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on the effect of post-curing temperature of vinylester/e-glass composites with wet hand layup and vartm: an experimental investigation for material characterisation

The overall performance of composites is influenced by the constituent materials, their distribution and their interaction.

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Abstract

The overall performance of composites is influenced by the constituent materials, their distribution and their interaction. The properties of the composite structure are determined by the properties of fibre and resin, ratio of fibre to resin in the composite (fibre volume fraction) and geometry & orientation of fibres in the composite. Quality of the composite end product also depends on the manufacturing process. Composite manufacturing is a complex process requiring an understanding of the various polymers, their chemical reactions, gel time and emissions that may be produced. Low/high level temperature post-curing promotes completion of the cross-linking process of the resin and achieves a higher mechanical stability. The increase of cross-linking density improves the mechanical stability of the material, and relaxation of molecular network can increase its ductility and, thus, the energy absorption during fracture. Post-curing also facilitates large-scale deformation involving a substantial portion of the polymer chains, which enhances fracture toughness. Commonly used post-curing techniques are thermal, microwave, ultraviolet radiation, electron beam and radio-frequency-energy curing.

In this paper, extensive coupon test is carried out to characterise the material properties for E-Glass/Vinylester with Tensile and Flexure tests. Post-curing is done using conventional heated oven. Both stiffness and strength were determined from the experimental investigation. As expected, VARTM specimens had higher tensile and flexure strength compared with the hand laid specimens with higher fibre weight fraction. The actual fibre weights are determined using burn-off tests. Concluding, it was ascertained that with a temperature of 82o for 2 hrs, provided optimum increase in the material properties for E-Glass/Vinylester composite laminate.

Introduction

Vinylester is a thermoset resin which is most commonly used in the composites industry. Vinylester resins have excellent chemical resistance & tensile strength, low viscosity and fast curing. They have a volumetric shrinkage of 5–10 per cent. It is often used in environments that require high strength and corrosion resistance, preferably required in the marine, industrial, energy and infrastructure engineering. Vinylesters are halfway between polyesters and epoxies as far as their typical mechanical properties including toughness. Their longer double bonded vinyl groups that link the ester groups together contribute a flexible nature to the resin. Their flexible nature, produces parts that withstand impacts and repeated flexing without developing cracks. Vinylesters provide excellent resistance to water, organic solvents and alkalis, but less resistance to acids. Vinylesters are also stronger than polyesters and more resilient than epoxies. Both vinylesters and polyesters in their raw form are solid chunks. To turn the polymer into a liquid form, usable by manufactures, a monomer is added. Vinylesters contain high quantities (30-60%) of styrene monomer by weight. Once in the liquid polymer resin state, all that is required is a peroxide initiator (catalyst), to create a chemical reaction which cross-link the polymer molecules. The polyester or vinyl resin will become rock solid, and permanently stay that way, even when heated to high temperatures.

The strength, stiffness, life-cycle, fatigue life and other mechanical properties of composites are directly related to the degree of cure temperature, cure time and type of resin. Post-curing involves subjecting the composite panel/specimen to varying levels of temperature for a certain duration, beyond which the composite starts losing its properties. Post-curing also facilitates large-scale deformation involving a substantial portion of the polymer chains, which enhances the fracture toughness. Different methods of post-curing used are thermal, microwave, ultraviolet radiation, electron beam and radio-frequency-energy curing. Optimised curing duration is essential to achieve maximum permissible mechanical properties.

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Literature Review

Low/high level temperature curing of composites promotes completion of the cross-linking process of the resin and achieves a higher mechanical stability. The increase of cross-linking density improves mechanical stability of the material, and the relaxation of molecular network can increase its ductility and, thus, energy absorption during fracture [Pearson and Yee, 1989]. The optimum level of post-cure temperature and duration combination is often suggested by the manufacturers through technical data sheets. For thermoset-based fibre composites, post-cure has been known to affect the bond strength at fibre–matrix interface [Lu & Hutchinson, 1995; Lindsey & Rudd, 1993]. These studies concluded that post-curing induces residual stresses due to difference in thermal expansion between fibre and matrix, which in-turn decreases the interficial bond strength. The bond strength may be increased by having covalent bonds across the interface in E-glass/Vinylester composites. Karbhari and Lee [2002] focussed on different post-cure temperatures – 400, 700, 800 and 900 C using differential scanning calorimetry. Durability evaluation of moderate temperature cured E-glass/Vinylester systems were investigated [Wellington et al. 2004]. Wonderly et al. [2005] observed explosive failure in the gauge area for E-glass/Vinylester 8084 post-cured specimens for uni-axial tensile test. Comparative study on the analytical and experimental results on uni-axial tensile test for E-glass/Vinylester using VARTM was studied [Rahul Kumar et al., 2005; Jason et al., 2006]. Flexure properties were also tested [Marcia Borba et al., 2009].

