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14. ABSTRACT

NAVAIRSYSCOM's Materials Division has teamed with fellow Navy, university, and industry partners to demonstrate the viability of resin transfer modeling (RTM) for naval aviation applications. The Polymers and Composites Branch's research involved three major objectives. The first objective was to evaluate the design and performance of the automated RTM equipment designed by Northwestern University (NWU) as part of the Advanced Materials Intelligent Processing Center (AMIPC) program. The second objective was the selection and characterization of materials and processes for RTM of primary structural composites using two-part resin systems. The third objective was to manufacture aerospace quality composites utilizing the selected resin systems and the NWU equipment. The resin injection equipment was designed with the aims of providing increased control and repeatability of material metering, mixing, and delivery. NAVAIRSYSCOM equipment trials have demonstrated these concepts and identified potential areas for improvement. This equipment was used to fabricate a series of test panels using two different two-part resins, studying various factors such as injection flow rates and pressures, cure process parameters, mold styles, and fiber reinforcements. Through physical and mechanical testing, the selected materials have demonstrated processing flexibility and excellent performance.

15. SUBJECT TERMS

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Evaluation of Automated RTM Processes and Materials for Naval Aircraft

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ABSTRACT

The Naval Air Systems Command's (NAVAIR) Materials division has teamed with fellow Navy, university, and industry partners to demonstrate the viability of resin transfer molding (RTM) for naval aviation applications. The Polymers and Composites Branch's research involved three major objectives. The first objective was to evaluate the design and performance of the automated RTM equipment designed by Northwestern University (NWU) as part of the Advanced Materials Intelligent Processing Center (AMIPC) program. The second objective of this research was the selection and characterization of materials and processes for RTM of primary structural composites using two-part resin systems. The third objective was to manufacture aerospace quality composites utilizing the selected resin systems and the NWU equipment. The resin injection equipment was designed with the aims of providing increased control and repeatability of material metering, mixing, and delivery. NAVAIR equipment trials have demonstrated these concepts and identified potential areas for improvement. This equipment was used to fabricate a series of test panels using two different two-part resins, studying various factors such as injection flow rates and pressures, cure process parameters, mold styles, and fiber reinforcements. Through physical and mechanical testing, the selected materials have demonstrated processing flexibility and excellent performance.

KEY WORDS: Resin Transfer Molding (RTM), Epoxy Resin, Equipment and Machinery

1. INTRODUCTION

Currently, Resin Transfer Molding (RTM) is used to fabricate only a small number of composite components for naval aircraft. RTM is a flexible net-shape manufacturing process for fiber reinforced composite components of all shapes, sizes and degrees of complexity. Most RTM processing is performed with single component or premixed resin systems, which are inadequate for structural naval aircraft components. Few aerospace grade resins can be premixed, making RTM of aircraft components more challenging. Recent improvements in RTM processing technology of multi-component resin systems offer great potential to increase the number of RTM-able components on Naval aircraft. Furthermore, RTM will enhance the cost effectiveness of using composites leading to affordable composites for primary structural components

Naval Air Systems Command (NAVAIR), Materials Division (Code AIR-4.3.4) has been supporting the Northwestern University (NWU) Advanced Materials Intelligent Processing Center (AMIPC) program. The overall goal of this program is to demonstrate agile manufacturing, also known as intelligent processing, of composite materials. This is to be achieved by improving Resin Transfer Molding (RTM) using automation. The equipment was designed and manufactured by the NWU AMIPC, led by Dr. Isaac Daniel. Neil Graf and Roland Cochran have led the NAVAIR Materials team evaluating the equipment, materials, and processes. Dr. Peter Joyce from the U.S. Naval Academy has also been assisting NAVAIR Materials in their evaluation efforts. Other participants in the overall AMIPC program are NAVAIR Structures Division and industrial partners Boeing, Production Products, and Packer Engineering.

NAVAIR Materials had three major objectives for this project. The first objective was to evaluate the design and performance of the Northwestern University (NWU) advanced resin injection system. The second goal was the selection and evaluation of materials and processes for study utilizing the NWU system. This included the determination of composite cure kinetics for these chosen materials. The third goal was to manufacture aerospace quality panels utilizing the selected resin systems and the NWU system. Quality would be determined by mechanical testing and other methods.

2. EQUIPMENT EVALUATION

Researchers at Northwestern University (NWU) led by Dr. Isaac Daniel have developed automated RTM equipment with the aim of providing increased control and repeatability of material metering, mixing, and delivery. This technology could potentially yield significant cost savings and a much greater degree of freedom in terms of resin selection for the composites design engineer.

