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Significant Effect of Vacuum Bagging Processing on Inter-Laminar Shear Strength and Voids of Composite in Oven Cure

Nur Hafzareen Md Hanafiah[1,*], Abdul Rahim Othman[1], Mark Ovinis[2]

[1] Department of Mechanical Engineering, Universiti Teknologi PETRONAS, 32610 Seri Iskandar, Perak, Malaysia
[2] College of Engineering, Birminghim City University, Birmingham, B4 7XG, United Kingdom

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ABSTRACT

Autoclave had been constrained to longer time consumption, higher production costs and excessive residual stress. The challenges regarding with out-of-autoclave (OOA) cure involved the need to devise the techniques in preserving high quality of composite product during service life. The current work was proposed to quantify the impact and influence of pre-forming parameter variations in vacuum-bagging-only with oven curing (VBO-oven cure) of OOA manufacturing composite processing on the inter-laminar shear strength (ILSS) of low-cost conventional composite laminates. The relationship of ILSS and void quality characteristics was also investigated. Series of conventional lower-cost glass/epoxy aerospace-grade material was fabricated using 16 different processing routes in conventional oven. Burn-off and ILSS test was conducted based on ASTM standard to compute the ILSS and void content of cured laminates. Scanning electron microscopy (SEM) analysis was performed on the post-test ILSS coupons to evaluate the relationship between void characteristics and ILSS. Results indicated that mould release type and intensifier contributed highest effects towards ILSS of approximately 31.3% and 27.6%, respectively. The assessment of ILSS-void relationship was further carried out and it was found that void geometry has a greater influence towards ILSS than the void content of composite laminate. The combination of these factors in processing routes yielded the lowest ILSS and void content at 24.85 MPa and 5.74%, respectively. These concluded that an optimized manufacturing technique could be expanded from the optimum settings of these processing parameters.

1. Introduction

Manufacturing of composite laminates for aerospace application typically involved the use of reinforcing woven fibre materials with pre-impregnated resin system. The fabrication of the prepreg into composite laminates were classified into pre-forming and curing process [1]. Throughout pre-forming, prepreg were prepared at controlled ambient temperature, laid up in a specified alignment and then fixed in a vacuum bagging. Subsequently, a vacuum pressure was applied to remove any gaps within the composite, expelling surplus air and resin from the vacuum bagging mould.

* *Corresponding author.*
E-mail address: hafzareen87@yahoo.com

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Typically, high pressure and temperature application was employed simultaneously during cure to consolidate the thermoset composite material [2]. An enormous pressure implementation was needed in order to eliminate any trapped air and volatile substances, while also compacting the individual layers and fibres. Nevertheless, the manufacturing of high-performance aerospace composite laminate usually involved vacuum bagging with autoclave cure, where controlled temperature, vacuum pressure and sets of immense compaction pressure were implemented throughout the cure cycle [3,4]. Though, this processing route was dealing with surprising limitations of immense capital, production and overall cost of approximately 30 to 50% higher than its counterpart of vacuum bagging with oven processing [5-7]. Moreover, unnecessary residual stress was found to exist within the cured laminate structure, which increased buckling loads up to 42% [8]. Hence, research in other processing techniques was essential in favour of shifting to Out-of-Autoclave (OOA) process, which involved the vacuum-bagging-only with oven cure (VBO-oven cure), as it represented the closest shift [9,10].

With reference to VBO-oven curing process using prepreg material, there were five fundamental steps involved; kitting, layup, vacuum bagging, curing and part removal. Even though the methodology resembled customary VBO-autoclave procedure; yet there was no bleeding condition and high-pressure application during cure profile. Consequently, produced laminates rarely attained the required mechanical qualities comparable to the autoclave counterparts [11,12]. Inadequate and poor manufacturing profile was found to reduce the mechanical properties of structural components especially the inter-laminar shear strength (ILSS), tensile strength, compressive strength and so on [13]. Additionally, since voids were known to affect the mechanical quality of composite, detailed and thorough investigations on voids that were generated throughout the manufacturing procedure were critical.

It was signified that the utmost influential parameter to bleed the entrapped voids from the prepreg stack during curing was the air permeability of the prepreg. Air permeability was claimed to have an inversely proportional relationship with compacting pressure because the dimension of the gas flow path was abridged under pressure conditions [14]. Several researchers suggested debulking before cure in vacuum bagging setup should be carried out to each individual prepreg layup for 20 minutes at ambient temperature to ensure the evacuation of trapped voids was effective [15,16]. The effect of heat treatment in conjunction with debulking on carbon/epoxy laminates was explored and it was revealed that the application of heated debulking resulted in a noteworthy reduction of the manufacturing cycle time, ranging from 26% to 71%. In comparison with the typical room temperature debulk counterparts, ILSS and void content were found to improve by approximately 5% and 15.3%, respectively. [17]. In addition, employing a rubber seal as the reinforcement beneath the steel plate successfully facilitated even distribution of the compacting pressure throughout the curing process [18].