Vinylester resins are similar in their molecular structure to polyesters, but differ primarily in the location of their reactive sites, these being positioned only at the ends of the molecular chains. As the whole length of molecular chain is available to absorb shock loadings, this makes Vinylester resins tougher and more resilient than polyesters. The vinylester molecule also features fewer ester groups. These ester groups are susceptible to water degradation by hydrolysis which means that vnylesters exhibit better resistance to water and many other chemicals than their polyester counterparts, and are frequently found in applications such as pipelines and chemical storage tanks. Figure 1 shows the idealised chemical structure of a typical vinylester. Note the positions of the ester groups and the reactive sites (C* = C*) within the molecular chain [SP Composites].

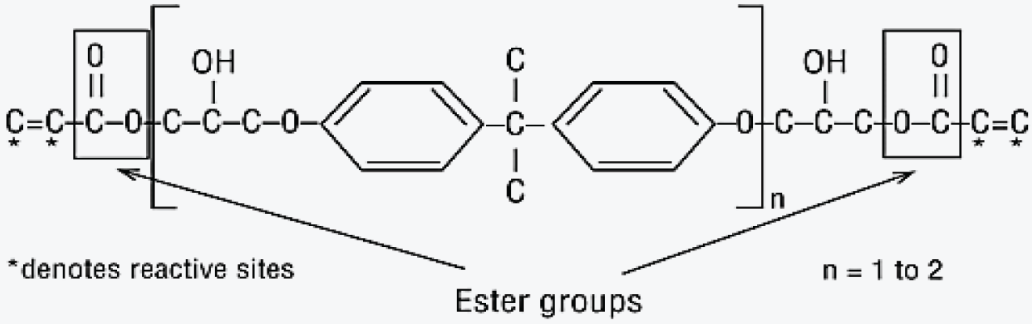

Figure 1: Idealised chemical structure of a typical vinylester resin[SP Composites]

The molecular chains of Vinylester, represented in Figure 2, can be compared to the schematic representation of polyester shown in Figure 1 where the difference in the location of the reactive sites can be clearly seen:

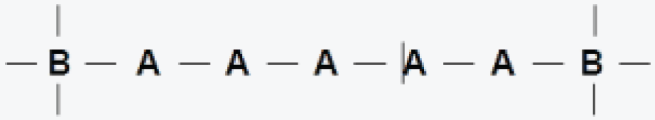

Figure 2: Schematic representation of vinylester Resin (Uncured) [SP Composites]

With the reduced number of ester groups in a vinylester when compared to polyester, the resin is less prone to damage by hydrolysis. The cured molecular structure of the vinylester also means that it tends to be tougher than a polyester, although to really achieve these properties the resin usually needs to have an elevated temperature post-cure [SP Composites].

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Curing at elevated temperatures has the added advantage that it actually increases the end mechanical properties of the material, and many resin systems will not reach their ultimate mechanical properties unless the resin is given this 'post-cure'. The post-cure process involves increasing the laminate temperature after the initial room temperature cure, which increases the amount of crosslinking of the molecules that can take place. To some degree this post-cure will occur naturally at warm room temperatures, but higher properties and shorter post-cure times will be obtained if elevated temperatures are used (Figure 3) [SP Composites].

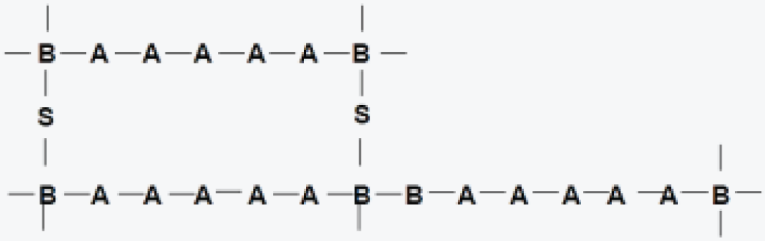

Figure 3: Schematic Representation of Vinylester Resin (Cured) [SP Composites]

In the current study, extensive coupon test is carried out to characterise the material properties for E-Glass/Vinylester. Post-curing is done using conventional heated oven.

