THE ELECTRON BEAM CURE OF FIBERGLASS/EPOXY PREPREGS

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ABSTRACT

Recently developed epoxy resin prepregs were electron beam cured and experimentally explored to determine their suitability for use in an aerospace-quality aircraft component. There were two major goals for this program. The first was to determine whether the electron beam-curable prepregs were capable of meeting the requirements of the U.S. Air Force T-38 supersonic jet trainer composite windshield frame. The T-38 windshield frame is currently manufactured using an aerospace-grade prepreg composed of 6781 S-2 woven fiberglass and various 250°F thermally-cured epoxies. The second goal was to develop the lowest cost hand layup and debulk process that could be used to produce laminates with acceptable properties. The laminate properties examined to determine prepreg suitability include laminate mechanical and analytical properties at room and elevated temperatures, prepreg tackiness, prepreg out-time capability, and the debulk requirements needed to achieve these properties. Four resins and three fiber sizings were experimentally examined using this criteria. One epoxy and one fiber sizing were found to have suitable characteristics in each of these categories and were later chosen for the manufacture of the T-38 windshield frame. This experimental study shows that by using low-cost debulk and layup processes, the electron beam-cured prepreg mechanical and analytical properties are in the range of comparable thermally-cured prepregs.

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KEY WORDS: Aerospace; Electron Beam Curing; Epoxy Resins

1. INTRODUCTION

The U.S. Air Force Advanced Composites Program Office (WL/MLS-OL), McClellan AFB, Sacramento, CA recently sponsored a project which studied the feasibility of electron beam (E-beam) curing the T-38 composite windshield frame. The T-38 is the U.S. Air Force's primary supersonic trainer and the composite windshield frame is an integral structural part of the windshield assembly system rated to withstand a 1.8 kg bird impact at 740 km/hr. The first step in the design effort for this windshield frame involved a prepreg materials and process optimization study using S-2 fiberglass fabric laminates incorporating a series of different electron beam-curable cationic epoxy resin systems and compatible S-2 fiberglass fabric finishes. The primary goal of this program was to develop materials that met the windshield frame's requirements, while minimizing layup time before their vacuum bag cure using an E-beam accelerator.

Electron beam curing is a non-thermal, non-autoclave curing process which potentially offers many favorable advantages compared to thermal curing methods. Electron beam curing has been demonstrated to be capable of very high rates of material processing. This technology can potentially be used to manufacture quality composite parts rapidly and inexpensively at selectable temperatures (ie., room temperature, sub-ambient) using only vacuum pressure. The cost of tooling for some composite parts may also potentially be reduced. When fully developed, it may also eliminate slower, high-temperature, high-pressure curing cycles, harmful chemical hardeners, and volatile emissions that are routine in traditional thermal curing processes. Additionally, it gives the fabricator the ability to manufacture unique products that cannot be built any other way. These include composite parts or tools having little, if any, residual stresses, and composite parts incorporating materials or components which are temperature- and/or pressure-sensitive. A major goal of this program was to determine if these benefits can be realized for a part such as the T-38 composite windshield frame, which has been in production using thermal cure methods for well over three years.

In 1994, a Cooperative Research and Development Agreement (CRADA), sponsored by the Department of Energy Defense Programs and 10 industrial partners, was established to advance electron beam curing of composites. The CRADA successfully developed hundreds of new, toughened and untoughened electron-beam-curable cationic epoxy resin systems, offering a vast formulation and processing flexibility (1-5) [several patents are pending.] Carbon-fiber-reinforced composites made from these easily processable, low-shrinkage materials were found to match the performance of thermally-cured composites in many areas and were shown to exhibit: low void contents comparable to autoclave cured composites (less than 1%); superb low water absorption values in the same range of cyanate esters (less than 1%); glass transition temperatures rivaling those of polyimides (greater than 390°C); mechanical properties comparable to high performance, autoclave cured composites; and excellent property retention after cryogenic and thermal cycling. These materials have been used to manufacture many composite parts using various fabrication processes including hand layup, tow placement, filament winding, resin transfer molding, and vacuum assisted resin transfer molding.

The project described herein successfully transitioned the previous resins and process work, developed within the CRADA, and further optimized their use for electron beam-cured S-2 fiberglass fabric materials.

2. EXPERIMENTAL

2.1 Electron Beam-Curable Cationic Epoxy Resin Systems The following proprietary resins were formulated by Oak Ridge and the Atomic Energy of Canada, Ltd in support of this program: 1) 3HS - a toughened epoxy resin system, 2) 1LO - a high-temperature toughened epoxy resin system, 3) 3K - a toughened epoxy resin system, 4) 1MO - a high-temperature epoxy resin system.

2.2 S-2 Fiberglass Fabric Sizings The following fabric sizings from JPS Fabrics Corporation in Slater, SC were selected for use in combination with the electron beam-curable cationic epoxy resins discussed above:

1) Z-6040 Sizing: An epoxy functional silane finish applied to heat-cleaned fabrics
2) S-550 Sizing: A high-strength finish designed for both wet layup and prepreg applications. This finish features both dual epoxy and polyester resin functionality.
3) S-977 Sizing: A proprietary silane finish that is compatible with epoxy, polyester, phenolic, melamine and some polyimide resin systems.

2.3 Electron Beam-Curable Cationic Epoxy, S-2 Fiberglass Fabric Prepregs The electron beam-curable cationic epoxy resins and S-2 fiberglass fabric sizings outlined above were used in combination with S-2 fiberglass fabric, Style 16781 (equivalent to Style 6781), to manufacture several hot-melt prepregs at YLA, Inc., Benicia, CA. Style 6781 is an 8-harness satin weave fabric supplied by JPS Glass Fabrics Corporation. It has a nominal cured ply thickness of 0.0254 cm and a fiber areal weight of 300 g/m^2. Ten different S-2 fiberglass fabric prepregs were manufactured at YLA, Inc. and are listed in Table 1.

2.4 Manufacture of Electron Beam Cured Cationic Epoxy Resin Woven Laminates All of the prepregs listed in Table 1 were laid up (warp-to-warp) using various different layup and debulk techniques. All of these techniques were evaluated for their ease of use as well as for the properties of the laminates that they produced. A synopsis of these layup techniques is given in Table 2. Debulks were conducted using vacuum bag pressure only (and heat blankets where noted). Unless otherwise noted, all of the laminates were cured using three passes under the accelerator at 50 kGy/pass, for a total dose of 150 kGy.

2.5 Manufacture of Autoclave Cured Epoxy Resin Woven Laminates The prepreg currently used to fabricate the T-38 composite windshield frame is 919/6781 from Cytec Engineered Materials, Anaheim, CA. This material is an epoxy-based, S-2 fiberglass fabric prepreg that is autoclave cured at 121°C and 0.31 MPa for 4 hours (7.5 hr. for all stages).