On top, the quality of the surface finish of the laminate was found to be influenced by the various types of mould release agents. The application of release spray, wax, or film serves to prevent significant stress transfer between the mould and the laminate. In the absence of a release agent, higher strain is anticipated, leading to the undesired development of spring-in within the laminate [19,20]. Despite that, the implementation of release agent, particularly adhesive film, was discovered to restrict the air permeability flow in through-thickness direction, which reduced the amount of void vented out during cure [12]. Moreover, dry glass fibres were positioned along the laminate's edge, serving as the edge breather within the vacuum bag. This placement was observed to effectively enhance air permeability for the evacuation of entrapped air, especially along the in-plane direction of the laminate [21].

In a review study of Judd and Wright, most researches contemplated on void effects towards ILSS of composite structures. They claimed that an average of 1% increase in void content formed a reduction of 7% in ILSS value [22]. Other researches also reported that the results of the studies on ILSS and tensile strength reduction by the increment of void content [23-25]. Hou *et al.*, [26] stated that the ILSS was one of the critical requirements for aileron ribs as the side walls of the structures encounter high flexural pressures and aerodynamics load throughout the service. It was claimed that ILSS properties was vital to the aircraft control-lift surfaces such as elevator and rudder to improve the aerodynamics performances during service [27].

Previous research findings underscored that the pre-forming procedures significantly influenced the ultimate properties of the cured composite. It was suggested that optimizing the VBO processing techniques could further enhance these qualities. The study examined the impact of the parameters, and it quantified the contributions of both individual and combined processing routes. A 2-factorial experimental study was suggested to explore the impact of individual and combined pre-forming parameters on the ILSS (interlaminar shear strength) of low-cost, conventional material composite laminates. This involved testing 16 distinct processing routes. An ILSS test, conducted in accordance with ASTM D 2344/D 2344M-00, was performed to ascertain the interlaminar shear strength of the laminate. Burn off analysis was performed to measure the void content and scanning electron microscopic inspection (SEM) was used to analyse the size and shapes of voids on the post-test ILSS coupons. The interaction among the combined parameters was then assessed utilizing an analysis of variance (ANOVA) tool. According to the findings, the impact of processing parameters in the VBO-oven curing technique on ILSS was clearly established. The assessment of ILSS-void relationship was further carried out to evaluate the relationship between void characteristics and ILSS.

2. Methodology

2.1 Materials

The prepreg employed was a plain weave glass fibre-reinforced epoxy, specifically Cycom 7668/7881-1, provided by Cytec Engineered Materials Inc. This prepreg is designed for structural laminates intended for use in aircraft exteriors. The woven fabric saturated with the 7668-epoxy resin was manufactured through the hot-melt process, with a resin flow percentage of 17±5%. It demonstrates favourable tack and drape for a minimum of 15 days at 24 ˚C. Once cured, the material exhibits exceptional resistance to thermal aging, notable flammability characteristics, and outstanding tension and compression properties throughout its service life.

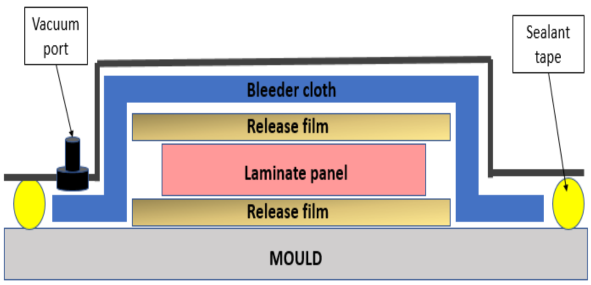
2.2 Manufacturing

The prepregs were trimmed into square measuring of 30 cm × 30 cm in a designated room where humidity and temperature were carefully controlled at approximately 50% and 24^{o}C, respectively. Nine plies of prepreg were laid up, resulting a nominal thickness of approximately 2 mm. The composite was manufactured using the VBO (Vacuum Bag Only) process, employing different configurations of bagging setups based on the processing routes outlined below. Four variables, namely vacuum debulking, various types of mould release agents, intensifier, and edge breather, were examined at two distinct levels through a complete factorial design comprising 24 experimental setups. Table 1 illustrates the details, resulting in 16 diverse processing combinations.