- Two types of hardeners– CHM 50 and MEKP 925 H
- Four reinforcement types – Chopped Strand Mat (CSM), Continuous filament mat (CFM), Cross ply double bias (DB) and Uni-directional (UD)
- Two manufacturing techniques – Wet hand layup and VARTM
- Three post-curing temperatures - 20o, 50o and 82o
- Two types of tests – Tensile and Flexure test (3-Point bend test) according to ASTM Standards

Materials

The materials studied in this research were selected to be representative of those used in the construction of composite marine vessels. The specimens were manufactured at Nuplex Composites manufacturing workshop. The materials used were from Fibre Glass International [Nuplex Composites]:

- **Fibre: E-glass**
  - ***Chopped strand mat (CSM):*** 450 gsm – from Owens Corning – OCV M705™ Emulsion Bound - made of randomly oriented chopped glass strands bonded together in mat form using an emulsion binder. The emulsion binder uniquely bonds the mat so that it conforms rapidly to highly contoured moulds. It is a porous, well bonded mat which maintains its integrity during the impregnation process and provides a uniform wet-through rate. The emulsion binder produces superior handling properties compared to powder bonded mats
  - ***Continuous filament mat (CFM):*** 450 gsm - from Owens Corning – Unifilo™ Continuous Filament Mat - consisting of randomly oriented strands in multiple layers held together with a suitable binder and silane coupling agent
  - ***Cross ply double bias (DB):*** (oriented ±45° to the loading axis) 611 gsm - Bi-diagonal stitched E-glass reinforcement
  - ***Unidirectional (UD):*** (oriented 0° to the loading axis) 450 gsm
  - ***Resin system:*** Vinylester SPV-6036™ from Nuplex composites. (3-5% elongation at break; 70-85 MPa Tensile strength; 40-50 min gel time).

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- **Hardener:**
  - ***Norox Methyl ethyl ketone peroxide (MEKP-925H):*** 1.5% by weight - Curing of unsaturated polyester resins and gelcoats in the presence of cobalt accelerator at room and elevated temperatures. This curing system particularly suitable for the curing of gelcoat resins, laminating resins, lacquers and castings; including the manufacture of light resistant parts. Suitable for producing light resistant parts, Low water content and absence of polar compounds
  - ***Norox Cumylhydroperoxide (CHM50):*** 1.5% by weight A comparison between the two hardeners (accelerators) is presented in Table 1

Initiator	Gel time	Peak Exotherm temp. (°F)	Barcol Hardness @ 3 hrs	4 hrs	5 hrs	24 hrs
CHM50	16 min	198	41	50	53	14
925H	13 min	289	28	35	44	53

Table 1: Comparison of the two hardeners [FGI, 2002]

Resin burn-off Test

A resin burn-off test was conducted for DB specimens to determine the fibre weight for hand-layup and VARTM manufacturing processes. The samples were weighed into crucibles, charred using a gas flame, then heated to 750o C for six hrs, cooled and re-weighed. The fibre weights for hand-layup and VARTM were 50.2 and 68.4%, respectively.

Test Methodology

Tensile Test

Tensile specimens were cut for different types of reinforcements. Tabs were manufactured using a three-layer double bias (611gsm) and Vinylester (SPV 6036) resin system. The tabs were glued to the specimens using FGI R045 epoxy resin base and FGI H045 epoxy hardener. The test was conducted as per the ASTM D3039-D3039M-00 standard. The specimens were tested on an INSTRON 50 kN testing machine. Longitudinal deflections were measured using 50 mm Instron extensometer. All specimens were tested under a displacement-controlled setup at the rate of 2 mm/min and the test was terminated when the load dropped to 40 per cent of the previous load increment. Tensile stress was calculated by the ratio of load and cross-sectional area and the Young's modulus was determined by the slope of the tensile stress v. tensile strain plot. The effects of post-curing the laminates at 20°C for 24 hrs, 50°C for 24 hrs and 82°C for 2 hrs were determined.