2.6 Property Testing of Electron Beam Cured Cationic Epoxy Resin Woven Laminates Several mechanical and analytical tests were conducted by the Physical Sciences Laboratory at McClellan AFB, CA to evaluate the electron beam-cured cationic epoxy prepregs. The following

properties were measured: percent void content (ASTM D 2734), percent resin and fiber content (V_f) (ASTM D2584), 0° tensile strength and modulus properties (ASTM D 638), 0º compressive strength and modulus properties (ASTM D 695), 0º flexural strength and modulus properties (ASTM D 790), and short-beam shear strengths (ASTM D 2344) for several of the prepregs. Unless otherwise noted, all properties were measured at room temperature under dry conditions. The experimental error for each of these tests was estimated to be: percent void content (±15%), percent resin and fiber content (±4%), 0° tensile strength and modulus properties (±3.5%), 0º compressive strength and modulus properties(±3.5%), 0º flexural strength and modulus properties (±4.5%), and short-beam shear strengths (±5.0). To facilitate comparison between the electron beam- and thermally-cured prepregs, all the testing was completed at McClellan AFB utilizing the same technicians. Unless otherwise noted, all data points in this paper represent an average of 5 coupons tested.

2.7 Dynamic Mechanical Analysis (DMA) DMA was performed using a Rheometric Solids Analyzer RSA II. The composite samples were trimmed to fit a dual cantilever tool, with an initial stress/strain of zero. The applied strain used for measuring the rheological properties was 0.1%. The temperature was raised at 2.5°C/step, with a dwell time of 15 seconds. The frequency of the applied strain was 6.28 radians/second. Sample size was normally 3mm x 7mm x 55mm. Measurements were obtained using the software provided by Rheometrics.

2.8 Electron Beam Accelerator All electron beam irradiations were performed at the Whiteshell Laboratories of AECL using the I-10/1 Electron Accelerator (10 MeV; 1 kW). The instantaneous dose rate was 1.5 MGy/s.

3. RESULTS AND DISCUSSION

As stated in the introduction, one of the primary goals of this program was to develop low-cost layup and debulk techniques that could be used to manufacture woven fiberglass prepreg laminates with properties similar to those currently obtained using thermally-cured resins. Figures 1 and 2 show the void content and fiber volume fraction as well as the tensile, compressive, and flexural strength properties of Prepreg J (see Table 1 for a listing of all prepreg materials) using 5 different layup and debulk procedures (as listed in Table 2). Each of the laminates was cured using a dose of 150 kGy. Figure 1 shows that the various layup procedures produced electron beam-cured laminates with fiber volume fractions between 37% and 45%. The void content for most of these laminates was between 3%-5%. Consistent with the objectives of the program the layup techniques that involved the simplest methods (P-1, P-4, (and later P-10)) were chosen for further analysis. Figure 2 demonstrates that some of the simplest layup techniques - plastic rubbing block with room-temperature vacuum debulks - were as effective as the more time-consuming warm debulk methods.

Prepreg A was chosen for the next set of tests because it was believed to have improved properties over Prepreg J. It was laid up by a USAF mechanic using a hand-held plastic rubbing block while using similar force and duration to manually compact the plies as is typically used to lay up an autoclave-cured prepreg. Three processes were used: 1) P-1, which utilized no debulks until the entire stack of eight plies was laid up, 2) P-4, which utilized a 15-minute room temperature vacuum debulk after every four plies, and 3) P-10, which utilized a 15-minute

vacuum debulk at 43°C after every four plies. Once again, Figure 3 shows that there was very little difference in the mechanical properties of the laminates that were laid up using the different techniques. For this reason, the P-4 process was chosen because it was the simplest process that was also thought to be capable of allowing full compaction of laminates - especially when used on real-world parts with curved surfaces.

Using a hand-held plastic rubbing block and 15-minute vacuum debulks every four plies (procedure P-4 was used for Prepregs A-F and the warm-debulk process, P-10, was used for Prepregs G-I to improve the flow of their resin), a USAF mechanic laid up eight-ply laminates of Prepregs A-I. Prepregs B, C, D, F, H, and I were found to have satisfactory fiber volume and void fractions as shown in Figure 4. Figure 5 shows that Prepregs C, D, F, and H had the best tensile, compressive, and flexural strengths. The room temperature tensile strength of Prepregs C, D, and F were found to be statistically equivalent to the 919/6781 prepreg. The compressive and flexural strength of these prepregs was measured to be 20%-30% lower than that of the 919/6781 prepreg. The tensile, compressive, and flexural moduli of each of these four E-beam cured prepregs were found to be statistically equivalent to that of the 919/6781 as shown in Figure 7. Figure 6 shows that the room temperature mechanical properties of prepregs A, B, E, G, and I are significantly lower than those of the 919/6781 prepreg. Figure 8 shows the moduli of these E-beam cured prepregs are also comparable to that of the 919/6781 prepreg. In general, Prepregs C, D, and F were found to have the best mechanical properties of those tested in this program.

Additional prepreg properties such as handling characteristics, out-time capability, and room temperature flow were considered in choosing a prepreg that would be used to manufacture the prototype composite windshield frames. The 1MO resin (prepregs G-I) was found to be very sensitive to the fluorescent lights within the layup room, actually undergoing partial cure during the layup. In the future, special filtered lighting could be used if this resin is utilized, but for this program that was not considered to be an option. As formulated, the 3K resin was found to be aggressively tacky, which made the layup of even the flat test panels difficult. This tack problem could certainly be adjusted in future formulations without adversely affecting the properties of the prepreg. This may be advisable, since Prepreg F was found to have the best mechanical properties of any of the E-beam cured prepregs. The 1LO resin (Prepregs A-C) was found to have a suitable out-time (at least a few days under fluorescent lighting) and to have tack very similar to that of the 919 resin (which has been optimized after years of testing and production usage). Prepreg C had the best mechanical and analytical properties of any of the 1LO prepregs (just slightly less, however, than Prepreg F) and for these reasons was chosen as the program's "optimum" prepreg.

Further laminates were laid up by a USAF mechanic utilizing Prepreg C, a plastic rubbing block, and the two competing layup procedures (P-4 and P-10). Figure 9 shows that the room temperature, vacuum debulk layup procedure, once again, produced laminates with a lower void content. The data presented in Figures 10 through 15 is normalized to V_f =46% to facilitate direct comparison between the different processes and materials.

Figure 10 shows the mechanical properties of Prepreg C tested at 25°C and 71°C after being laid up using procedures P-4 and P-10. It can be seen that the room temperature vacuum debulk

procedure (P-4) produces laminates with mechanical properties equivalent to those that were manufactured using the elevated temperature debulk scheme (P-10). Figure 10 also demonstrates that there is very little drop off in mechanical properties for prepreg "C" between the temperatures of 25°C and 71°C. Figure 11 shows that the two layup techniques produce laminates with very similar moduli.

Figures 12 through 15 show Prepreg C properties after being laid up and vacuum-pressure debulked using procedure P-4. Throughout these figures, there are two sets of data shown for Prepreg C. Both sets were laid up using P-4. They were, however, laid up and tested in separate instances and therefore represent two distinct tests. Also, each "Test 2" data point represents the average of 15 coupons whereas each "Test 1" data point represents 5 coupons. The room temperature (25°C) mechanical properties of Prepreg C are shown in Figure 12. The tensile strengths of Prepreg C are equivalent-to-just-slightly-less than the 919/6781 prepreg. However, the compressive and flexural strengths are 25% to 35% below that of the corresponding properties of 919/6781. Figure 13 shows that similar results are found when the laminates are tested at 71°C. The tensile strength of Prepreg C is nearly equivalent but the compressive and flexural properties are 30% to 35% below that of 919/6781.

Figures 14 and 15 demonstrate that the fiber-dominated modulus properties of Prepreg C are statistically equivalent to that of 919/6781. Stiffness properties are sometimes thought to be of a secondary nature to strength properties of a composite material but numerous aerospace designs are "stiffness-critical" rather than "strength-critical". This is true for the T-38 composite windshield frame, where excessive deflections during the bird-impact can cause the windshield assembly to fail prematurely.