The NWU design was innovative in the fact that separates metering from injection, which allows for independent optimization of each function (1). The system was built on a two-level cart. The bottom shelf contained two syringe-style metering pumps, one for each resin part, that work in tandem. These metering pumps sent the resin through a mixing tube and into a resin holding chamber on the top shelf. Two syringe pumps on the top shelf then reciprocated to inject the mixed resin into the part in a continuous flow. A laptop computer with custom software was used for equipment control and data acquisition.

This equipment had many benefits that addressed issues associated with RTM processing. First, the equipment allowed for mixing of multi-part resins. Second, the metering section operated at low pressure, which resulted in improved pump accuracy. Third, the syringe pumps provide the potential for very accurate metering of multiple component resins. Also, the reciprocating injection pumps provided a continuous resin flow into the mold, which minimized the impact of pump cycling on flow fronts. In addition, computer control eliminated some manufacturing guesswork related to resin mixing and injection and allows for repeatable processing. Finally, the resin holding tank and controlled injection allowed for resin recirculation. This process flowed resin through the mold and back into the holding tank to be injected again. This process could reduce the amount of waste resin, which reduces costs.

Through several "dry run" and clear-topped mold trials, the system successfully demonstrated accurate and repeatable mixing and dispensing of the resin system as well as continuous flow fronts. The system was also used to successfully fabricate multiple flat panels. However, areas of improvement were noted to incorporate into future generations of the equipment. These recommendations included improved reliability and ease of use for the software and improved plumbing design, especially in regards to resin control valves.

3. MATERIALS SELECTION

The second thrust of effort for NAVAIR Materials was selection and characterization of materials. The objective of this work was to select materials for study, and evaluate these selected resin systems for aerospace use. The primary resin system selected was an epoxy resin system, SI-ZG-5A, developed by ATARD Laboratories. This two-part anhydride-cured system was selected based in part on prior VARTM experience at AMIPC teammate Boeing (2). This system was advertised to have properties similar to leading epoxy systems, with the added benefit of additional flexibility in processing. It also provided a 1:1 mix ratio, which aided in initial equipment evaluation. Bryte EX-1510, a two-part cyanate ester resin was selected as a secondary resin for study. This resin was formulated specifically for RTM applications and has excellent thermal and mechanical properties. In addition, its 100:3 mix ratio would also test the equipment's ability to handle extreme ratio differences. These resins are being evaluated for several naval aircraft applications and are compatible with both RTM (resin transfer molding) and VARTM (vacuum assisted RTM) processes. This paper focuses on work performed with the SI-ZG-5A resin system.

The epoxy resin system was characterized thermally using differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and rheological dynamic analysis (RDA). This analysis yielded isothermal and kinetic cure models. The analysis also showed that the resin was flexible in terms of cure cycle as well as minor variations in the mix ratio (3).

For the panel fabrication, plain weave fiberglass and 5-harness satin weave AS4 graphite fabric were selected as the reinforcement for these evaluations. Two mold designs were also used. One was a "flow through" design, with the resin entering the front and exiting the rear of the mold. The second was a "top exit" model, where the resin entered the front of the mold, circulated around the fabric, and exited a port at the center of the top plate.

4. PANEL FABRICATION

The third thrust of effort involved fabrication and characterization of aerospace-quality flat panels using the selected resin systems and the NWU equipment. NAVAIR Structures was to later build upon this work to fabricate complex-shaped parts. The flat panel work was divided into five phases. Phase A consisted of trial panels to get familiar with the resin systems and equipment. No mechanical testing was performed on these panels. Phase B consisted of epoxy panels to investigate basic materials and processing parameters, such as mold design, reinforcement type, and process variations, such as shorter hold times. Phase C used the epoxy system to investigate the effects of mix ratio variances and additional process modifications. Phase D consisted of basic process parameters for the cyanate resin system. Finally, Phase E was to perform a comprehensive trade study of fabrication equipment and methods and quantify the benefits of intelligent processing. The focus of this paper is Phase B.

All panels in Phase B were made using the epoxy resin system and the NWU equipment. Four panels were made with the AS4 five-harness satin carbon fabric, and five panels used Style 7500 plain weave fabric that was several years old. In addition, one panel was made with new 7500 plain weave glass to see if the age of the glass (along with any potential degradation of the fiber or finish) was a factor. See Table 1 for the panel designations and descriptions. The mold used was the "top exit" mold unless noted otherwise.