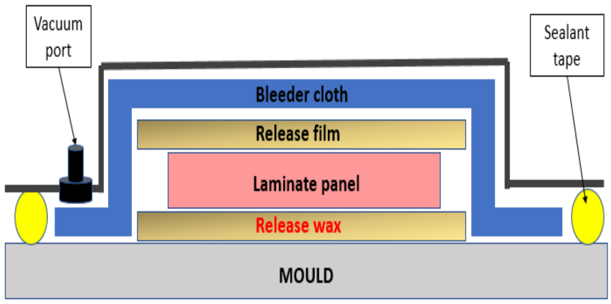
Table 1
Factors and levels for processing parameters

Symbol	Factors	Level 1	Level 2
A	Debulking	No	Yes
B	Mould release type	Wax	Film
C	Edge breather	No	Yes
D	Intensifier	No	Yes

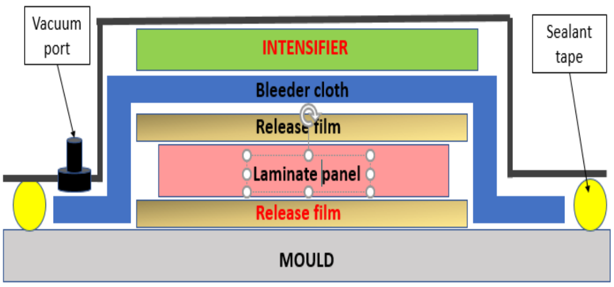
Figure 1 illustrates the variations in vacuum bagging utilized in the research. In the standard (baseline) bagging method, prepreg plies were arranged on a flat aluminium mould, and a mould release film was positioned between the initial layer and the metallic tool. Additional consumables were arranged above the materials in the sequence; non-perforated release film (RF260 by Tygavac Advanced Materials), Airweave N10 bleeder cloth (Airtech Advanced Materials), and WL7400 vacuum bag (Airtech Advanced Materials). The assembly was enclosed, and the edges of the vacuum bagging were sealed using GS2131/2 sealant tape from General Sealants Inc. A singular vacuum port channel (Airtech Advanced Materials) was utilized to remove air from the sealed bagging.



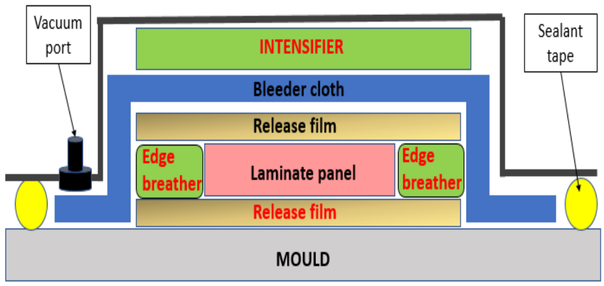
(a) Baseline ($A_1B_2C_1D_1$) configuration



(b) $A_1B_1C_1D_1$ configuration



(c) $A_1B_2C_1D_2$ configuration



(d) $A_2B_2C_2D_2$ configuration

Fig. 1. Different configurations of vacuum bagging pre-forming

The bagging configuration (i.e. $A_1B_1C_1D_1$) in Figure 1 (b) utilized release wax instead of release film. A fine coat of release wax was manually applied to the cleaned mould using a clean cloth or sponge, using a rotary motion. This procedure was repeated five times, with a 10-minute interval between each application to ensure thorough drying of the release wax layer. The intermediate configuration (i.e., A1B2C1D2) involved the use of an intensifier or caul plate to enhance the contact pressure. This addition aimed to reduce the porosity and void content in the cured laminates. Specifically, a 300mm x 300mm, 1kg stainless steel plate served as the intensifier, positioned on top of the laminate plies and over the bleeder in the bagging setup.

In the complete bagging configuration (i.e., A2B2C2D2), vacuum debulking was executed during the layup phase using a JK-VP-3C single-stage vacuum pump. An Airtech-Airflow 65R hose and VV 401 Airtech vacuum port were connected to the vacuum bagging for this process. Debulking was performed by applying a low vacuum pressure to each individual prepreg layup for 20 minutes at room temperature, continuing until all nine prepreg plies had been laid up. Additionally, dry glass fibre strip measuring 1 cm in thickness and 2.5 cm in width, specifically Tygavac Y-0094, served as the edge breather. It was positioned at each corner of the laminate plies to aid in the evacuation of in-plane voids. The breather cloth also functions as the in-plane bleeder material, facilitating the expulsion of voids by drawing entrapped air and excess resin out from the laminate plies via in-plane direction.

