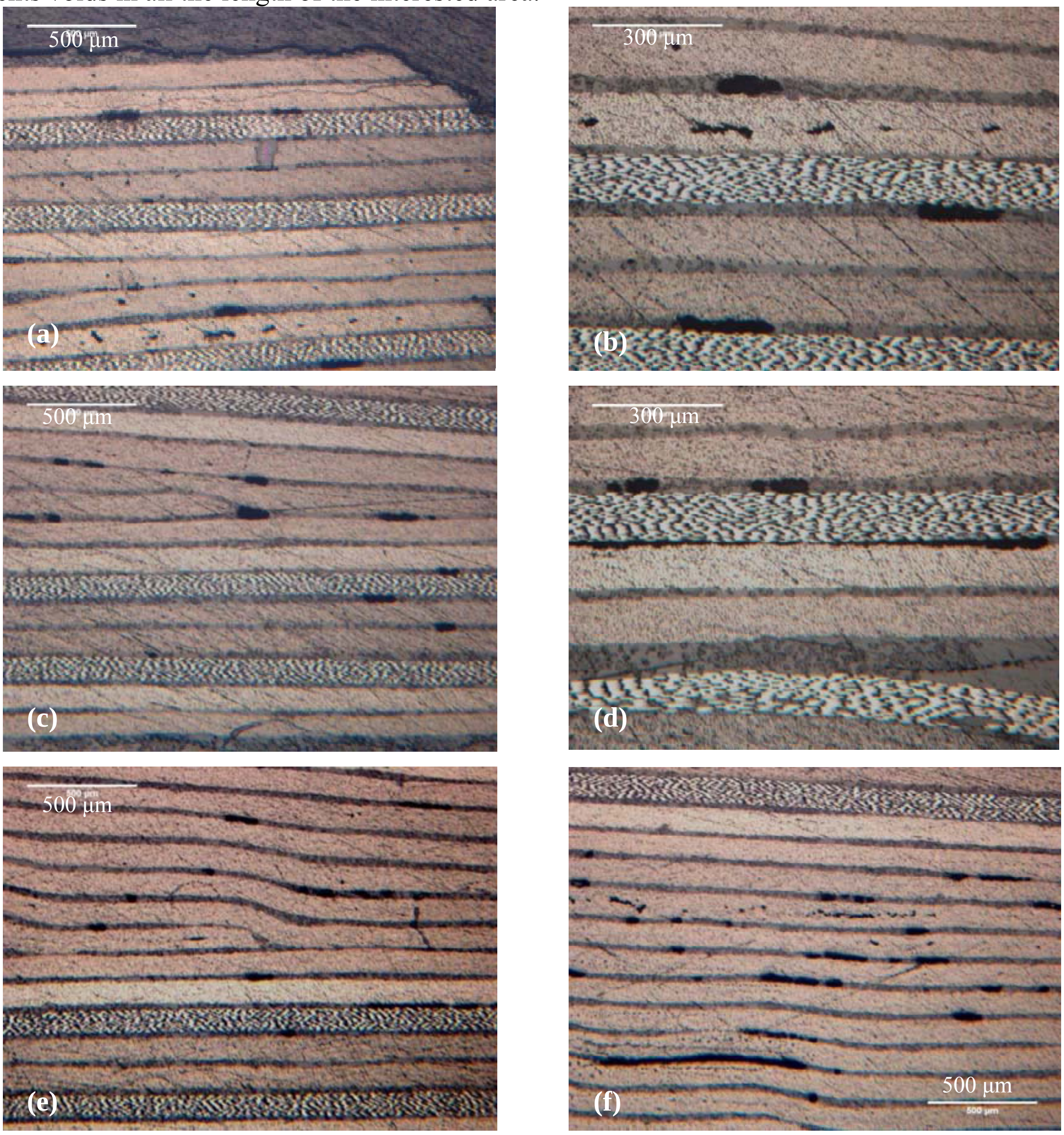

Figure 4. Microscopicphotos of MS-B-F-1.
(a)1, voids, (b)1, voids, (c)3, voids, (d)3, voids, (e)5, voids, (f)6, voids.

Figure 5 shows 12.5X of the bonding between laminate and core ramp cross section MS-B-F-3. We selected areas and marked as 1, 2, 3, 4for higher magnification observation.