Table 1: Phase B Panel Designations and Descriptions

Designation	Description
B1	Baseline Carbon
B2A	Baseline Glass
B5	"New" Glass
B2B	Replicate of B2A
B2C	Replicate of B2A
B9	Glass, Intentional Resin-Rich panel
B3	Carbon, Flow-Through Mold
B10	Carbon, Flow-Through Mold with weatherstripping
B6	Carbon, 4 hour hold at 350°F
B7	Glass, lower temperature injection

Prior to injection, the resin parts were degassed in a vacuum oven set to 66°C (150°F) minimum for 2 hours minimum. After degassing, the resin parts were placed in the RTM system and held at 49°C (120°F) minimum. The resin was then mixed and injected into the mold. The mold was in a press set at 40 tons with both platens at 66°C (150°F). Resin was circulated through the mold (but not recirculated) until the resin at the exit contained no air bubbles, usually about 15 minutes. Injection rate was initially set using the software to 40 ml/minute but was reduced accordingly to maintain pressure in the mold at approximately one megapascal (150 psi). After injection was completed, the mold was held at 66°C (150°F) for 30 minutes minimum. After this hold, the press was ramped up to 177°C (350°F) at 1.6°-2.7°C (3°-5°F) per minute and held at 177°C (350°F) for six hours.

However, some variations to the cure process were attempted. Panel B6 used an abbreviated hold of four hours at 177°C (350°F). The thermal analysis of the resin indicated that the hold could be shortened without significant loss of glass transition temperature to verify the thermal data which indicated that the hold could be shortened without reduction (2), but the effect on mechanical properties of the shortened hold was unknown. Panel B7 injected the resin at the lower temperature of 49°C (120°F) and ramped at 1.6° C (3°F) per minute. There was no hold at 66°C (150°F). This was an attempt to improve properties by eliminating the possibility of voids caused by "boil-off" of the resin volatiles when "cold" resin enters a "hot" mold. Processing anomalies during the fabrication of Panel B2 (later B2A) led to replicating that panel with B2B. However, this also had processing anomalies, so it was replicated again as B2C.

Another process variation was the use of silicone foam strips known as weatherstripping. In the flow-through mold, it was imperative that the resin flow through the fabric and not in the gap between the fabric and the mold, an effect called racetracking. Applying weatherstripping along the edges of the mold compresses the fabric, filling the gap between the mold and fabric, and forces the resin through the fabric. Panel B3 was the flow-through mold without weatherstripping, but weatherstripping was added for B10. The majority of the panels were made in the top-exit mold, however. In this mold, the fabric is undersized to allow resin to flow all the way around the fabric, then through the fabric to the exit at the center of the panel. In this case, the racetracking is intentional, and weatherstripping would not be necessary.

5. EXPERIMENTAL

5.1 Evaluation Methods The panels were evaluated and tested using several methods. First, the panels were scanned nondestructively (NDI) using ultrasonic inspection. Then, density measurements were taken on samples from each panel. These measurements were then used to calculate fiber volumes. Finally, mechanical properties were performed on specimens taken from each panel. Mechanical testing performed included tension (ASTM D638), compression (ASTM D 695), short beam shear (SBS) (ASTM D-2344), flexure (ASTM D790), and a four-point shear test (not standardized). In addition, the relatively new combined loading compression (CLC) test (ASTM D6641) was performed where samples were available.

5.2 NDE Results All of the panels were scanned ultrasonically for defects and porosity. The scans showed that the majority of the panels of the same fiber type were equivalent. All of the carbon panel scans indicated no voids or defects. The scans for B1 and B6 were nearly identical, indicating hold time had no effect. Panel B10 had slightly better quality than B3, possibly indicating a benefit to weatherstripping. B1 and B6 were slightly higher quality than B3 and B10, indicating that panels made with the top-exit mold may be slightly better than those from the flow-through mold. However, these differences were minor and may not be significant.

However, the glass panels scanned significantly worse than the carbon panels. The scans of the glass panels repeatedly showed damage across the width of the panel about one-third down the length of the panel. To the eye, this showed up as a white band on the panel. After some discussion and experimentation, the damage was attributed to bending of the panels upon mold removal. The glass fabric, having a lower modulus than the graphite, allowed the panel to bend when removing the panel form the mold. This in turn caused resin breakage and separation of the fiber-resin interfaces. The carbon panel, with higher modulus, did not bend upon mold removal, and therefore no damage was indicated.

In addition, panels B2A and B2B scanned worse than B2C, as would be expected due to the processing difficulties. Panel B9, which was intentionally resin rich, also scanned slightly worse than average. However, B5 and B7 scanned fairly well, indicating minimal if any effect of glass age and lower temperature processing.

5.3 Density and Fiber Volume Results A minimum of 10 specimens were taken from each panel and measured for density using a Mettler Toledo balance and density determination kit. The mean and standard deviation are shown in Table 2. Using the measured density from each sample and using known fiber and resin densities, a fiber volume was calculated for each specimen. The mean and standard deviation fiber volumes are also shown in Table 2.