Each laminate was subjected to an identical curing cycle, carried out in a Grieve WRC 566-500 oven, ensuring a continuous vacuum supply to the bagging throughout the curing process. The curing process comprised a dwell section lasting 120 minutes at a temperature of 180 ± 12 °C. The curing cycle involves heating and cooling rates set at 2 °C/minute. A main vacuum pump, connected to the vacuum bag, sustained a vacuum pressure of 1 kPa throughout the curing process, starting from the initiation of the cure until the laminate panel was taken out, aligning with the cooling of the temperature to 60 °C.

2.3 Testing Analysis
2.3.1 Inter-laminar shear strength test

ILSS test was conducted based on ASTM standard of D 2344/ D 2344 M–00. A total of 10 samples per panel were prepared with dimensions of 25 mm x 8 mm. Test coupons were subjected to a quasi-static loading at a constant speed of 1 mm/min using an Instron universal testing machine. To meet the ILSS value calculation requirement outlined in the ASTM D 2344/D 2344 M-00 standard, a consistent span length of 18 mm was mandated for each ILSS specimen.

2.3.2 Burn-off test

In accordance with the ASTM standard D 3171-99, specimens measuring 10 mm x 20 mm and weighing 1.0 gram per panel were ground and polished before the test. The resin burn-off process occurred in a Vecstar Naber Therm NII F-24389 muffle furnace at a temperature of 565 ±30^{o}C for a duration of six hours. This process aimed to eliminate the resin, leaving only the fibre behind. Subsequently, the remaining fibre was cooled to room temperature within desiccators, and its weight was re-evaluated to determine the void content of the panel.

2.3.3 Scanning electron microscopy

Scanning electron microscopic (SEM) analysis was carried out on the post-test coupons to evaluate the relationship between void characteristics and ILSS value of the laminate. It was anticipated that void percentage would have a significant influence on laminates' ILSS results, thus the SEM analysis was performed to characterize the cracks of inter-laminar fracture due to void. Tested specimens were firstly polished using 100, 400 and 600 grids of sandpapers, and subsequently coated with gold/palladium alloy using mini sputter coater machine. An SEM machine was used to complete the analysis. The samples were photographed at various magnifications (from 50x to 300x) via the electron microscope. Void distribution and geometry of voids were also analysed to examine their effects on ILSS. For the image analysis, a software within SEM machine was used to quantify void aspect ratio (length: width), which represented the void geometry.

3. Results
3.1 ILSS and Void Content

Results of ILSS and void content were tabulated in Table 2. It was found that ILSS values for the laminates were at the range of 24.85 MPa to 17.74 MPa. Three highest ILSS measured were for laminates 14, 12 and 13 with the value of 24.85 MPa, 24.62 MPa and 24.25 MPa, respectively. The ILSS values were lowest for laminates 8, 4, and 16, measuring 17.74 MPa, 18.10 MPa, and 18.64 MPa, respectively. The standard deviation errors calculated was between ± 0.57 MPa to ±1.60 MPa for laminates 12 and 16, respectively. The void content in the laminates ranged from 5.74% to 8.36%, with the highest observed in laminate 3 (baseline laminate), produced through the standard pre-forming processing route. The void content reached its minimum, measuring at 5.74%, 5.81%, and 6.02%, when the specified combination of edge breather and wax mould release was employed. Since the characteristics of voids affected the value of ILSS critically, a low value of standard deviation showed that the distribution and size of voids within the laminate panel was consistently distributed and uniform, and vice versa. Hence, an examination of the characteristics of voids that led to the formation of cracks was necessary to understand the impact of void geometry and distribution on the ILSS of the composite panel.

Table 2
Results for ILSS and void content

Trial	Factor				ILSS (MPa)	Void content (%)
	A	B	C	D		
1	1	1	1	1	20.27	7.43
2	2	1	1	1	21.71	6.88
3 (baseline)	1	2	1	1	19.95	8.36
4	2	2	1	1	18.10	8
5	1	1	2	1	23.98	6.02
6	2	1	2	1	21.07	6.91
7	1	2	2	1	19.72	7.43
8	2	2	2	1	17.74	7.44
9	1	1	1	2	22.47	6.77
10	2	1	1	2	20.82	7.53
11	1	2	1	2	20.39	7.21
12	2	2	1	2	24.62	8.1
13	1	1	2	2	24.25	5.81
14	2	1	2	2	24.85	5.74
15	1	2	2	2	22.85	6.06
16	2	2	2	2	18.64	7.79

3.2 Analysis of Variance (ANOVA)

In order to evaluate the significant effects of individual and combined input variables on the ILSS property, a thorough analysis was conducted using ANOVA, as depicted in Table 3. It was observed that factors B and D contributed significant model terms, indicating that these factors had the most pronounced effect on ILSS, as illustrated in Figure 2.