The fiberglass sizing can have an affect on the electron-cure of the resin. If the sizing is basic in chemical reactivity, the sizing will inhibit the cure of the resin because the initiation of the resin is acidic in nature. As a rule of thumb, there should be no, or minimum, transition peaks below the α transition for the loss modulus (E") and tan(δ) curves. These peaks usually are indicators of uncured resin, present only after inhibition of the cure process takes place. Table 3 gives a summary of the DMA results for all the resin/sizing combinations. Figure 16 shows the prepreg "C" DMA curves. The tan(δ) curve shows several peaks at 113, 237 and 400°C. The peak at 113°C indicates there may be a slight inhibition in the resin's cure. This slight inhibition does not appear to affect the flexural modulus (E'). The peak at 235°C is the glass transition temperature (T_g) for the toughener used in the formulation. The last peak at 400°C indicates the T_g for the epoxy used in the formulation. The intensity of the peak at 235°C, suggests an indication of a phase separation of the toughener and the epoxy.

The DMA curves for Prepreg C and 919/6781 are shown in Figure 17. The flexural modulus (E') is shown for each prepreg. The temperature at which the flexural modulus (E') is reduced to 50% of its 25°C value is defined as the service temperature. The 919 prepreg's service temperature is estimated to be 143°C. The service temperature for Prepreg C is estimated to be 224°C or greater, considerably higher than that for the 919 prepreg.

Samples of Prepreg C were irradiated at several doses to determine the minimum cure dose. The χ peak on the tan(δ) curve was used. Figure 18 shows that the χ peak intensity levels off after about 100 kGy. 100 kGy was, therefore, considered a minimum prepreg cure dose.

The high-temperature capability of these prepregs was also explored by using the short-beam shear test. The prepregs were once again laid up by a USAF mechanic using process P-4. Figures 19 and 20 show that Prepregs F, H, and I all have very high temperature capabilities. The properties do drop off significantly from that at room temperature but this is similar to numerous BMI and polyimide resins that are currently in use throughout the aerospace community. The figures show that the 1MO resin (Prepregs H and I) seems to have better short-beam shear properties at the highest temperatures explored (200°C - 315°C).

Figure 21 shows the effect of "over-dosing" the prepreg. Here Prepreg C was cured using doses from 250 kGy to 1000 kGy. The short-beam shear strength of the prepreg is found to slightly decrease over the dose range but to still hold a significant portion of its room temperature strength. This fact could be very important in applications where certain portions of a structure are exposed to more than the typically-intended dose of 150 kGy.

4. CONCLUSIONS

This program developed electron beam-curable prepregs utilizing four different electron-curable resins, three fiber sizings, and a 6781 fiberglass fabric. Initially, ten different layup and debulk procedures were screened for their suitability towards meeting the program's goals. The goal was an efficient process that would produce electron beam-curable laminates with the material properties required for the T-38 composite windshield frame. It was determined that a hand pressure layup and room temperature vacuum bag debulk every four plies produced laminates with properties as good as, if not better than, the other more elaborate layup and debulk processes and met the goals of the program.

Using this process, laminates were produced that had void fractions in the range 1%-4%. Three of the prepregs studied generated laminates with tensile strengths statistically equivalent to the thermally/autoclave-cured 919/6781 prepreg. The compressive and flexural strengths of these E-beam prepregs were measured to be in the range of 20%-30% lower than those of the 919/6781 prepreg. The moduli for the best electron beam-cured prepregs were equivalent to or better than that of the 919/6781 prepreg. The T_g's of all the electron beam-cured prepregs were significantly higher than the 919/6781 prepreg T_g. These high T_g's were demonstrated through the DMA testing as well as the short-beam shear testing for prepregs F, H, and I.

The high temperature properties of these E-beam resins strongly suggest two things. First, their properties should be compared to other high-temperature, aerospace-grade prepregs (this is being done in other programs) to determine if they may have the very important high-temperature capabilities that many aerospace systems require. The electron beam-cured prepregs would have the significant advantage of requiring only a room-temperature vacuum bag cure whereas BMI's and polyimides require cure temperatures well above 177°C. Secondly, the data suggest that there is sufficient high-temperature capability within the E-beam curable resins that additional

"tougheners" could be added to improve the lower-temperature (below 177°C) properties which are important to a large community within the aerospace industry.

5. ACKNOWLEDGEMENTS

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Prepreg	Resin	Fiber Sizing
A	1LO	S-550
B	1LO	S-977
C	1LO	Z-6040
D	3K	S-550
E	3K	S-977
F	3K	Z-6040
G	1MO	S-977
H	1MO	S-550
I	1MO	Z-6040
J	3HS	Z-6040

Table 1 Cationic resins and fiber sizings used for prepregs labeled A-J.

Process	Description
P-1	Lay-up: By hand using a plastic rubbing block
	Debulk: No debulks
P-4	Lay-up: By hand using a plastic rubbing block
	Debulk: 15 minute Room Temperature debulk after every four plies
P-6	Lay-up: By hand using a plastic rubbing block
	Debulk: 15 minute Room Temperature debulk after every two plies
P-7	Lay-up: By hand using a plastic rubbing block
	Debulk: 30 minute Room Temperature debulk after 4 plies, 71C after 8 plies
P-8	Lay-up: By hand using a plastic rubbing block
	Debulk: 30 minute 71C debulk after every four plies
P-10	Lay-up: By hand using a plastic rubbing block
	Debulk: 43C Debulk for 15 minutes after every four plies of prepreg

Table 2 Layup and debulk techniques used to manufacture the prepreg coupons.

Resin	Sizing	Service Temp. °C	Glass Transition Temperature, °C Loss Modulus χ peak	β peak	α peak	Tan(δ) χ peak	β peak	α peak
1L0	S-550	219	106	218	376	106	239	388
1L0	S-977	242	93	227	379	101	235	400
1L0	Z-6040	224	113	222	376	113	237	400
3K	S-550	204	100	184	380	101	212	398
3K	S-977	187	--	185	383	--	207	398
3K	Z-6040	182	103	197	380	103	212	395
1M0	S-550	295	143	338	376	141	346	400
1M0	S-977	313	--	--	348	--	--	400
1M0*	Z-6040	100	--	--		96	88	
3HS	Z-6040	159			169			176
919		143		65	154		65	161

Note: * - indication of thermal-curing taking place at temperatures higher than 113°C.

Table 3 Glass Transition Temperatures of the prepregs.