Flexure test

The flexure properties of the materials were determined by conducting a three-point bend test, carried out as per ASTM D7264-D7264M [ASTM D7264 2007] standards. Displacement-controlled loading was applied at the rate of 1 mm/min. Flexure stress was calculated using: . The flexure modulus was determined by the slope of the flexure stress v. flexure strain plot. The effects of post-curing the laminates at 20°C for 24 hrs, 50° C for 24 hrs and 82°C for 2 hrs were determined.

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Results and Discussion

A number of specimens were tested to determine the tensile and flexure properties of the composite laminate. Tables 2 and 3 present the test matrix for tensile testing while Tables 4 and 5 present the test matrix for the three-point bend test. The effects of three post-curing temperatures (20°C for 24 hrs, 50°C for 24 hrs and 82°C for 2 hrs) on tensile and flexure strength were determined.

Post-curing	CSM925H	DB925H	UD925H
20°C 24 hrs	3	3	3
50°C 24 hrs	3	3	3
82°C 2 hrs	4	4	4

Table 2: Number of Tensile test specimens for hand-layup (HL) process-Test matrix

Post-curing	CFM 925H	CFM CHM50	DB 925H	DB CHM50	UD-90deg CHM50	UD-0deg CHM50
20°C 24 hrs	6	5	4	4	5	5
50°C 24 hrs	0	0	0	4	0	0
82°C 2 hrs	0	0	0	4	0	0

Table 3: Number of specimen Tensile specimen test for VARTM process-Test matrix

Tensile Test Summary

For hand-layup CSM, DB and UD were tested for three different post cure temperatures: 20°C for 24 hrs, 50°C for 24 hrs and 82°C for 2 hrs. Only 925H hardener was used for the hand-layup specimens. Though CSM offers adequate permeability, the binder in CSM may hinder the resin flow. Hence, for the VARTM process, the continuous filament mat (CFM) is generally used in place of CSM. The tensile test results are presented in Tables 6 and 7 for the specimens manufactured by hand-layup and VARTM process. The effects of the two hardeners, MEKP 925H and CHM50, were compared to optimise the VARTM process.

Fibre	Hardener	Post-curing	Tensile Stress (MPa)	Young's Modulus (MPa)	% diff. Stress (MPa)	% diff. Modulus (MPa)
CSM	925H	20°C 24hrs	176.30	9607	0.00	0.00
CSM	925H	50°C 24hrs	144.41	9961	-18.09	3.68
CSM	925H	82°C 2hrs	143.97	10161	-18.34	5.77
DB	925H	20°C 24hrs	75.59	7872	0.00	0.00
DB	925H	50°C 24hrs	115.75	9508	53.13	20.78
DB	925H	82°C 2hrs	97.88	9001	29.49	14.34
UD-0deg	925H	20°C 24hrs	443.81	23576	0.00	0.00
UD-0deg	925H	50°C 24hrs	461.40	22512	3.96	-4.51
UD-0deg	925H	82°C 2hrs	434.86	22849	-2.02	-3.08

Table 6: Hand-layup tensile test summary

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Fibre	Hardener	Post-curing	Tensile Stress (MPa)	Young's Modulus (MPa)	% diff. Stress (MPa)	% diff. Modulus (MPa)
CFM	925H	20°C 24hrs	137.82	8344	N/A	N/A
CFM	CHM50	20°C 24hrs	111.43	7074	N/A	N/A
DB	CHM50	20°C 24hrs	103.18	6703	0.00	0.00
DB	CHM50	50°C 24hrs	123.68	7737	19.87	15.43
DB	CHM50	82°C 2 hrs	119.45	7928	15.77	18.28
UD-90deg	CHM50	20°C 24hrs	72.21	15128	N/A	N/A
UD-0deg	CHM50	20°C 24hrs	747.66	37232	N/A	N/A

Table 7: VARTM tensile test summary

For CSM laminates manufactured by hand-layup, the tensile strength was reduced by 18 per cent for post-curing temperatures of 50 and 82°C; however, the stiffness (Young's Modulus) increased marginally—by 3.68 and 5.77% with post curing at 50 and 82°C, respectively. For DB, post curing at 50°C increased the stiffness by a massive 53.13% and the strength by 20.78%. Surprisingly, for UD laminates, post-curing decreased the stiffness by 4.51 and 3.08% for 50 and 82°C, respectively. The strength increased by a marginal 3.96% for 50°C and decreased slightly (2.02%) at 82°C. These mixed results suggest that post-curing may increase the stiffness for CSM and DB but actually reduce the tensile strength for CSM.