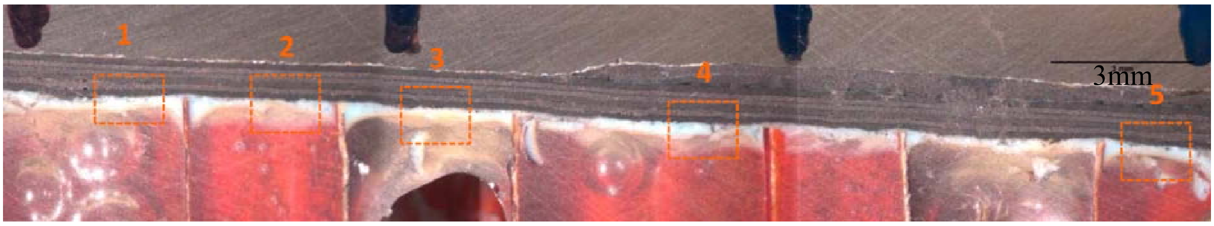

Figure 5. Low magnification picture of MS-B-F-3.

Figure 6(a) shows morphology of the bonding 1 area, the defects, voids, porosity in interface laminate and core can be observed. Figure 6(b), 6(c), 6(d) shows morphology of the bonding 2, 3, 4 area porosity presents in interface laminate and core. The existence of porosity promotes the formation and propagation of inter-laminar resin cracks.

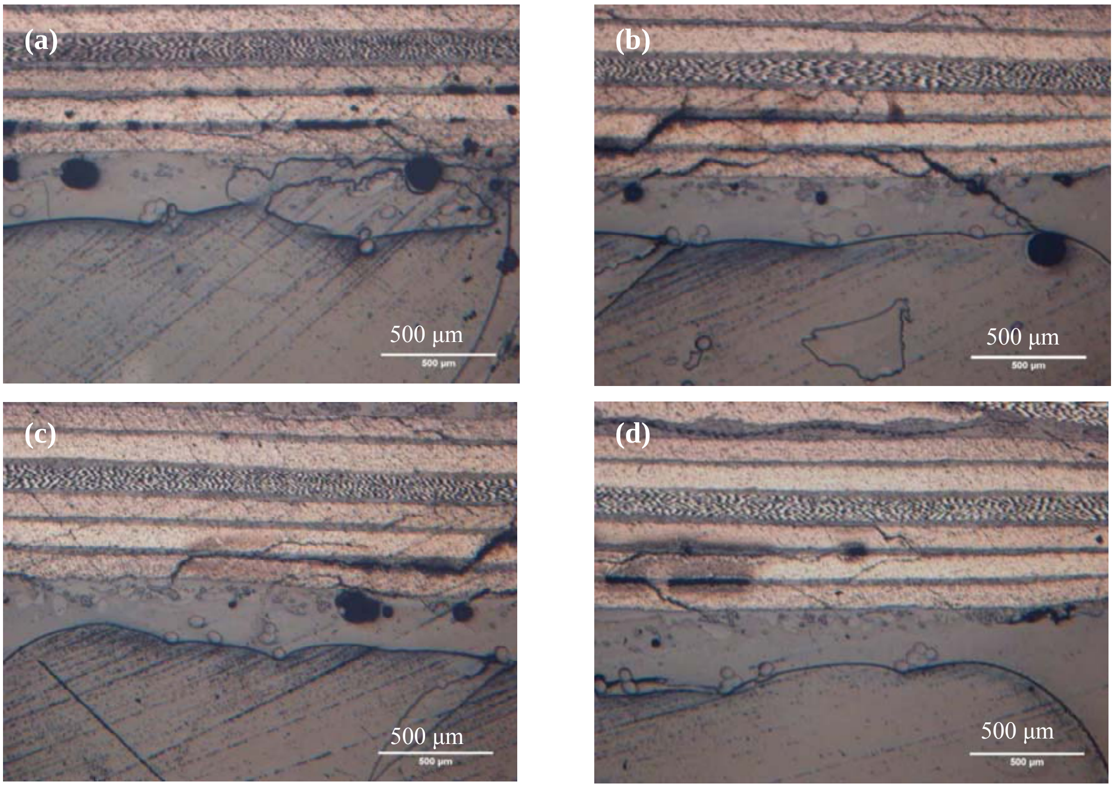

Figure 6. Microscopicphotos of MS-B-F-3.