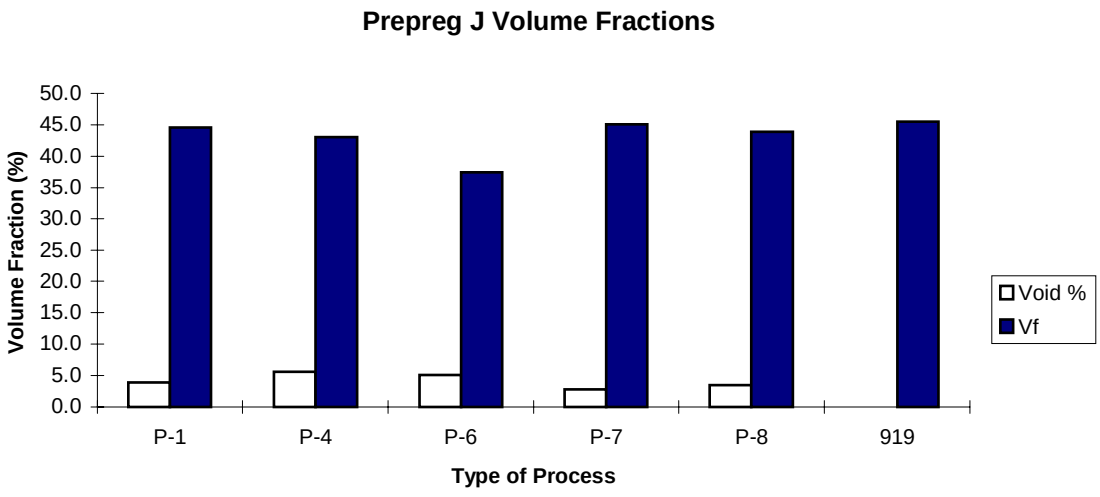

Fig. 1 Fiber and void fractions of Prepreg J using various lay-up techniques.

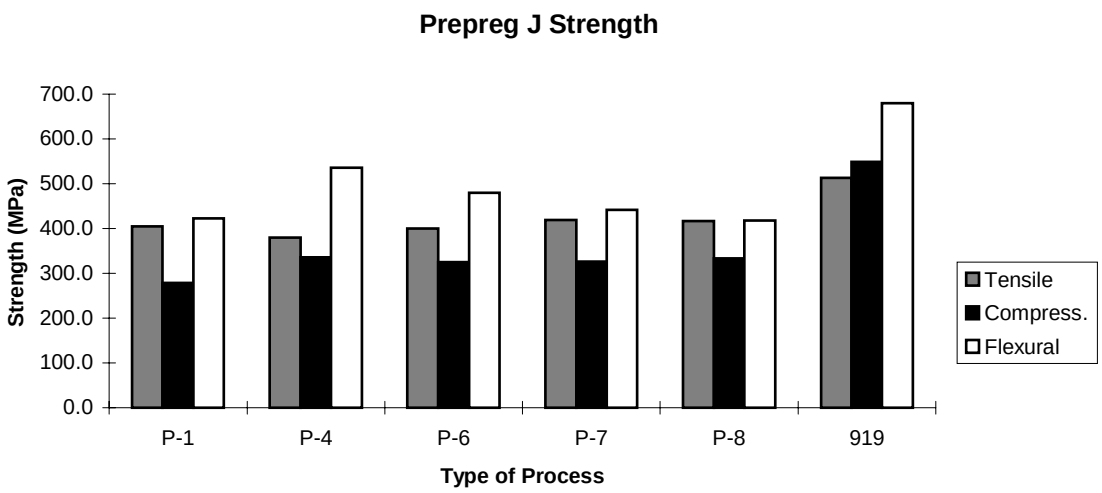

Fig. 2 Strength of Prepreg J using various lay-up techniques.

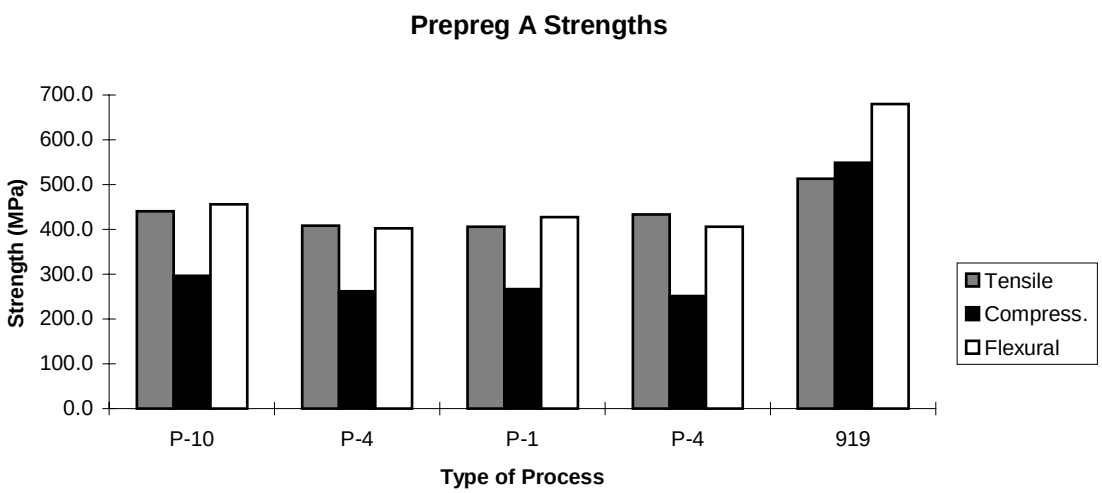

Fig. 3 Strength of Prepreg A using various lay-up techniques.

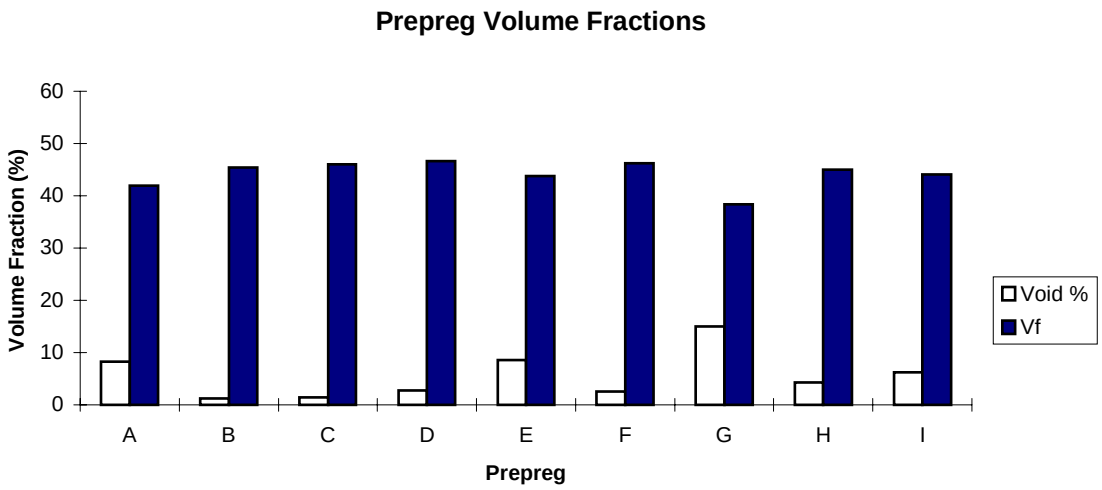

Fig. 4 Fiber and void fractions of the E-beam prepregs compared to the 919 prepreg.