Table 3
ANOVA for ILSS

Source	Sum of Squares		Df	Mean Square	F-value	P-value
Model	60.467		10	6.0467	1.56	0.0
B	18.944		1	18.944	4.87	0.078
D	16.708		1	16.708	4.3	0.093
Std. Dev.		Mean		C.V. %	PRESS	
1.972		303.74		9.68	199.04	
R-Squared		Adj R-Squared			Adeq Precision	
0.757		0.27			5.78	

The factors of A, C, AB, AC, AD, BC, BD, CD, ABC, ACD, BCD, and ABCD, were excluded from the contribution bar plot in Figure 2 and Table 3. This exclusion was due to their P-values being greater than 0.100, indicating insignificant model terms. As a result, the percentage contribution of factor B (31.3%) and D (27.6%) calculated by ANOVA exemplified the highest contribution value than the other model terms.

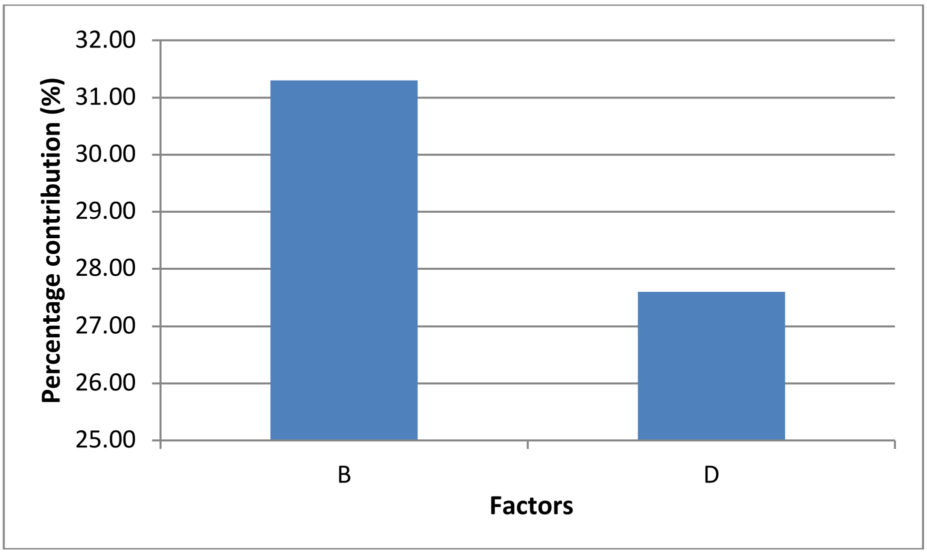


Fig. 2. Percentage contribution of significant factor parameters towards ILSS

Figure 3 depicted the primary effects of processing parameters on the ILSS of the laminate panel in this study. The application of wax mould release, edge breather, and intensifier resulted in an increase in the ILSS value of the laminate. Notably, the intensifier and release wax had the most substantial impact, contributing to an increment of approximately 10.11% and 10.62%, respectively. Edge breather enhanced the ILSS by 2.61%, while release film reduced the ILSS value of laminate by approximately 5.75% and 9.62%, respectively. The implementation of the debulking process clearly had an adverse impact on the laminate, leading to a reduction in ILSS by 3.91%.