Voids generally occur in the between layer and the junction resin/fiber. There are two main reasons for the voids formation: The air from the process of manual lay-up and the volatile component from the heat process. It is generally believed that there are three main reasons for the growth of void: (1) Diffusion of gas in the interface between the core and the resin. (2) The temperature or pressure change around the voids. (3) There is a temperature gradient in the resin that causes voids expansion. For the production and application, the most convenient method is to adjust the compaction parameters, but there is no relevant research report.

3.2. Voids Content Evaluation

In order to analyze the local porosity, the voids content was calculated by statistical method. Figure 7 and figure 8 show the typical voids content evaluation results through microscopic analysis in the samples of the 30% vacuum compaction and 100% vacuum compaction, respectively.

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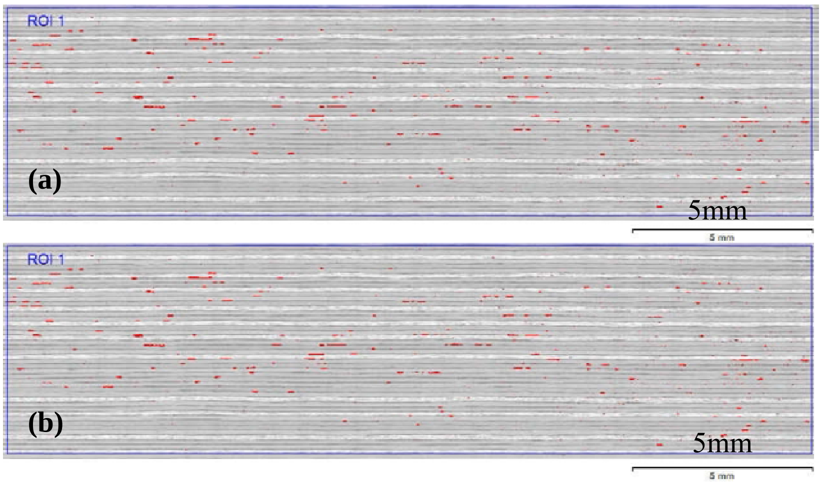
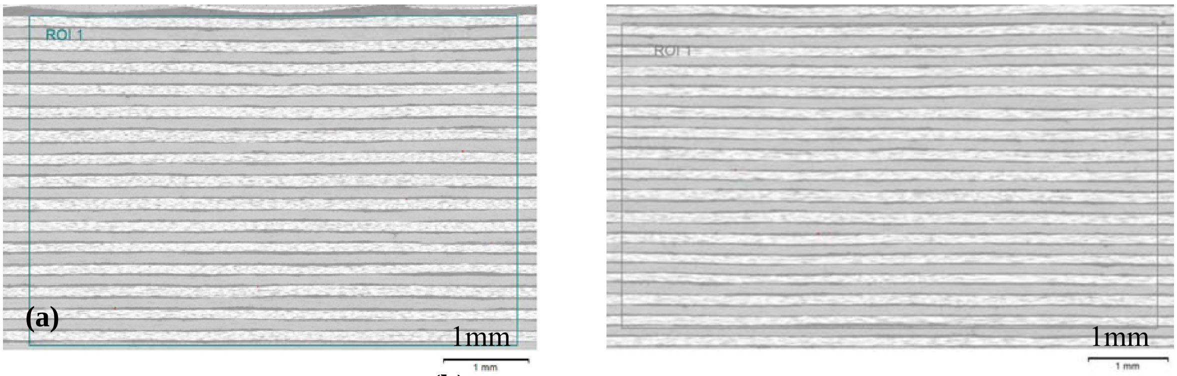


Figure 7. Voids content of 30% vacuum compaction part. (a)voids content: 0.4 %, (b)voids content: 0.4 %.



(b)

Figure 8. Voids content of 100% vacuum compaction part. (a)voids content: ＜0.1 %, (b)voids content: ＜0.1 %.

At the beginning, the air was laid up with the prepreg and the air from the volatile substance formed the air bubbles in the resin under the action of vacuum pressure. Under the action autoclave pressure, the air bubbles move upward from below, became more and more big when moving. The pressure inside the bubble is getting lower and lower. Finally, the bubbles burst discharge air and volatile content. When honeycomb core sandwich structure manufacturing in autoclave, the pressure should not as high as laminate manufacturing. The pressure inside the bubble is reduced slowly, before the resin gel, the bubbles and volatiles were not completely removed, then the voids formation. As can be seen from the figure 7, the voids are generally clustered in the middle and the upper layer of the composite structure. Increase the lay- up compaction pressure, the honeycomb core structure was not affected, but it can be as much as possible to reduce the trapped air. Finally reduce the porosity generation.

