Jurnal Mekanikal
December 2007, No. 24, 32 - 39

THE EFFECT OF MICRO-BUBBLES ELIMINATION PRIOR RESIN INFUSION PROCESS

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ABSTRACT

The treatment of resin mixture prior to resin infusion is discussed. The elimination of micro-bubble due to entrained air during mixing process had been done using a novel technique of capillary separation method. In this process micro-bubbles were separated from viscous resin mixture, thus bubble-free resin was produced and then infused. Tested samples using treated resin experimentally showed less void contents and higher tensile strength.

Keywords: *Micro-bubbles, voids content, resin infusion, strength, moulding.*

1.0 INTRODUCTION

It is found that composite laminates strength is very sensitive to the present of voids. The reduction in mechanical properties due to voids as shown by Judd et al [1] clearly emphasized the need of special treatments during the manufacturing of composite structures. From background studies, a number of researchers have tried many methods in reducing the amount of voids. Loburdus et al [2] had discovered that the amount of dissolved gas and volatile component was one of the causes that promoted void contents development during vacuum infusion process, as it tend to outgas under low pressure medium. Labordus et al [3] suggested that the degassing procedure before the infusion process helped reduce the formation of voids. Other researchers such as Wood et al [4] introduced post treatment like curing under high pressure, either reducing the size of the bubbles or collapsing the micro-bubbles to prevent void formations.

From recent studies the author found that air bubbles not only nucleate from the resin mixture but also entrained during the mixing process [5]. During an industrial visit the author learned that the mixing of epoxy resin and amine hardener was conducted in a continuous manner to ensure a continuous supply of resin. It was found that the infused resin contained a lot of micro-bubbles. These bubbles either coalesced with other bubbles or dispersed in between fibre tows or stayed localized during curing. As this problem was found to be a major contributor to void content formations, a novel method had been developed for preventing void formation and perfecting the of vacuum infusion process [5].

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1.1 The Method of Micro-Bubbles Elimination

Relieving micro-bubbles from viscous fluid by natural flow is a slow process. The size of the bubble is too small to make the bubble rise up fast enough to the surface in an appreciable time. Howell [6] showed that bubble also took time to break when it had surfaced. This procedure is not very productive as pot life is a major concern to the manufacturer.

By employing capillary separation method, the author found that resin saturated with micro-bubbles can be filtered, immediately after mixing. The filtered resin was found to be clear of micro-bubbles. Basically, this method used a special kind of fabric that could absorb the resin via capillary action. After the fabric medium was fully saturated with resin, the potential force of the resin overcame the surface tension of the resin; therefore, a continuous flow of resin through the filter occurred. At the same time micro-bubbles were constrained from entering the fabric medium because gas bubble behaves like non-wetting fluid. The stranded bubbles coalesced together forming bigger bubbles before breaking up. To ensure no bubbles nucleation from volatile component this filtering process was performed under reduced pressure of 150 mbar.

2.0 EXPERIMENTAL

One layer lamination was made from 2 x 2 twill weave E-glass fibre woven fabric of 800 g/m^2 (weft/warp direction 0°/90°, weft/width size 1000 mm and warp/length size 75 m, 60 kg per roll as supplied by Allscot Distributors Ltd). It was infused at 60.0 mbar with an ultra low viscosity epoxy laminating system (RS-L135i) using hardener RS-H137i as supplied by PRF Composite UK Ltd. This resin system was specifically formulated for injection moulding (RTM) and vacuum infusion processes. The mixture viscosity was about 200 mPa-s at 25°C.

Tensile test was conducted to determine the strength of the samples that had been infused with untreated resin and treated resin that had been filtered by capillary separation method and vacuum degassed. Void content analysis had also been made to the samples in order to establish the relationship between void and tensile strength. Samples using treated resin were tagged A, and samples using untreated resin were tagged B. Samples A was cured under room temperature and samples B at elevated temperature so that the bubbles in the lamination would stay localized rather than diffused in between fibre tows which would create additional void content.