Table 2: Density and Fiber Volume Comparison

Panel ID	Description	ρ, g/cc	St Dev	FV, %	St. Dev.
B1	Baseline Carbon	**1.545**	0.018	**54.9%**	3.3%
B2A	Baseline Glass	**1.951**	0.005	**54.5%**	0.4%
B5	"New" Glass	**1.985**	0.010	**57.1%**	0.8%
B2B	Replicate of B2A	**1.942**	0.022	**53.8%**	1.7%
B2C	Replicate of B2A	**1.972**	0.026	**56.1%**	2.0%
B9	Glass, Intentional Resin-Rich panel	**1.765**	0.014	**40.0%**	1.1%
B3	Carbon, Flow-Through Mold	**1.561**	0.006	**57.7%**	1.1%
B10	Carbon, Flow-Through Mold with weatherstripping	**1.553**	0.006	**56.3%**	1.1%
B6	Carbon, 4 hour hold at 350°F	**1.583**	0.006	**61.8%**	1.2%
B7	Glass, lower temperature injection	**1.975**	0.018	**56.3%**	1.4%

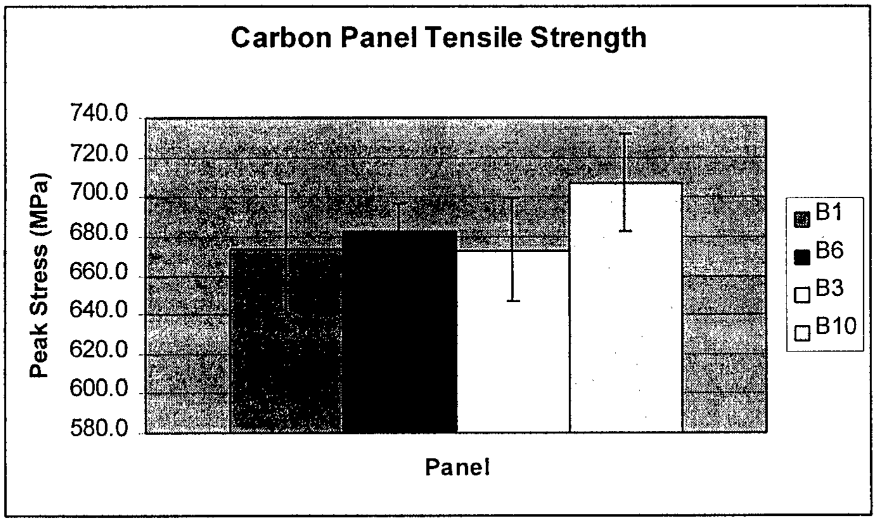
Once again, panels with like reinforcement had similar densities and fiber volumes. Among the carbon panels, panel B1 had seemingly lower density and fiber volume, and higher standard deviations, than the other carbon panels. The reason for this unknown; it may be related to "learning curve" in that B1 was the first Phase B panel made, the first carbon panel made, and the first top-exit mold panel made (all Phase A panels were glass and made with the flow-through mold). Also, Panel B6, with the 4 hour hold, had higher density and fiber volume than the others. Again, the reason for this is unknown. The differences between B3 and B10 are within standard deviations, indicating no significant effect of the weatherstripping.

Panel B9, the intentionally resin-rich panel, obviously had lower densities and fiber volumes than the other glass panels. Also, B2C had higher numbers than B2A and B2B, as one might expect given the processing difficulties with the latter. However, B2C, B5, and B7 were all statistically equivalent, indicating no significant effect of glass age or lower temperature injection.

5.4 Mechanical Property Results-Carbon Panels All mechanical property tests were performed at ambient conditions. Testing was performed at NAVAIR to ISO9001/IEC25 procedures and regulations.

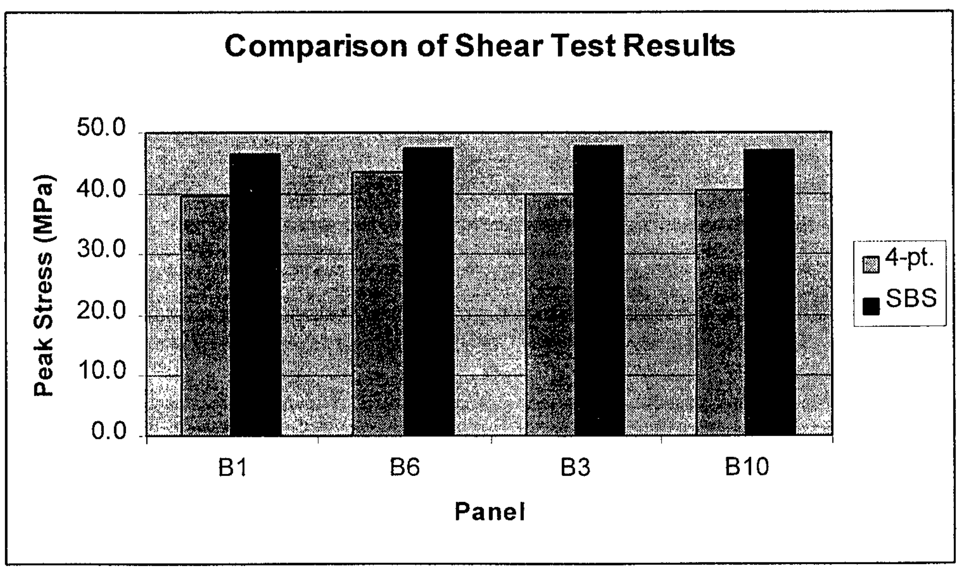
5.4.1 Tensile Properties Six specimens from each panel were tested using ASTM D638. Figure 1 shows a graph of tensile strength for the carbon fabric panels. All four panels showed similar peak tensile strengths. Panel B6 (4-hour hold) was slightly higher than the baseline B1, but well within scatter. Likewise, Panel B10 (weatherstripping) outperformed B3 (no weatherstripping), but the results were within the margin of error. No trend was apparent between mold types.