The voids content evaluated microscopic samples can be used as reference standards for ultrasonic inspection. The reference standards can be used to verify that the inspection technique for a specific configuration is consistent with the applicable classification for reporting, to check inspection program set-up, probes, accuracy and deterioration of equipment and to calibrate the whole system.

3.3. Mechanical Test

Table 1 and table 2 show FHC specimen dimensions and test results of the 30% vacuum compaction and 100% vacuum compaction respectively. The results show that the effects of compaction vacuum on FHC were small. The mechanical properties are related to the size of the voids.

Table 1. FHC specimen dimensions and test results of 30% vacuum compaction.

Specimen ID	Length (mm)	Thickness (mm)	Width (mm)	Free length (mm)	Tab length (mm)	Hole diameter (mm)	Pu (KN)	Strength(*) (MPa)
FHC-1	159.99	2.59	35.97	36.30	61.77	7.960	31.10	340.43
FHC-2	160.07	2.57	35.90	36.29	61.77	7.964	31.97	350.63
FHC-3	160.03	2.59	35.93	36.29	61.78	7.956	36.36	398.42
Mean	160.03	2.58	35.93	36.29	61.77	7.96	35.15	363.16
S.D	0.04	0.01	0.03	0.01	0.01	0.00	2.82	30.96
COV(%)	0.02	0.45	0.07	0.02	0.01	0.05	0.90	8.53

(*) Values based on nominal thickness (2.54 mm) and measured width

Table 2. FHC specimen dimensions and test results of 100% vacuum compaction.

Specimen ID	Length (mm)	Thickness (mm)	Width (mm)	Free length (mm)	Tab length (mm)	Hole diameter (mm)	Pu (KN)	Strength(*) (MPa)
FHC-1	160.05	2.59	35.98	36.39	61.81	7.936	36.17	395.74
FHC-2	160.07	2.57	36.03	36.43	61.76	7.962	36.10	394.48
FHC-3	159.97	2.58	35.99	36.45	61.73	7.959	35.58	389.13
Mean	160.03	2.58	36.00	36.42	61.77	7.95	35.95	389.19
S.D	0.05	0.01	0,03	0.03	0.04	0.01	0.32	3.48
COV(%)	0.03	0.39	0,07	0.08	0.07	0.18	0.90	0.88

(*) Values based on nominal thickness (2.54 mm) and measured width

Figure 9(a) and figure 9(b) show FHC test failure mode of the 30% vacuum compaction and 100% vacuum compaction, respectively. Form the failure mode figures of compressive strength test we can see that the failure occurred under the fastener or at the fastener edge. The matrix cracking and fiber breakage caused the failure of the samples. There was no big difference between specimens with different void content by adjusting the compaction vacuum.

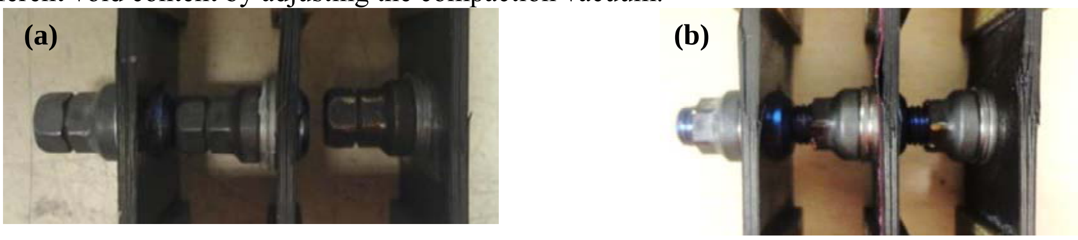


Figure 9. FHC test failure mode. (a)30% vacuum compaction, (b)100% vacuum compaction.

The research shows that the existence of the voids can cause local deformation of carbon fiber, and the local deformation of carbon fiber can reduce the compressive of the composites.

4. Conclusion

(1) The microscopic analysis showed that the voids exist in all the laminate area and the porosity presents in interface laminate and core.
(2) The porosity ultrasonic non-destructive measurement method are developed and verified through microscopic examination.
(3) Using the automatic image analysis system, the voids content was compared for different compaction pressure in the manufacturing process. Increasing the compaction pressure will reduce the voids content of the laminates.

(4) The results of the mechanical properties for sandwich structure composites indicate that the less voids, the higher composites mechanical properties, but the influence is not great for the case that the void content of the test panel are relatively low.

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