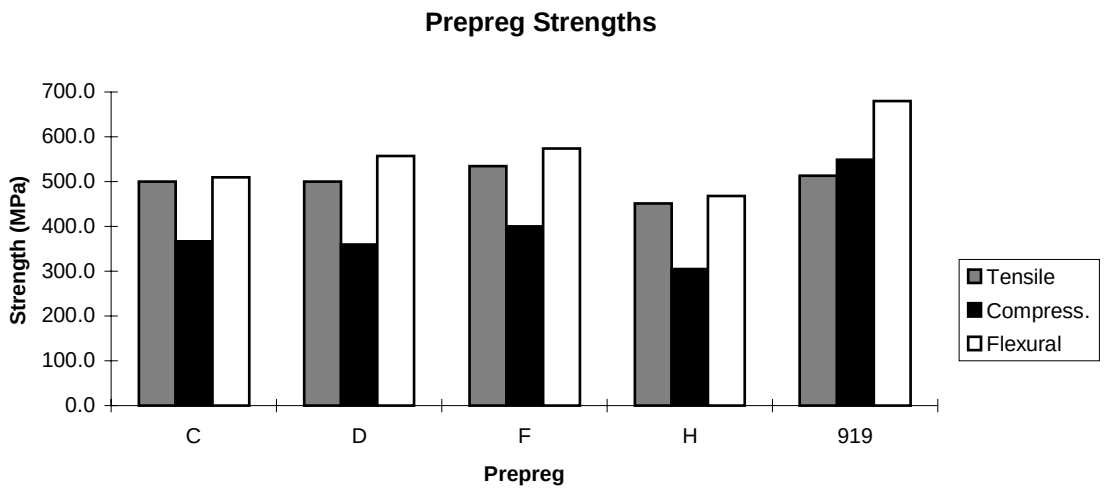

Fig. 5 Strength of the E-beam prepregs compared to the 919 prepreg.

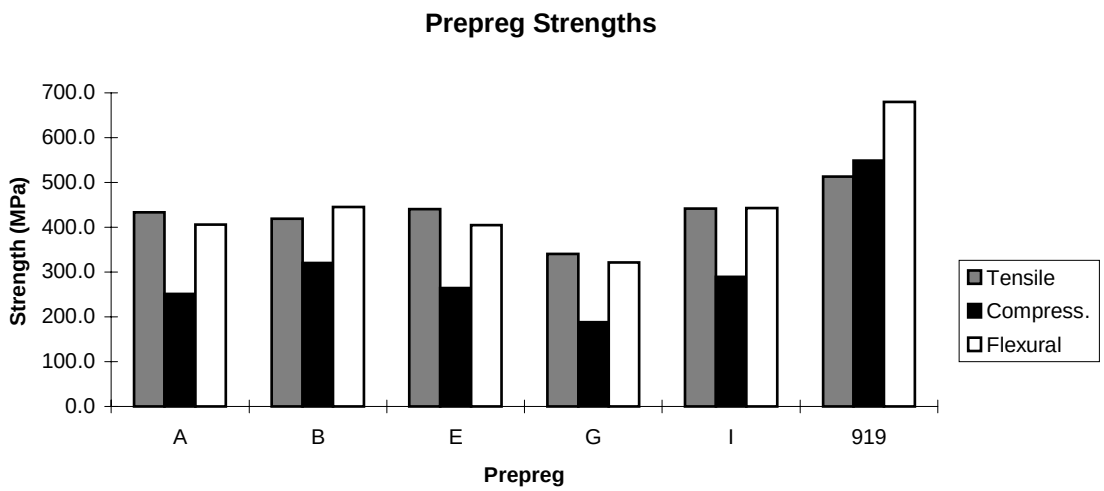

Fig. 6 Strength of the E-beam prepregs compared to the 919 prepreg.

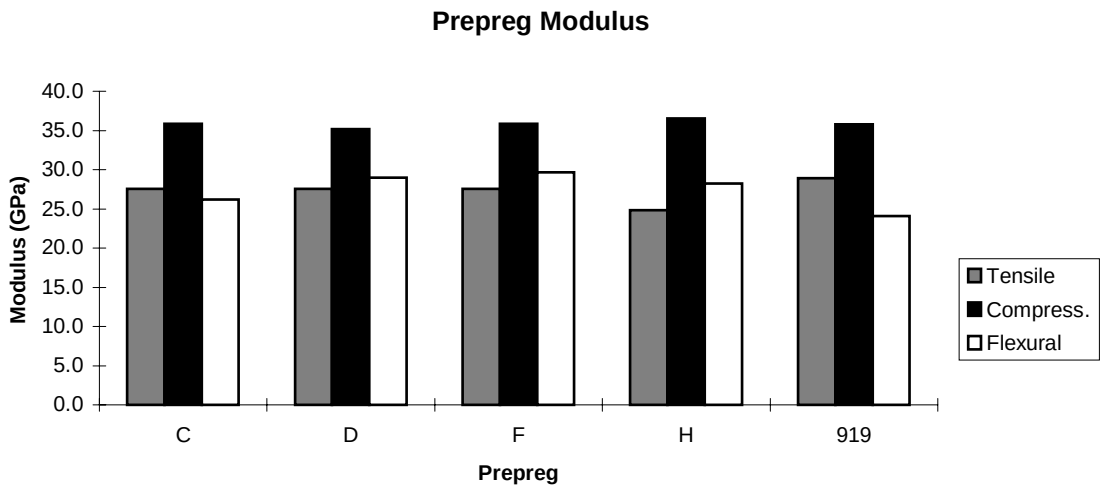

Fig. 7 Modulus of the E-beam prepregs compared to the 919 prepreg.

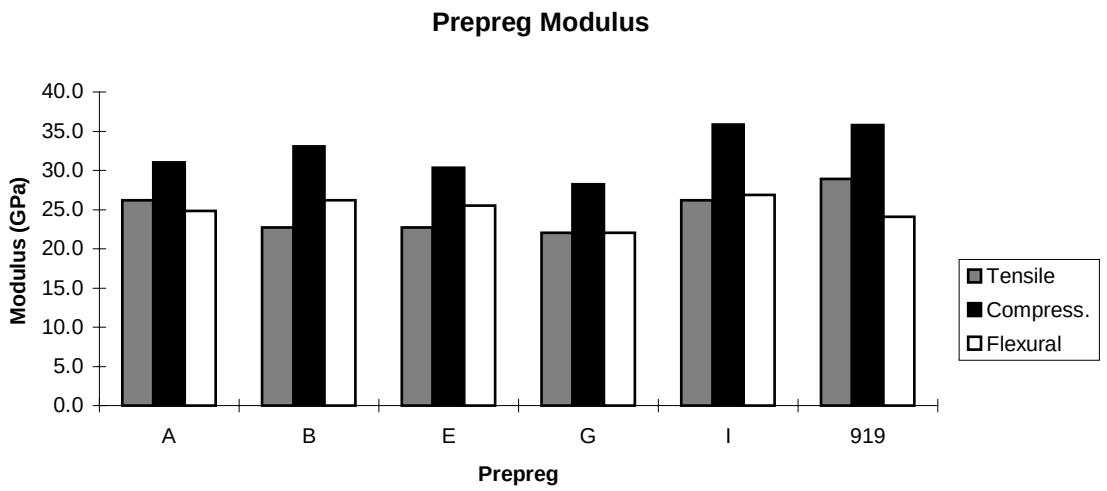

Fig. 8 Modulus of the E-beam prepregs compared to the 919 prepreg.