Figure 1



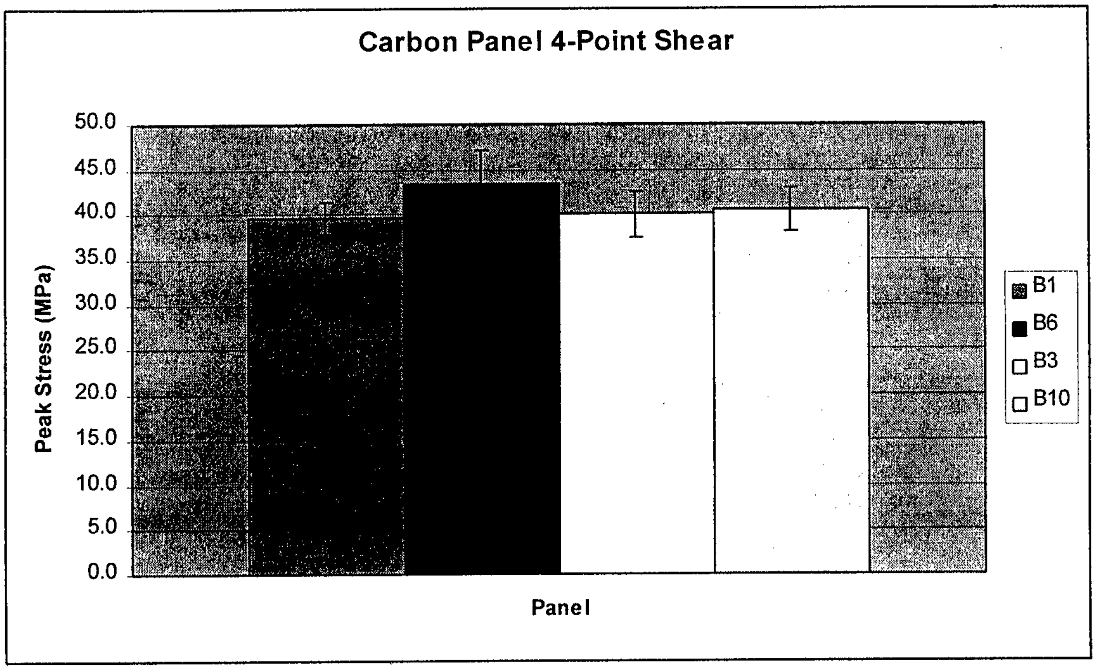
5.4.2 Shear Properties Two types of shear tests were performed. The first was short beam shear. In addition, a four-point shear test was performed on larger specimens (which were the same size as the flexure coupons). The results of both the shear tests are shown in Figure 2. Once again, all of panels showed very similar results. One item of note was that the short beam shear results were consistently higher than the four-point shear tests. This trend is consistent with previous NAVAIR comparisons of the two test types. In both test types, panel B6 (4 hour hold) outperformed the baseline, but within the margin of error. Likewise, neither test indicated significant effects of mold type or the use of weatherstripping.

Figure 2:



5.4.3 Flexure Properties Five samples from each panel were also tested using the ASTM D790 three-point flexure test. The results are shown in Figure 3. Like the other tests, the four-hour hold B6 outperformed the baseline, but within the margin of error. Also like the other tests, no effect of mold type or weatherstripping was observed.