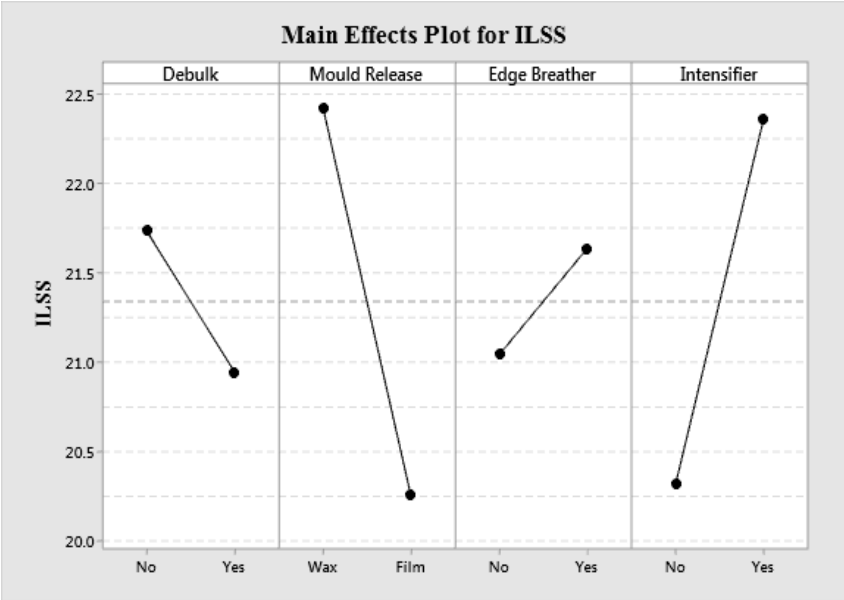


Fig. 3. Main effects plot of single factor parameter towards ILSS

It was found that the ILSS value of the laminated composite was primarily influenced by the quality of the resin [28]. As a result, the characteristics of voids within the resin played a crucial role in determining the ILSS value in these panels. Hou *et al.,* [26] claimed that ILSS property was inversely proportional to void percentage of laminate. As single factor of debulking was claimed to increase void content by blocking the in-plane air permeability [17], ILSS value was subsequently decreased which was further shown in Figure 3. Debulking alone was insufficient to compress the large void that led to the crack during the ILSS test, without assistance from other VBO parameters to further reduce void size. Nevertheless, the ILSS demonstrated an increase when the debulking process was combined with both release wax and intensifier, as illustrated in Figure 4.

The interaction plots of two combined processing parameters towards ILSS of laminate panels were illustrated in Figure 4. It was observed that there were no interactions between any two combined factors considered in relation to the ILSS value. The graphs depicting parallel lines in the ANOVA analysis indicated that the factors remained independent when both factors were merged. However, it was discovered that there were some diverse patterns observed for ILSS with the two combinations; edge breather with mould release, and edge breather with debulk. The ILSS value was reduced by approximately 9.52% when edge breather was combined with release film than single release film factor. Whereas, when edge breather was combined with release wax, ILSS was increased by approximately 11.9% than those of counterparts without the use of edge breather. If edge breather was combined with debulk, ILSS value was reduced by approximately 10.87% than those of counterparts without edge breather. These meant that edge breather provided a negative effect when combined with debulk and release film, where edge breather should only be used as a single factor or only combined with release wax and intensifier.

In addition, ILSS was increased by 17.4% when debulking was combined with intensifier, rather than employing debulking alone, as shown earlier in Figure 4. These proved that the entrapped voids were compressed by both debulking and intensifier. Moreover, ILSS increment of 16.98% was also observed with combination of debulking and mould release wax. These attributed to the fact that release film acted as the inter-plane dam between mould and laminate, hence excess resin and entrapped voids were obstructed from being evacuated. Whereas, wax acted as the inter-plane permeability pathway for the excess resin and entrapped porosity to be evacuated out from the laminate. Therefore, debulk, mould release wax, edge breather and intensifier produced the highest ILSS value of 24.85 MPa which was exhibited in laminate 14.