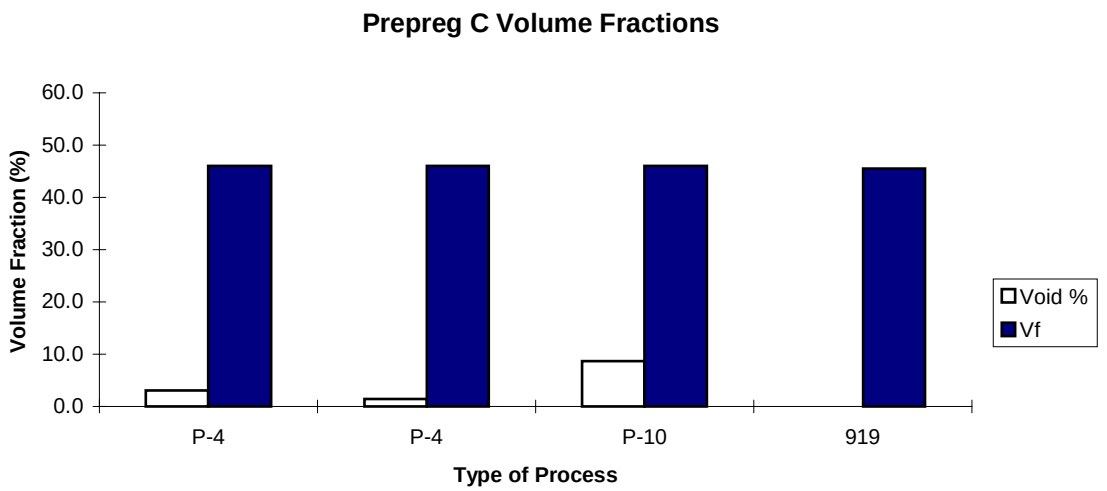

Fig. 9 Fiber and void fractions of Prepreg C compared to the 919 prepreg.

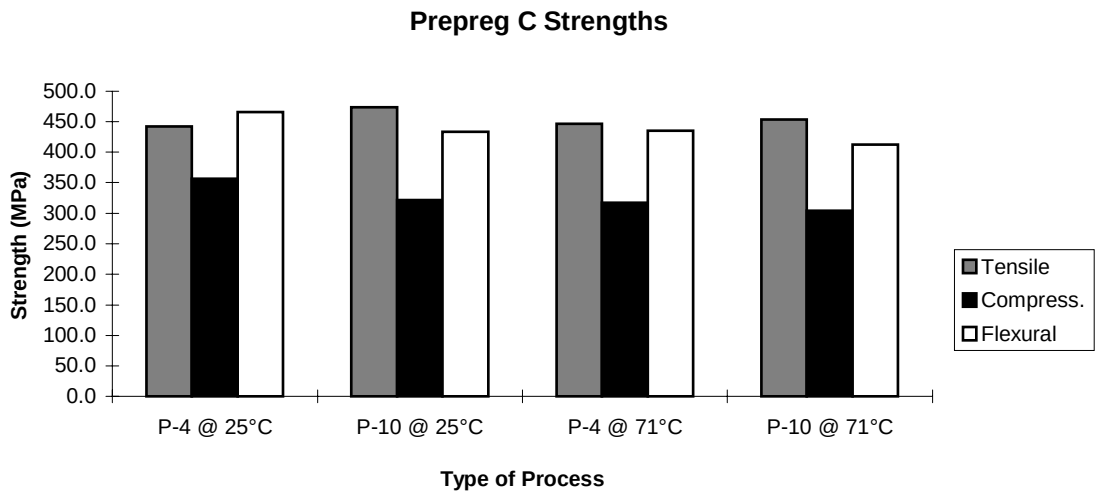

Fig. 10 Strength of Prepreg C at two test temperatures.

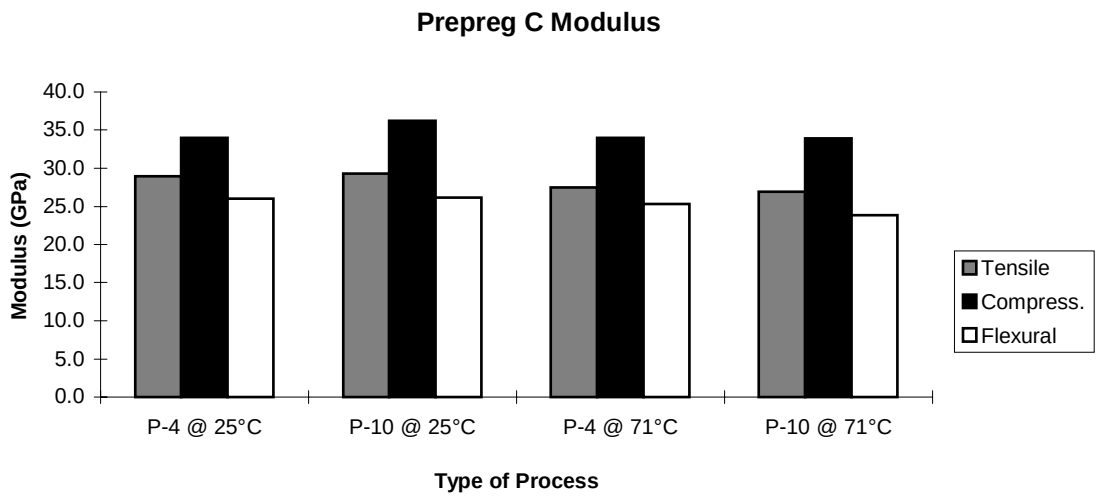

Fig. 11 Modulus of Prepreg C at two test temperatures.

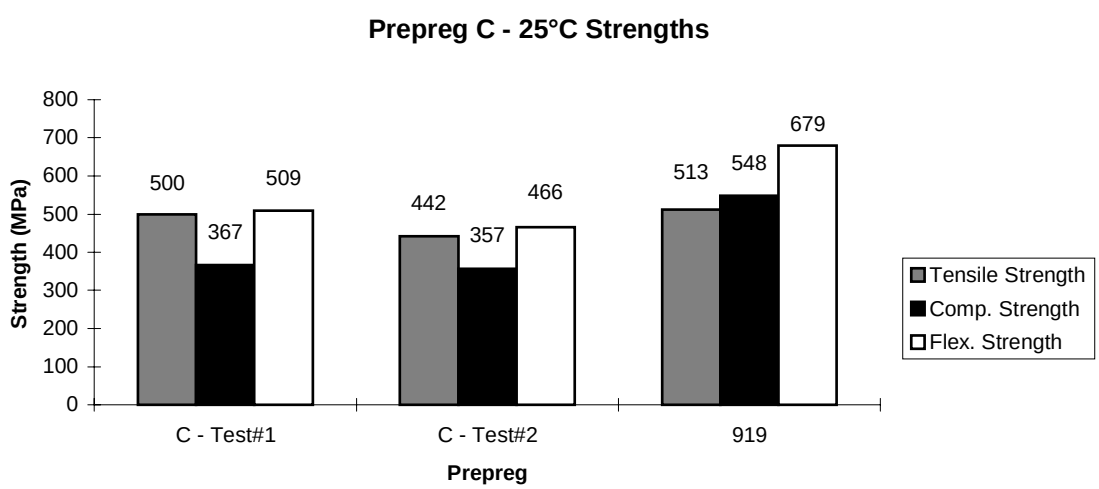

Fig. 12 Strength of Prepreg C compared to the 919 prepreg - both tested at 25°C.