Figure 3



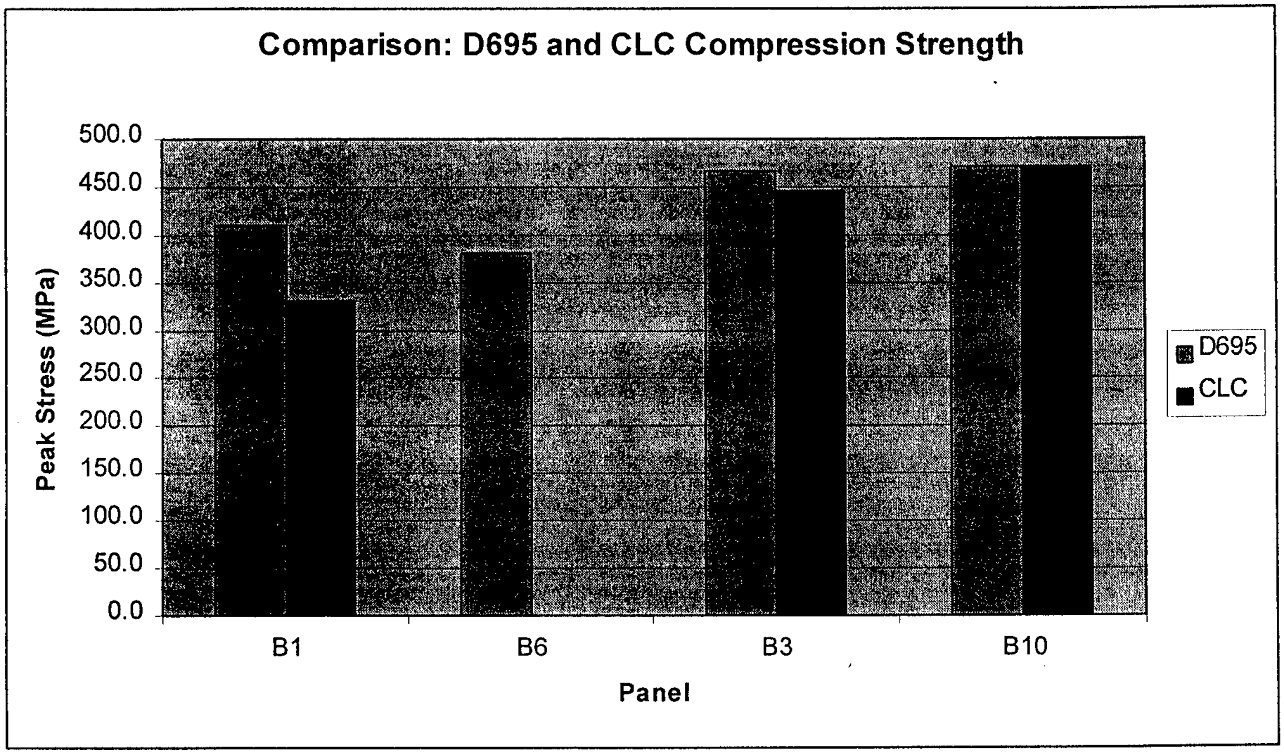
5.4.4 Compression Properties Specimens from each panel were also tested in compression. Standard compression testing using ASTM D695 was performed. In addition, where samples were available, combined loading compression (CLC) is performed. The CLC test is standardized in the recently adopted ASTM D6641.This test, a modification of the End-Loaded, Side-Supported (ELSS) compression test, allows for the combination of end and shear loading of the specimen. In previous testing with materials such as fabric composites, the CLC test has been shown to provide equivalent results to more complex compression tests such as IITRI and Celanese compression tests (4).

During D695 testing, several specimens from each set began to "broom" at the ends. For example, only three of seven specimens for panel B1 provided data. As a result, the decision was made during testing to test the ASTM D695 dogbone specimens in and open-hole compression fixture. This provided more stability at the specimen ends, but made comparison of results more difficult. Also, the CLC test was performed on a material-available basis. No specimens were tested from panel B6, and only two were tested for B1.

Figure 4 shows the results of the compression testing. Due to the reasons noted above, determining the significance of any trends is difficult. However, testing shows higher results for the flow-through mold (B3, B10) compared to the top-exit mold (B1, B6), although the results are well within test error. Similarly, no significant effect of weatherstripping can be determined.

The four-hour hold panel (B6) is lower than the baseline in D695 testing, but again, the appearance of any true trend is muddled due to fixture and number of test specimen variances.