2.1 Void Content Analysis

Void content analysis was done according to BS EN ISO 7822:1999. For void content, it was necessary to have the theoretical densities of both the resin and the reinforcing material in order to determine the theoretical density ρ_t of the composite. The individual theoretical densities were as follows:

- Epoxy resin density $\rho_e = 1.17$ gcm^{-3} (PRF Composite UK Ltd)
- Glass fabric density $\rho_g = 2.54$ gcm^{-3} (Allscot Distributors Ltd)

After the actual measured density of the composite material was determined, the weighed sample (m_1) was placed into a weighed crucible (m_2) and burned in a 600°C muffle furnace until only the reinforcing material remained. The crucible

was cooled and weighed (m_3). The resin content (m_e- ignition loss) was calculated from the available data. For further simplification:

- Epoxy resin mass w% $m_e = 100((m_1 + m_2) - m_3)/ m_1$
- Glass Reinforcement mass w% $m_g = 100(m_3 - m_2)/ m_1$
- Theoretical density $\rho_t = 100 / (m_e / \rho_g + m_g / \rho_e)$

By comparing the actual density to the theoretical density, void content was calculated as follows:

- The void content % $void = 100(\rho_t - \rho_m) / \rho_t$

Two types of sample were prepared, sample A1, A2, A3 used treated resin which is free of micro-bubbles and sample B1, B2, B3 used untreated resin that contains micro-bubbles. Each sample is weighed and its dimensional is taken for the determination of the measured density ρ_m as shown in Table 1.

Table 1: Measured length and thickness for determination of ρ_m

Sample	W 01 (mm)	L 01 (mm)	W 02 (mm)	L 02 (mm)	Area (cm^2)	t (cm)	Volume (cm^3)	m (g)	ρ_m g/cm^3
A1	50.00	101.80	50.50	101.90	51.18	0.075	3.84	6.39	1.66
A2	49.92	101.98	50.70	102.10	51.34	0.075	3.85	6.38	1.65
A3	49.86	102.18	50.00	102.36	51.06	0.075	3.83	6.34	1.65
								Average	1.66
B1	50.66	102.36	50.74	102.36	51.90	0.075	3.90	6.39	1.64
B2	49.56	102.36	49.66	102.42	50.80	0.075	3.82	6.22	1.63
B3	50.40	102.36	51.30	102.36	52.05	0.075	3.91	6.45	1.65
								Average	1.64

Table 2: Loss ignition test result

Specimen of sample	m_1 (g)	m_e (g)	m_g (g)	m_e (%w)	m_g (%w)	ρ_t (g/cm^3)	ρ_m (g/cm^3)	*void* (%)
A1	0.93	0.43	0.50	45.90	54.10	1.67	1.66	0.87
A2	0.79	0.37	0.42	46.85	53.15	1.66	1.66	0.19
A3	0.83	0.38	0.45	46.02	53.98	1.67	1.66	0.79
A4	0.87	0.41	0.46	46.74	53.26	1.66	1.66	0.27
							Average	0.53
B1	0.84	0.31	0.53	36.44	63.56	1.79	1.64	8.68
B2	0.96	0.37	0.59	38.19	61.81	1.77	1.64	7.44
B3	0.95	0.34	0.61	35.95	64.05	1.80	1.64	9.03
B4	1.02	0.38	0.64	37.31	62.69	1.78	1.64	8.07
							Average	8.305

The samples were then cut into a smaller size specimen of 25 mm by 25 mm as proposed by the standard method. Due to the shear cutting process, visible delamination was seen at the edges of every specimen. To ensure this edge-void did not affect the measuring of the actual void content, the delaminated edge was removed using waterproof silicon carbide paper (FEPA P-1200). The result of the loss ignition test is presented in Table 2. From the analysis, it was found that samples A exhibited very low void content as compared to sample B.