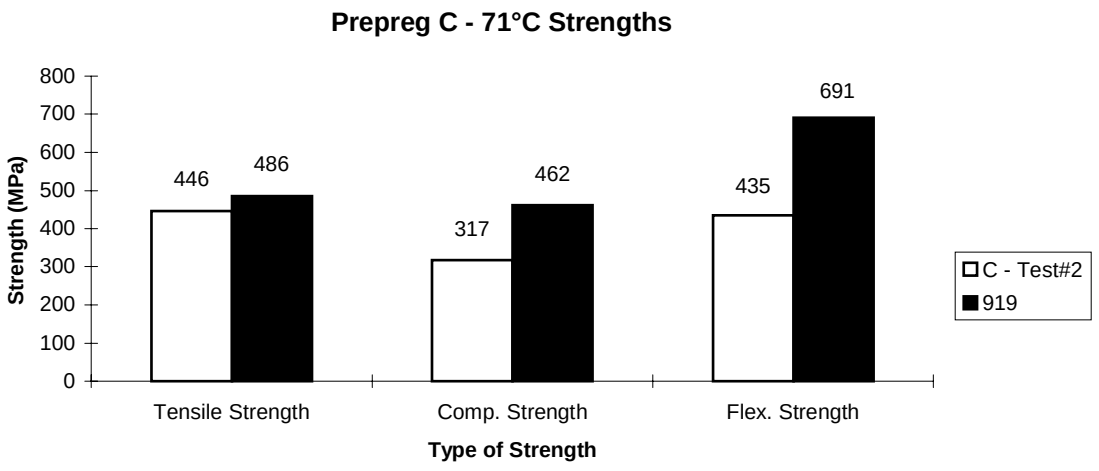

Fig. 13 Strength of Prepreg C compared to the 919 prepreg - both tested at 71°C.

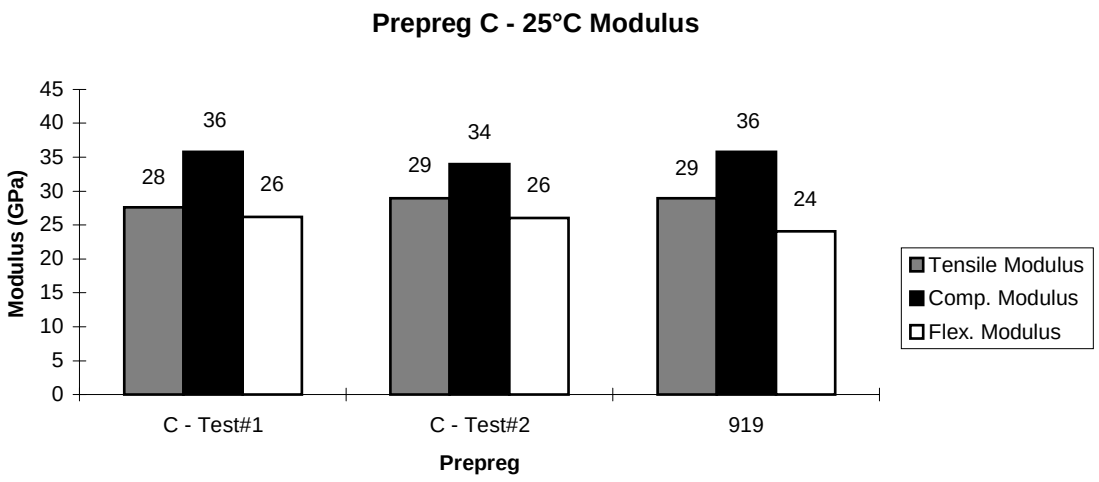

Fig. 14 Modulus of Prepreg C compared to the 919 prepreg - both tested at 25°C.

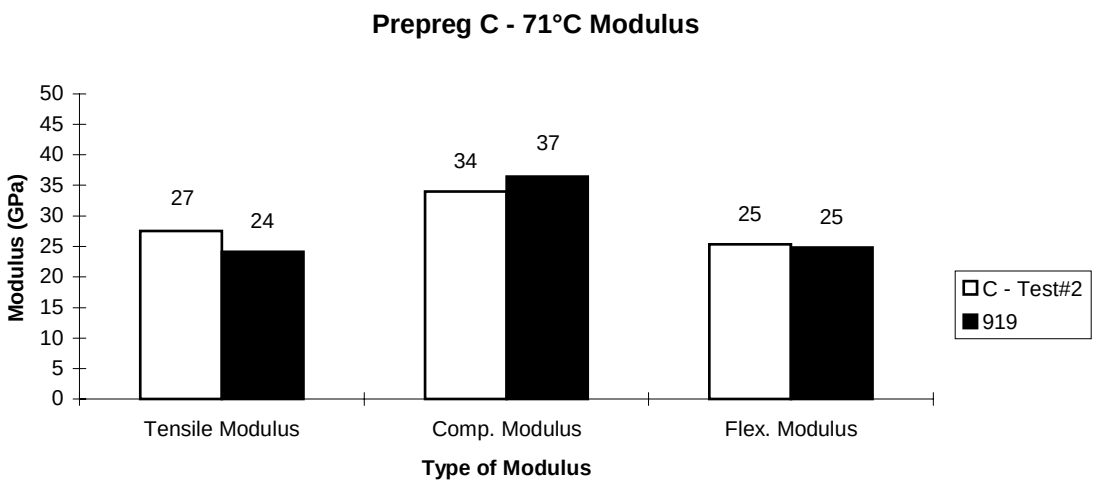

Fig. 15 Modulus of Prepreg C compared to the 919 prepreg - both tested at 71°C.

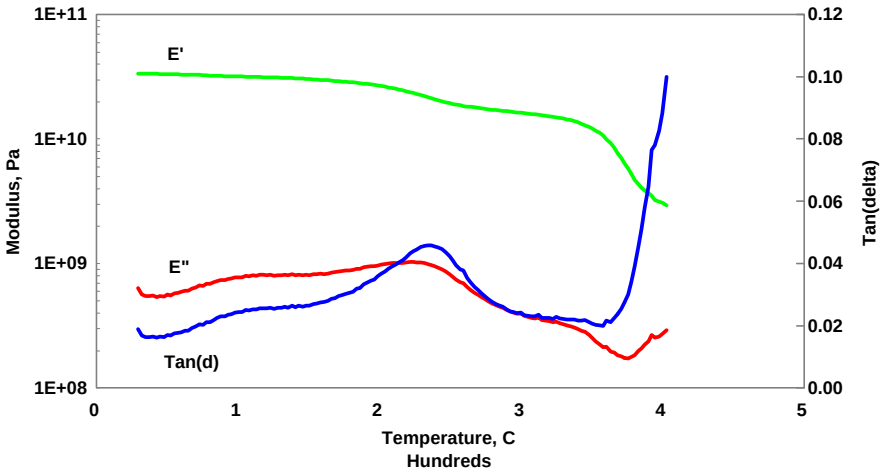

Fig. 16 DMA curves for Prepreg C.

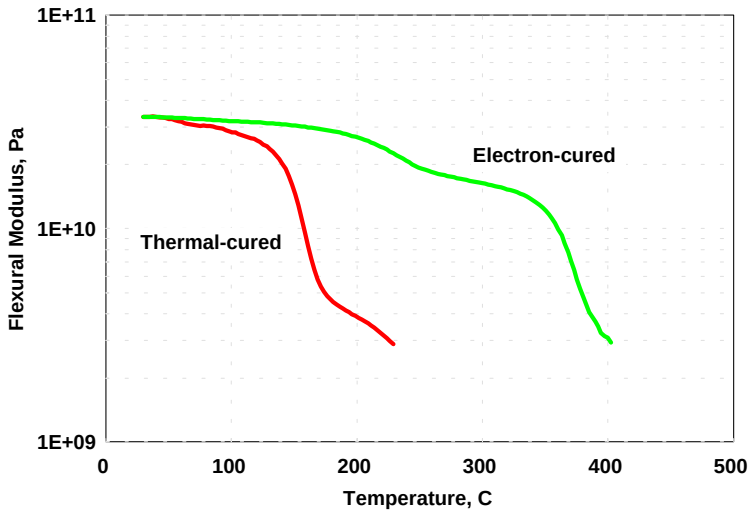

Fig. 17 Comparison of glass transition temperatures for Prepreg C and 919/6781.