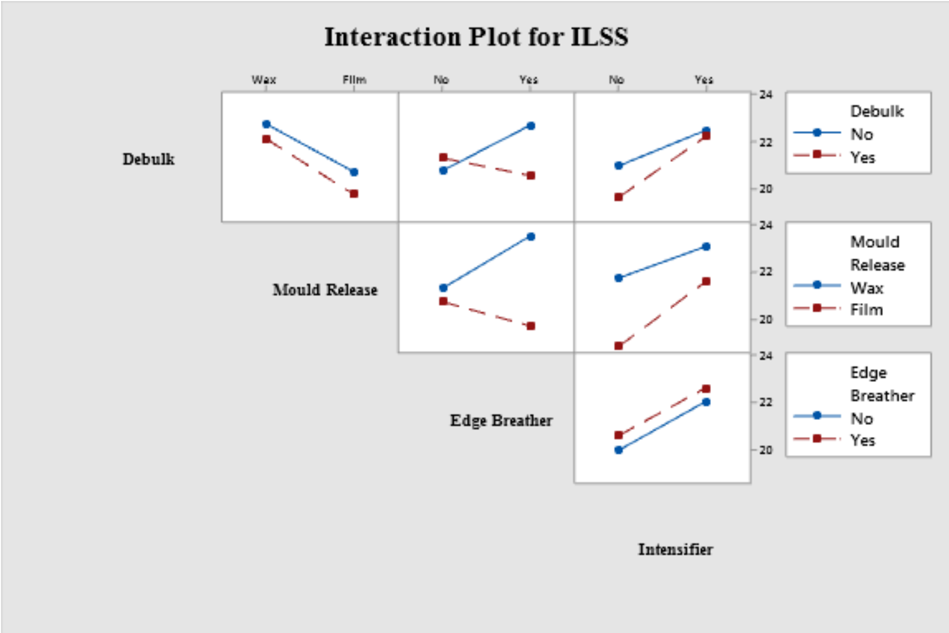


Fig. 4. Combined interactions plot of two factor parameters towards ILSS

3.3 Relationship of ILSS with Void

The inter-relationship of ILSS and voids was investigated to acquire voids characteristic that initiated the crack which resembled the maximum flexure stress of ILSS test coupon. SEM was carried out on the tested ILSS panels to examine volume fraction, shapes, geometry and distributions of post-tested local void. Figure 5 and Figure 6 showed the SEM images of voids emanating the cracks of different laminate panels from single large voids and multiple distributions of small voids, respectively. It was found that voids which initiated the crack of post-tested ILSS panel were classified into two categories; single large voids and multiple distributions of small voids.

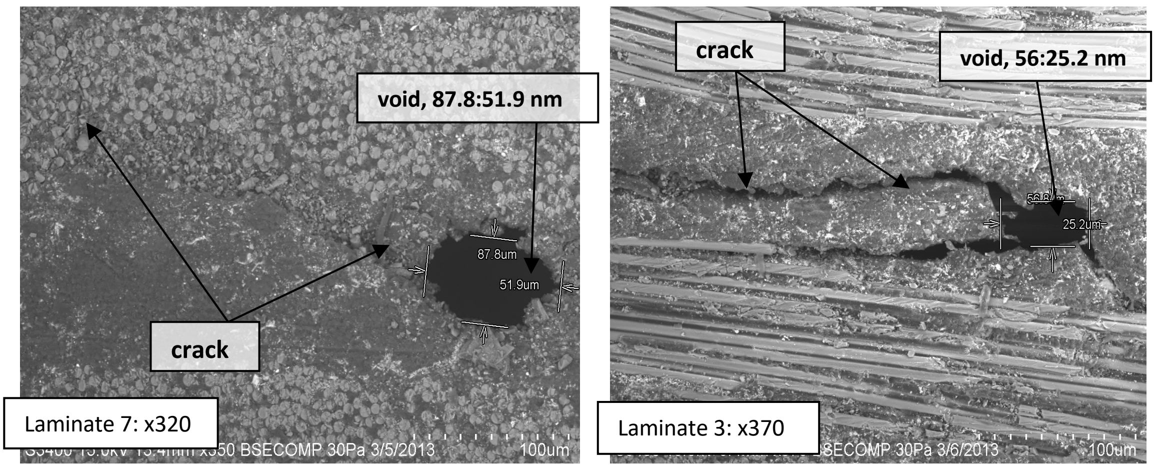


Fig. 5. SEM images of post-ILSS panels revealing cracks originating from single large voids with length to width ratio in nanometres

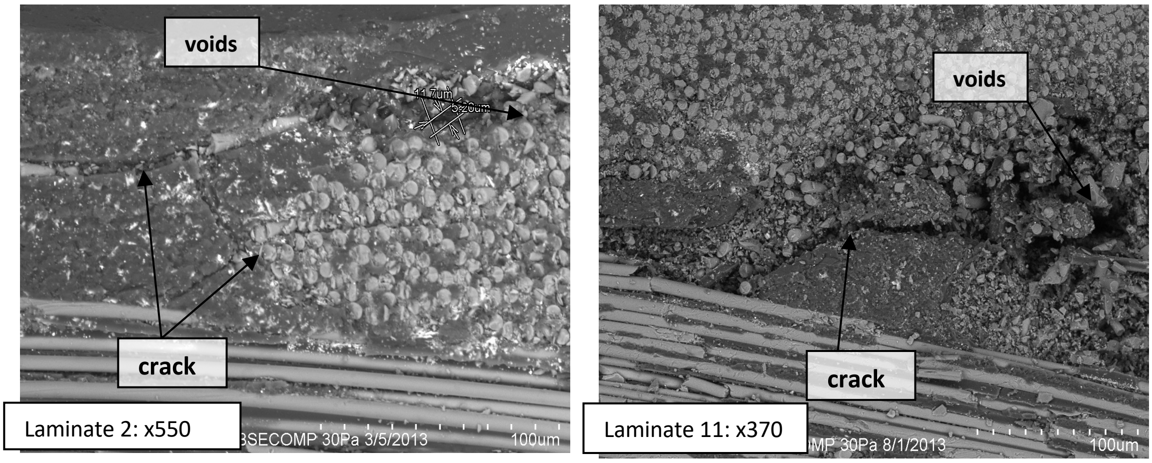


Fig. 6. SEM images of post-ILSS panels revealing cracks originating from multiple small distributed voids