For CFM tensile test specimens manufactured by the VARTM process, the 925H hardener yielded a higher strength (137.82 MPa) compared to the CHM50 hardener (111.43 MPa). The stiffness was also slightly higher for 925H specimens. The effects of post curing were determined for DB specimens for three different post-cure temperatures. The strength increased by 19.87 and 15.77% while the stiffness increased by 15.43 and 18.28% for specimens post cured at 50 and 82°C, respectively. CHM50 hardener (103.18 MPa) yielded higher strength than 925H hardener (95.01 MPa) for DB specimens. Longitudinal (0 deg) and transverse (90 deg) tensile strength of 72.21 and 747.66 MPa were obtained for UD specimens. The detailed results of the tensile test are given in Appendix A. Graphical representation of the tensile strength and Young's modulus for CSM, CFM, DB and UD are presented in Figures 4 to 9.

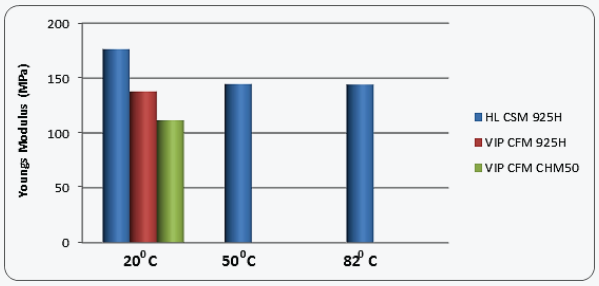

Figure 4: CFM and CSM tensile strength comparison

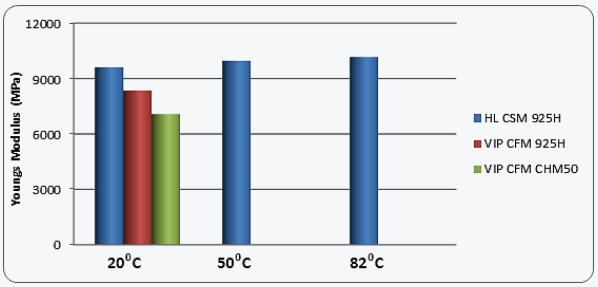

Figure 5: CFM and CSM Young's modulus comparison

Tensile test: Hand-Layup and VARTM Manufacturing Process Comparison

CSM specimens (176.30 and 9607 MPa) yielded higher strength and stiffness compared to CFM specimens (137.82 and 8344 MPa) when cured at room temperature with 925H hardener. However, for CFM with VARTM, CHM50 provided lower tensile strength and stiffness of 111.43 and 7074 MPa, respectively.

DB specimens manufactured by VARTM with CHM50 showed a significant increase in strength (103.18 MPa) but lower stiffness (7074 MPa) compared to those made with the hand-layup process.

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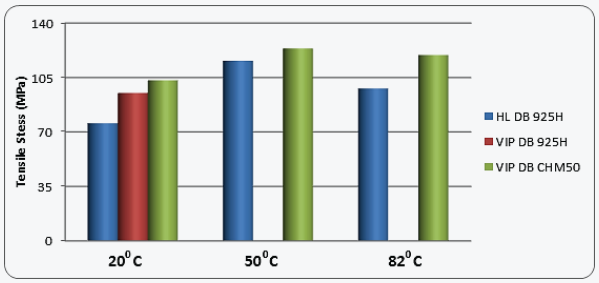

Figure 6: Double bias tensile strength comparison

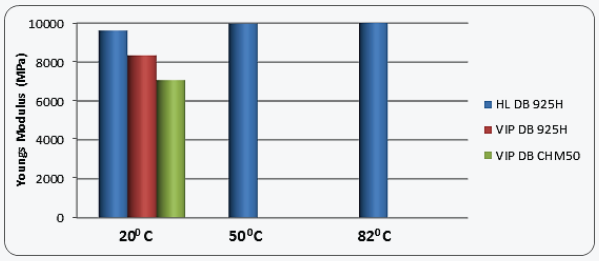

Figure 7: Double bias Young's Modulus comparison

For UD specimens, a huge increase in the strength (68 per cent) and stiffness (58 per cent) was observed by using the VARTM process with CHM50.