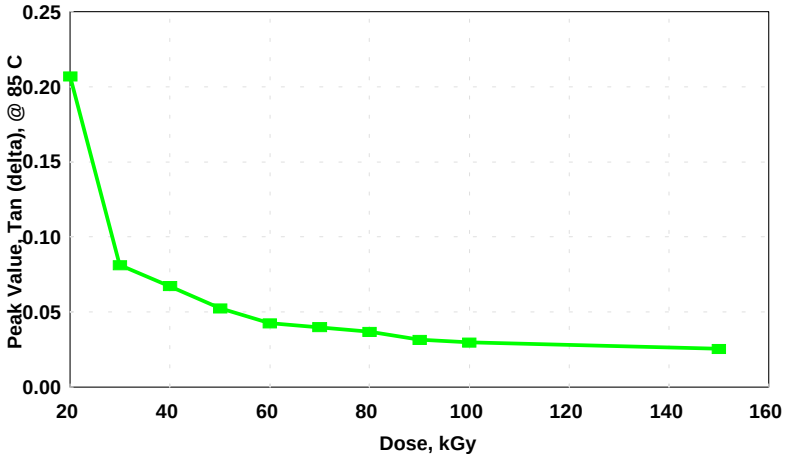

Fig. 18 Glass transition temperature of Prepreg C vs. cure dosage.

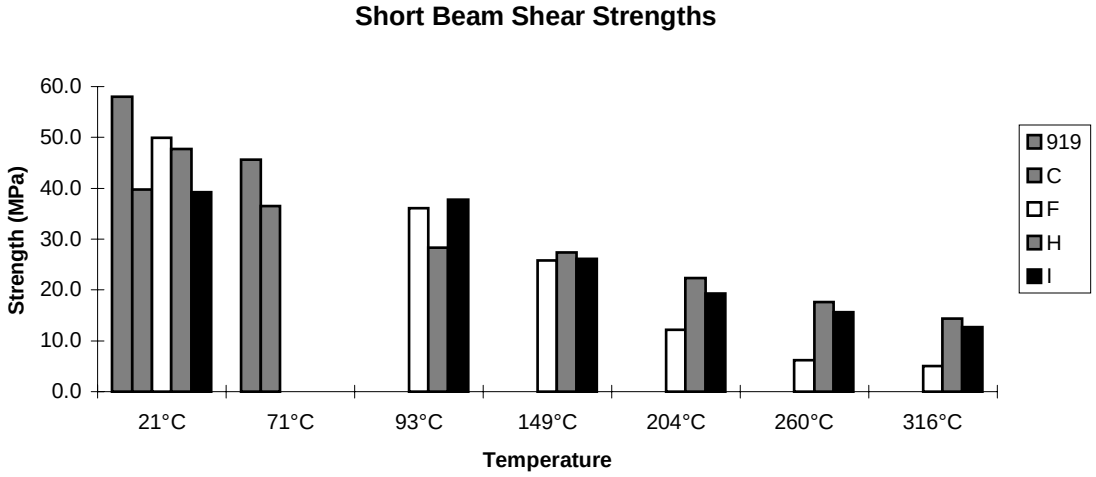

Fig. 19 Short-beam shear strength of E-beam prepregs compared to the 919/6781 prepreg.

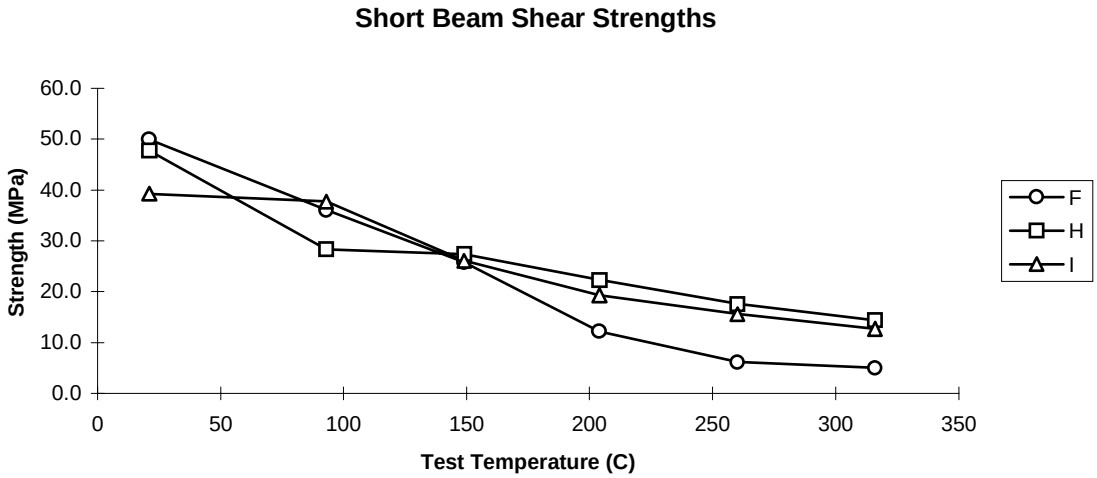

Fig. 20 Short-beam shear strengths of Prepregs F, H, and I at various test temperatures.

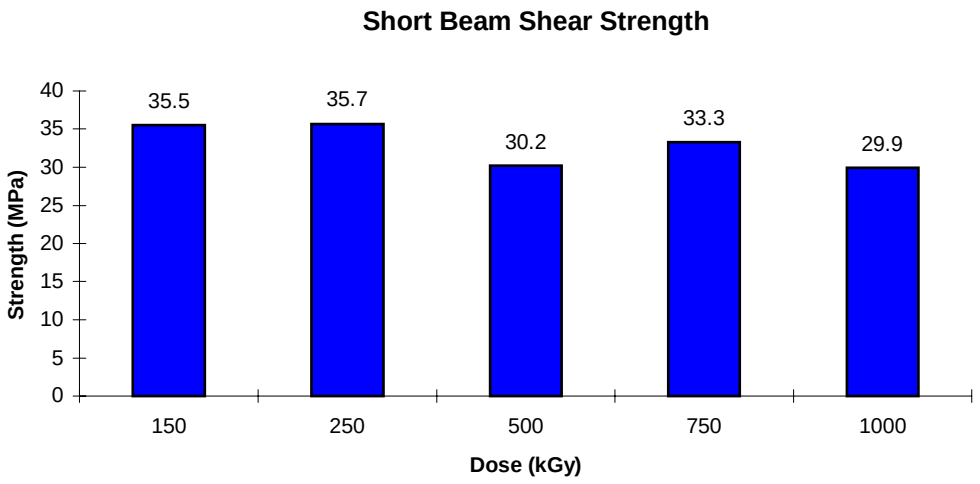

Fig. 21 Short-beam shear strength of Prepreg C laminates processed at various doses.