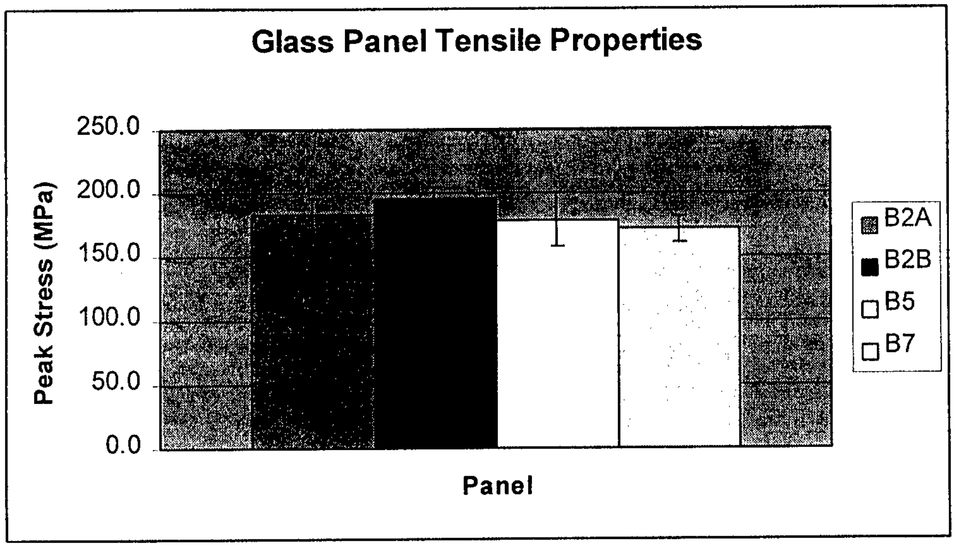
Figure 4



5.5 Mechanical Property Results-Glass Panels Specimens from panels B2A, B2B, B5, and B7 were tested in the same mechanical tests and conditions as the carbon panels. However, results for panels B2C and B9 were not received in time for this publication. Therefore, the effects of process anomalies (B2C vs. B2A and B2B) and resin rich (B9) cannot be made. Comparisons on age of glass reinforcement (B5) and effect of lower temperature injection (B7) may be determined.

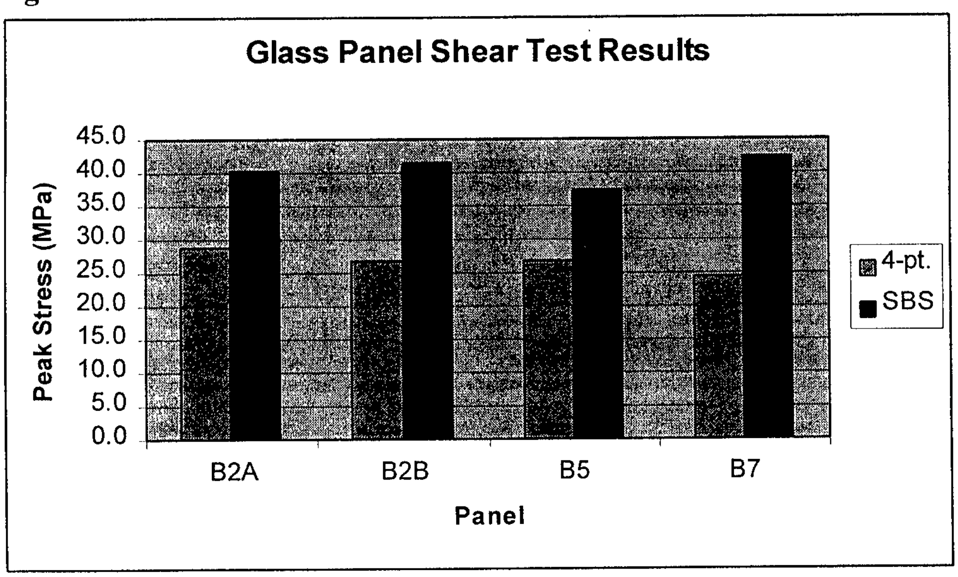
5.5.1 Tensile Properties Figure 5 shows the results of tensile testing of the four glass composites. As was common with the graphite panels, all of the panels had very similar properties. Although there is quite a bit of scatter, B7's tensile strength is slightly lower than that of the others, in particular that of B2B. This could be indicative of a reduction in properties associated with the lower temperature injection. There appears to be no significant correlation between tensile strength and age of the glass reinforcement.

Figure 5



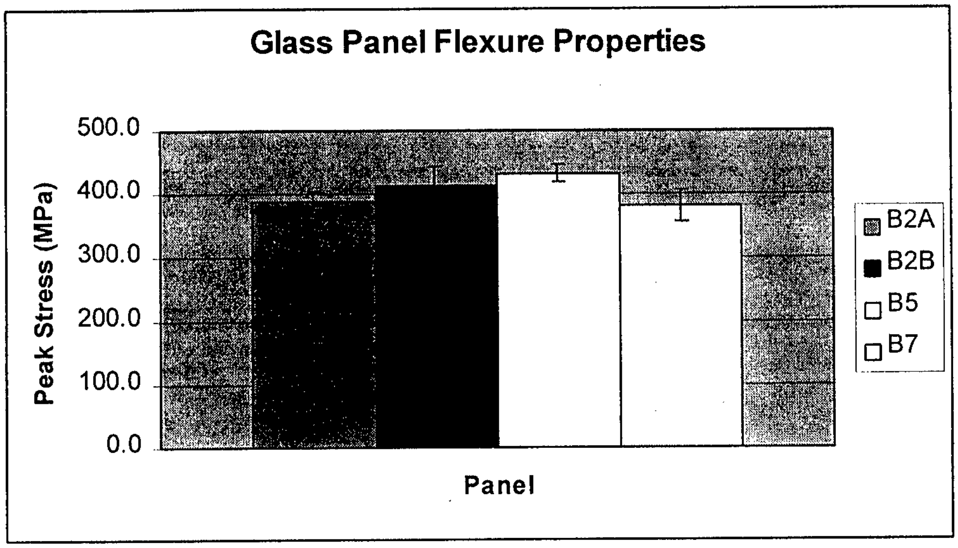
5.5.2 Shear Properties The glass panels were tested for shear both using the short beam method as well as the four-point shear method, similar to the carbon panels. Again, the 4-point shear test shoed lower values than the short beam method. Figure 6 shows a comparison of the shear values. As with the other tests, no significant trends can be determined form these results; all results for each test were within scatter. It is interesting to note that B7 had the lowest four-point shear results but the highest short beam test results.