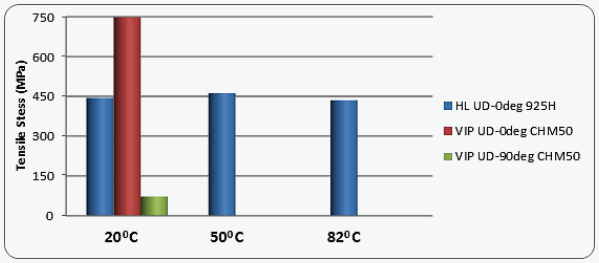

Figure 8: Unidirectional tensile strength comparison

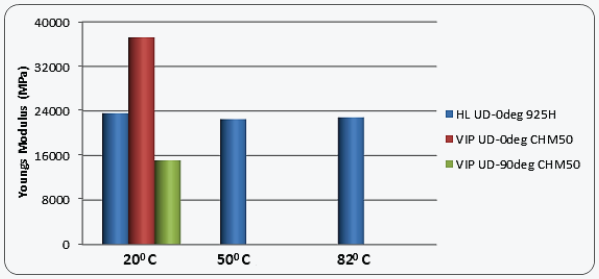

Figure 9: Unidirectional Young's Modulus comparison

The short fibres in CSM layers are dense; they restrict smooth resin flow. Hence a continuous filament mat (CFM) is generally used to ease the resin flow. The major risk in using CFM is that it can wrinkle at bends and corners, resulting in decreased strength and stiffness. The difficulty in resin flow for CSM layers could be reduced by decreasing the amount of catalyst (hardener), thus, increasing the gel time with VARTM process. A significant increase in the tensile strength and stiffness of the laminates with the VARTM process and CHM50 hardener was observed for DB and UD specimens. CSM was preferred for the VARTM process.

Three-point Bend Test Summary

The flexural strength and stiffness of laminates were determined using three-point bend test, in accordance with ASTM D7264. Test samples were cut from the same panel as for the tensile test samples. The span and width of the specimen were 110 and 15 mm, respectively. The results are tabulated in Table 8.

Fibre	Hardener	Post-curing	Flexure Stress (MPa)	Flexure Modulus (MPa)	% diff. Stress (MPa)	% diff. Modulus (MPa)
CSM	925H	20°C 24hrs	222.80	1822	0.00	0.00
CSM	925H	50°C 24hrs	250.74	2356	12.39	29.31
CSM	925H	82°C 2hrs	243.24	2150	9.17	18.00
DB	925H	20°C 24hrs	74.67	1169	0.00	0.00
DB	925H	50°C 24hrs	127.82	1561	71.18	33.53
DB	925H	82°C 2hrs	138.78	1631	85.86	39.52
UD-0deg	925H	20°C 24hrs	436.81	4235	0.00	0.00
UD-0deg	925H	50°C 24hrs	535.31	4407	22.55	4.06
UD-0deg	925H	82°C 2hrs	536.61	4462	22.85	5.36

Table 8: Hand-layup three-point bend test summary

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The flexural stress and modulus for CSM and CFM made with hand-layup and VARTM are presented in Figures 10 and 11. The results are presented in Table 9. The detailed results of the tensile test are given in Appendix B. For 20°C post-cured CSM/CFM specimens, the performance of VARTM with 925H was around 20 per cent greater than that of the hand-layup specimens. Similarly, for post-cured specimens, VARTM with 925H had a 10 per cent increase in strength. Surprisingly, for VARTM with CHM50, the strength was reduced by 30 per cent. On the other hand, CFM with VARTM and 925H had 46, 26 and 42 per cent increases in stiffness for the three post-cure temperatures, respectively. With CHM50, the stiffness increased by 29 per cent. VARTM with CHM50 provided higher strength and stiffness for DB and UD specimens as shown in Figures 12 to 15.

Fibre	Hardener	Post-curing	Flexure Stress (MPa)	Flexure Modulus (MPa)	% diff. Stress (MPa)	% diff. Modulus (MPa)
CFM	925H	20°C 24hrs	265.99	2668	0.00	0.00
CFM	925H	50°C 24hrs	273.34	2966	2.76	11.17
CFM	925H	82°C 2hrs	277.88	3052	4.47	14.39
DB	925H	20°C 24hrs	152.12	1881	0.00	0.00
DB	925H	50°C 24hrs	153.85	1814	1.14	-3.56
DB	925H	82°C 2hrs	171.07	1945	12.46	3.40
CFM	CHM50	200C 24hrs	152.71	2341	N/A	N/A
DB	CHM50	200C 24hrs	176.90	2869	N/A	N/A
UD-90deg	CHM50	200C 24hrs	108.44	2467	N/A	N/A
UD-0deg	CHM50	200C 24hrs	579.01	6748	N/A	N/A