2.2 Tensile Test

The test was done based on BS 2782-10: Method 1003:1997, EN 61:1977. The method specified is applicable to reinforced thermosetting resins and reinforced thermoplastics.

A universal testing machine (ZWICK 2061) was used to perform this testing. The machine servo hydraulic system and the load cell maximum capacity is 50 kN. The machine was calibrated to the specification of BS EN ISO 500. An Avery Denison 25 mm extensometer was used to measure the elongation and recording was done on Rubicon system data logging equipment. The machine pulling speed was set at 2.0 mm/min, taking into account the strain sensitivity of the material tested.

The specimen dimension was measured using a micrometer. From these measurements, the cross sectional area was calculated and used to determine the stress value at corresponding tensile load.

From the data logging system, the elongation and tensile load data were extracted and transformed into strain and stress values, which were plotted for further analysis. Five testing specimens were taken from each infusion sample. These testing specimens were cut in parallel to the flow front. Each specimen decreases in thickness with respect to distance from the flow inlet. The variation and average thickness of the specimens is shown in Table 3.

Table 3: Specimen thickness

	Thickness (mm)					
Sample	1	2	3	4	5	Average
A	0.514	0.512	0.512	0.511	0.509	0.512
B	0.532	0.531	0.529	0.527	0.525	0.529

The testing specimens were cut to size of 170 mm long and 25 mm wide (Figure 1). The grip ends of each piece of the samples were protected using glued adhesive (epoxy) aluminium plates (30 mm x 25 mm x 1 mm). The contact surface was first roughened using silicon carbide paper.

After testing most of the specimens were found to have fractured at different locations. Specimen with breaking points located in the grips or at a distance of less than 5 mm from the grips is discarded from the analysis. The failure is due to the non-uniform micro-bubble distribution that forming weak spot on the specimen. Figures 2 and 3 show the stress-strain plot of each sample with accounted error of 5%, which included errors in measuring and testing equipment. The line shown in each figure represents linearized points using the least square method.