Figure 6



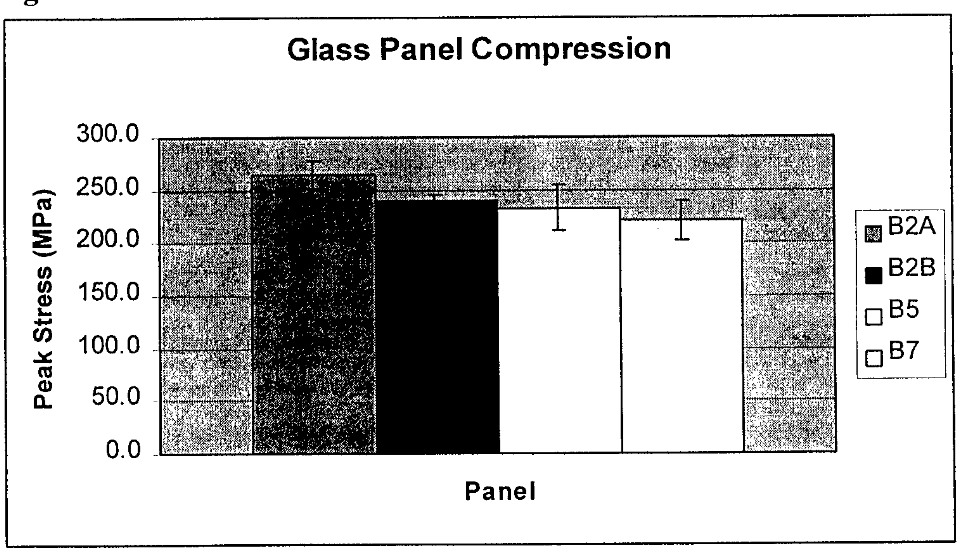
5.5.3 Flexure Properties The flexure test results are shown in Figure 7. Once again, it is difficult to determine any trends, since all of the panels are within one standard deviation of one another. It is interesting to note that B7, as indicated in the 4-point shear and tension tests, is slightly lower than the other panels, although by only a small amount.

Figure 7



5.5.4 Compression Properties Like the carbon panels, specimens from each glass panel were tested in ASTM D695. However, sample "brooming" was not a factor in these tests. Also, the combined loading compression test was performed on a material-available basis. Two specimens from B2B and B7 were tested, while five specimens from B5 were tested. No specimens from panel B2A were available for test. Figure 8 shows the results of the compression testing.

Figure 8



The D695 results show that once again, panel B7 (lower temperature injection) is lower than that of the others, and significantly lower than B2A. In fact, the B2A results are also significantly higher than the B2B results. This contradicts most of the other tests, which possibly show B2B to have slightly better properties than B2A. Once again, the age of reinforcement does not appear to have a significant effect.

6. CONCLUSIONS AND FUTURE WORK

The NAVAIR Materials Division has made significant progress in their work evaluating novel RTM equipment and materials. The NWU equipment, with its computerized control of separate mixing and injection steps, has been evaluated and shown to perform adequately. The two-part epoxy system has likewise been evaluated. Several panels have been fabricated and tested in a variety of ways to determine the effects of various materials and process parameters. These results indicate that factors such as mold design, use of weatherstripping, and age of reinforcement material, have no significant effect. Some possible trends do appear, with an abbreviated hold time possibly seemingly better than the full hold time, and with lower temperature injection seemingly worse than elevated temperature injection. However, additional testing is required to fully explore these possibilities.

Future work includes progressing with Phase C, which investigates mix ratio changes as well as additional process variations. Later phases of wok include investigation of the two-part cyanate ester resin, as well as performing a trade study of various processes to quantify the benefits of intelligent RTM processing. Mechanical testing at naval environments, such as UV and salt-fog exposure, is also likely for these later project phases. Finally, the NAVAIR Structures division has plans to use this resin and equipment to inject complex shaped preforms.

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