Table 9: VARTM three-point bend test summary

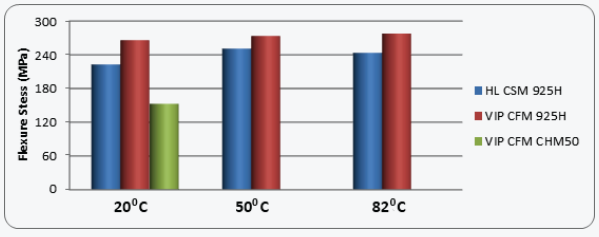

Figure 10: CSM/CFM flexure strength comparison

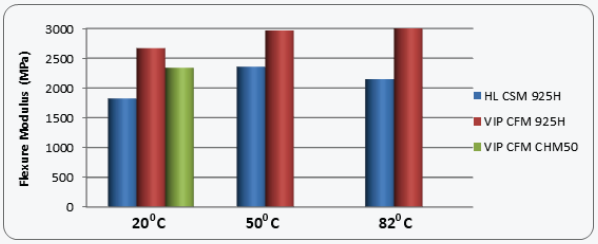

Figure 11: CSM/CFM flexure modulus comparison

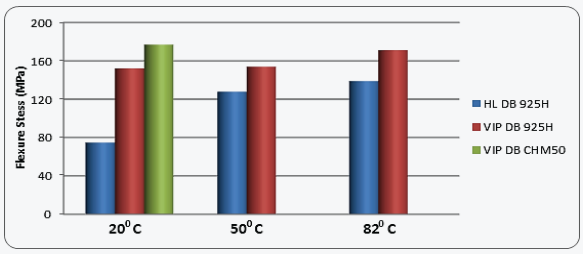

Figure 3.12: Double bias flexure strength comparison

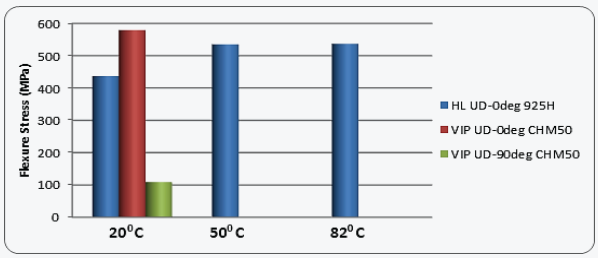

Figure 13 Unidirectional flexure strength comparison

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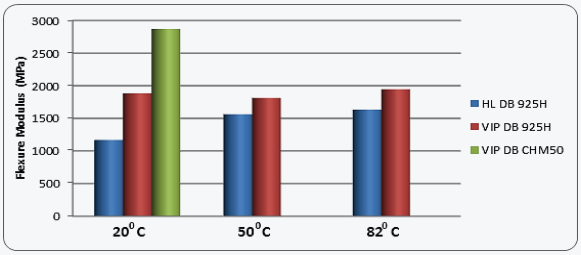

Figure 14: Double bias flexure modulus comparison

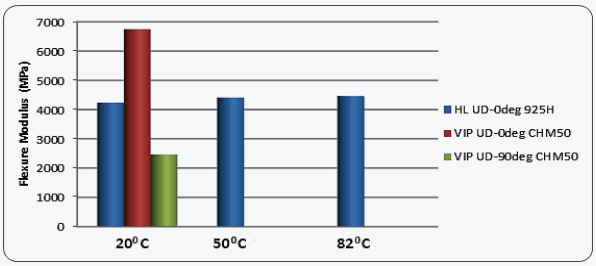

Figure 15: Unidirectional flexure modulus comparison

Conclusions

The two manufacturing processes currently used in the marine industry, hand-layup and VARTM, are discussed in this paper. The specimens were post-cured at three different temperatures-20°C for 24 hrs, 50°C for 24 hrs and 82°C for 2 hrs. Two hardeners were tested: 925H and CHM50. The corresponding weight fractions for Hand layup and VARTM are 50.2% and 68.4%. As expected, the VARTM process provided higher strength and stiffness due to its higher fibre volume fraction; however, large ship structures still must be manufactured by hand-layup, while small and critical structures are manufactured by the VARTM process.

Acknowledgements

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