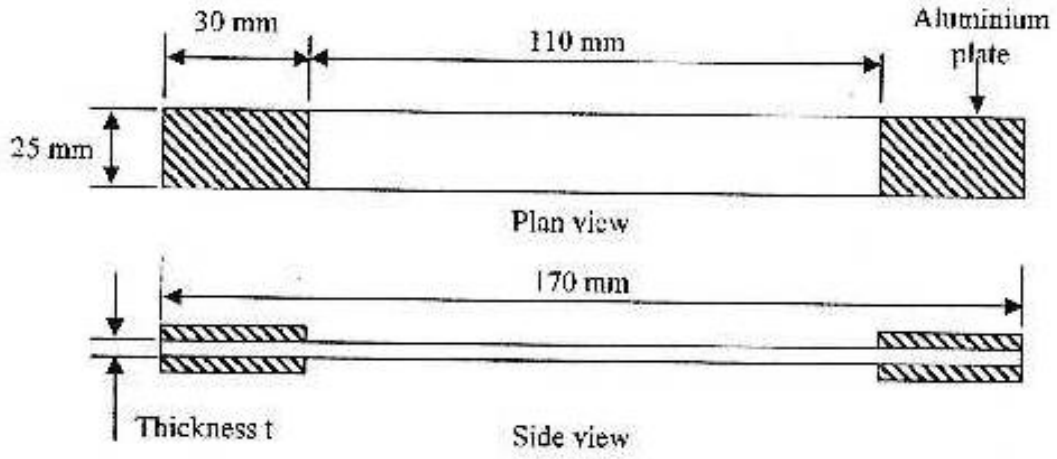

Figure 1: Sample dimension schematic diagram

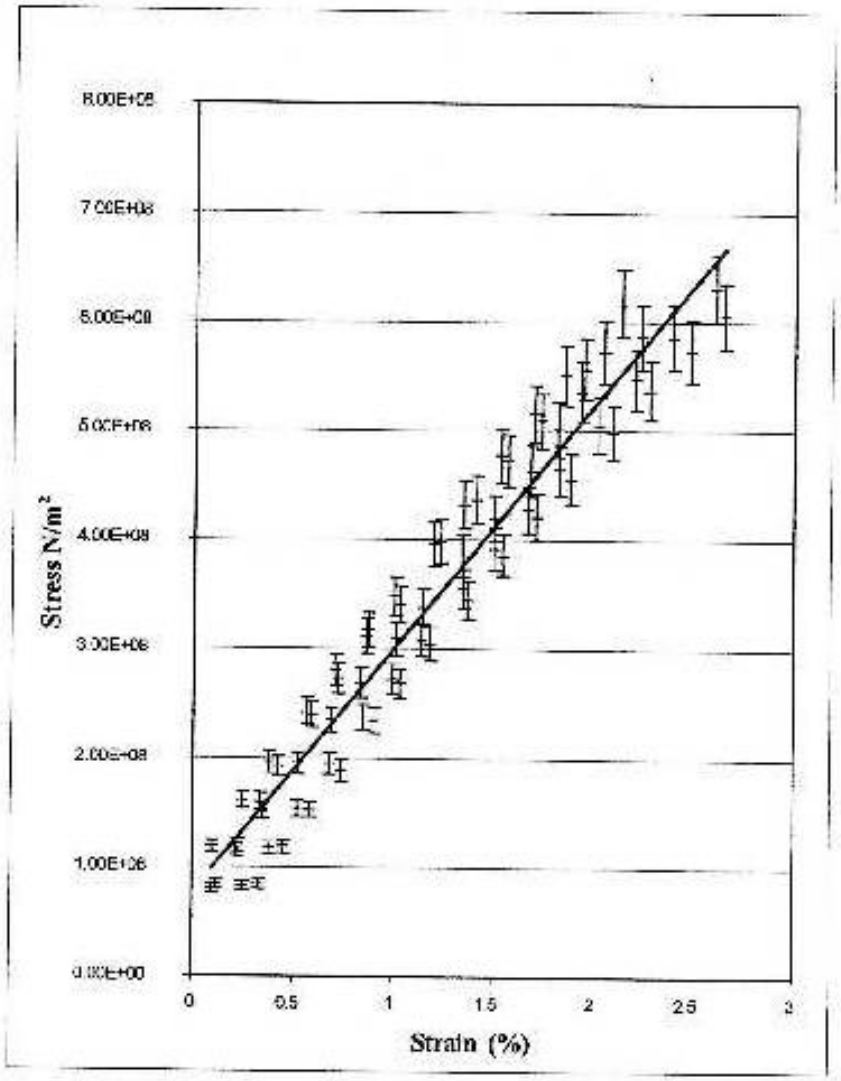

Figure 2: Stress strain of sample A specimens

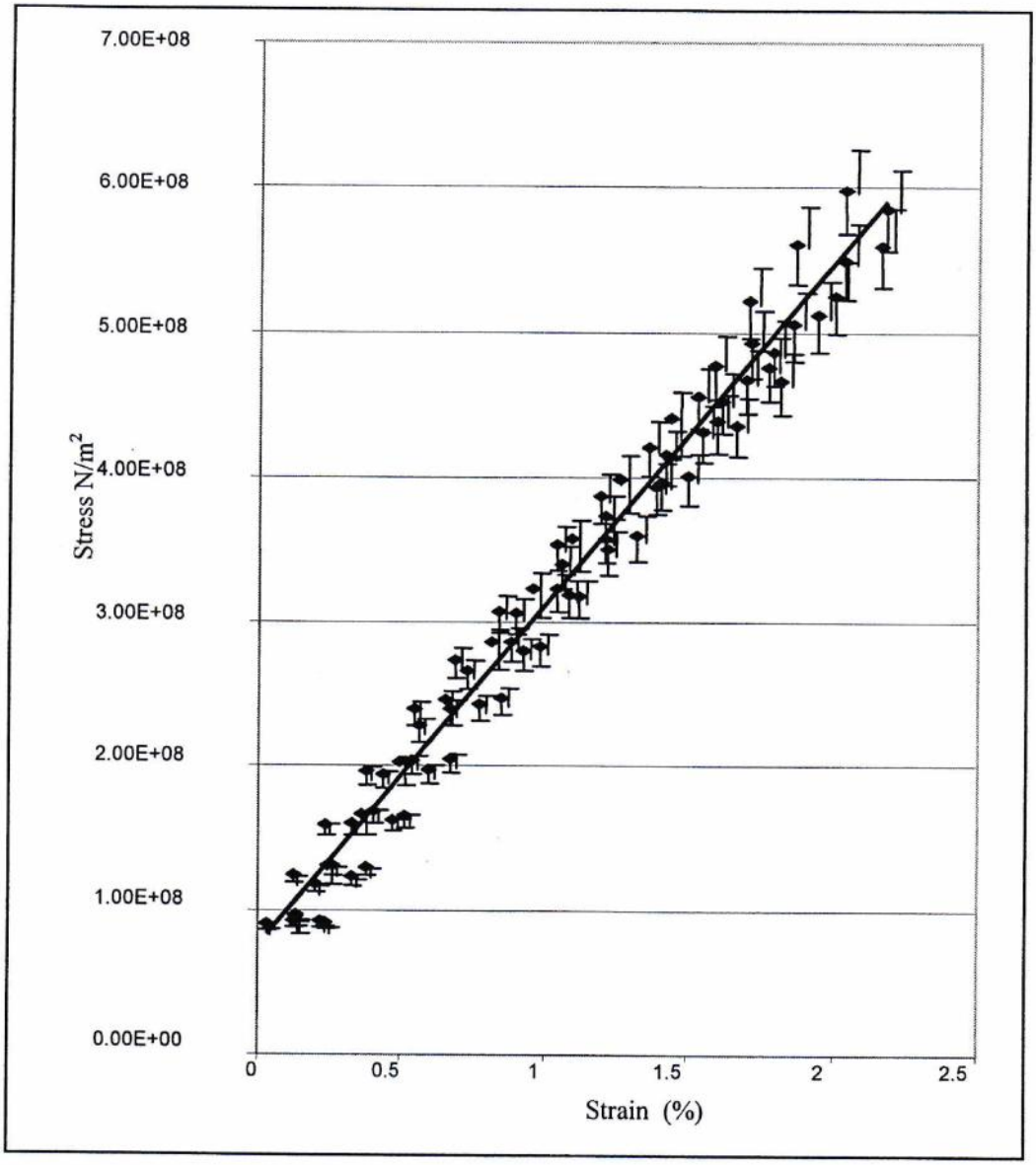

Figure 3: Stress strain of sample B specimens

Table 4: Comparison of specimen's mechanical properties

Sample	Tensile strength MN/m^2	Maximum strain %	Young's modulus GN/m^2
A	631	2.65	23.8
B	596	2.23	26.7

Table 4 shows samples A with very low void content presented higher strength and less stiffness. Samples B was much stiffer but were lower in strength. This illustrated the effect of elevated temperature curing process, which increased the mechanical properties of the epoxy resin and at the same time prevented the micro-bubbles from diffusing into dispersed void formation as stated in Section 2.0. However, the effect of infusion with micro-bubbles, which resulted in the reduction of strength, was clearly shown here.