The classification of these voids was defined based on length to width ratio of voids that emanated the cracks prior to ILSS test. Figure 7 illustrated that ILSS had an inconsistent inverse proportional relationship with void content. Researchers proved that voids initiated the cracks which contributed to ILSS value of composite panel [23]. It was found that void size and distribution had a higher influence on ILSS value than void content within laminate. Panels with large voids delaminated more rapidly than the panels with multiple small distributed voids. Laminate panels with multiple small distributed voids were likely to have higher ILSS value than the panels with single or less quantity of large void. As a comparison to void content, it was found that the declined value of ILSS was not consistently following the inversely proportional pattern assumption to void content. Figure 7 showed the inversely proportional relationship plot of ILSS versus void size (length x width) in micrometres for cracks emanating from one big size void of the laminate panels in Figure 5.

It was found that laminate 14 exhibited lowest void content of 5.74%, with highest ILSS, while highest void content of 8.36% was recorded for laminate 3 with middle range of ILSS of 19.95 MPa. It was also proved that for the case of panels with single large void, ILSS value was reduced when the width to length ratio was increased, which were displayed in Figure 7. These exposed that panels with larger void size tended to delaminate more rapidly as compared to the smaller size of void [23]. It can be concluded that ILSS of composite specimen was critically dependable on size of voids, then on voids distribution and lastly on void content of laminate. During VBO preforming technique, the epoxy resin of the prepreg was in B-stage system wherein the polymerization between matrix and curing agent/hardener was inadequate, thus the system was in a partially cured stage. Since the debulk process was performed when resin was in the stage between gel and solid, the large voids and entrapped air have been compressed and divided into a higher quantity of multiple small distributed voids within the laminate. Thus, void content within the whole laminate was still similar, but differed in size and distribution.

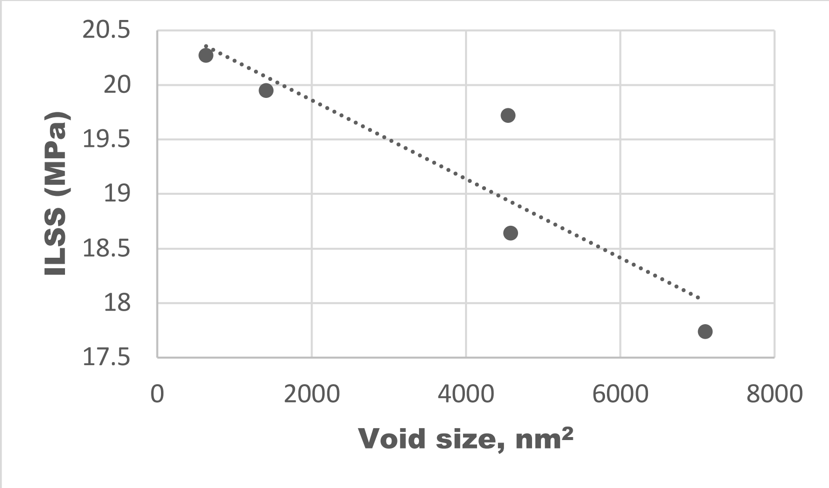


Fig. 7. Plot of ILSS relationship with void size

4. Conclusion

Experimental study had been conducted to quantify the contribution of individual and combination of pre-forming parameters on ILSS of conventional low-cost glass/epoxy laminates. Analysis of ILSS-void relationship was further carried out via SEM to evaluate the relationship between void characteristics and ILSS. A factorial design based on four factors, including debulking, mould release type, intensifier and edge breather, had been proposed for 16 different processing routes, to establish the interaction between the combined factors. It was observed that mould release type and intensifier contributed highest effects towards ILSS of approximately 31.3% and 27.6%, respectively. Void geometry was found to give greater impact on ILSS than the void content of composite laminate. It can be concluded that ILSS was critically dependable on size of voids, then on voids distribution and lastly on void content of laminate. The aggregation of all processing parameters in this study yielded the lowest ILSS and void content at 24.85 MPa and 5.74%, respectively. These concluded that an optimized procedure by using these processing parameters could be potentially extended to develop an optimum quality of composite laminate.

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