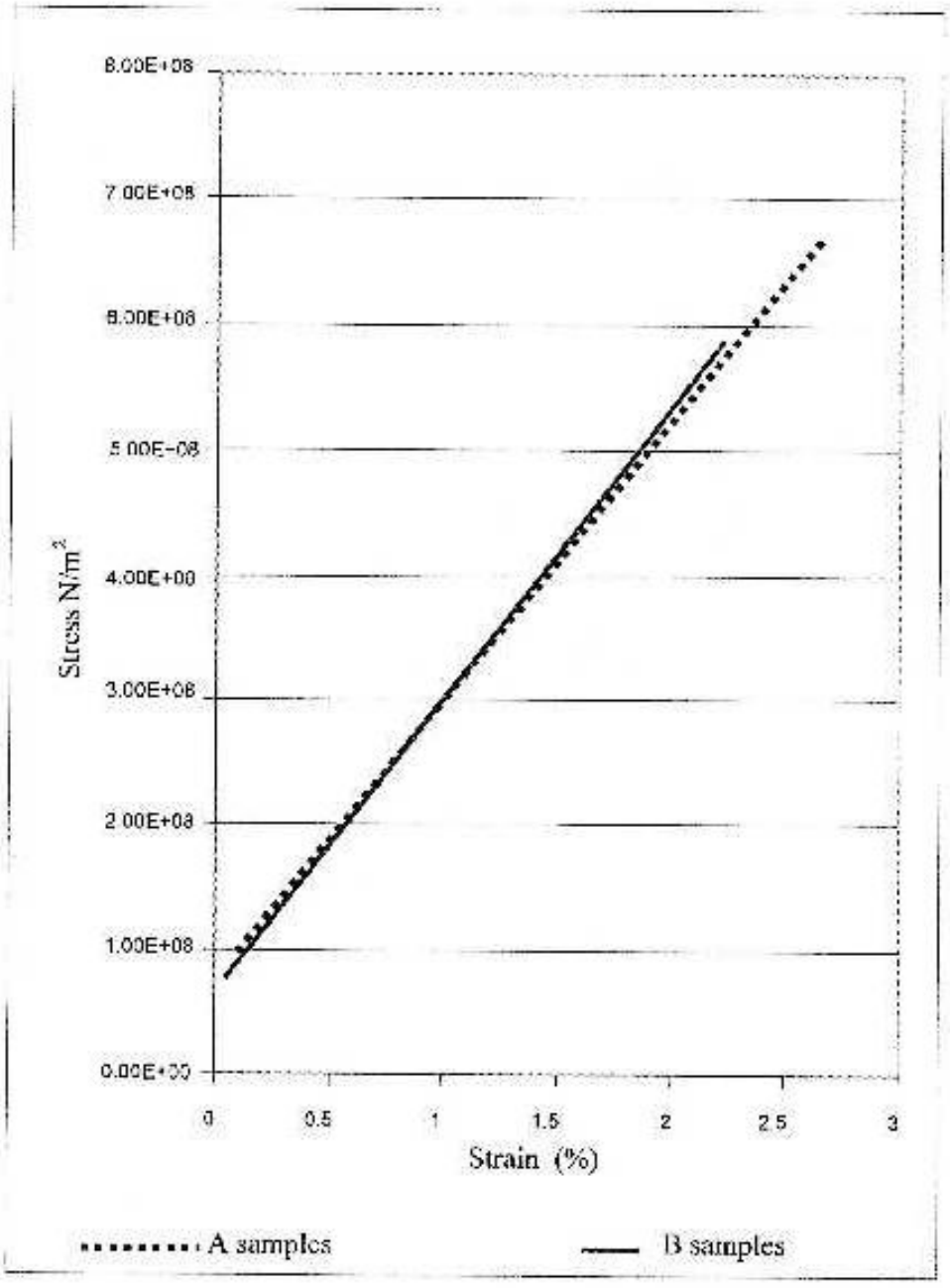

Figure 4: Stress-strain curve of A and B samples

3.0 CONCLUSION

The result of the tests confirmed that the method of removing micro-bubbles prior to vacuum infusion process is very beneficial in controlling the formation of void content thus increase the strength of the composite laminate about 5%. The micro-bubbles filtering process via capillary separation method allows continuous supply of bubble free resin is found to be very versatile for large manufacturing operations which could reduce the formation of micro-bubbles as low as 1%.

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