

RESULTS OF ELECTRON BEAM PREPREG DEBULKING STUDY

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ABSTRACT

Electron beam curing of composites is a non-autoclave process, and consolidation pressure during cure has therefore been limited to that which may be provided by vacuum bagging the part, or by the application of direct mechanical force. Tooling to apply mechanical force tends to be limited to simple geometric shapes and may also block (shadow) the electron beam's path to the composite. Vacuum bagging is readily adapted to most complex three-dimensional parts but the applied consolidation is limited to roughly atmospheric pressure and therefore cannot be relied on to collapse the expansion of volatiles during cure of the electron beam cured laminate. Preventive measures are therefore needed to reduce the factors that cause voids. A critical opportunity to reduce voids in the cured laminate is to minimize air entrapment between plies during lay-up. This paper reports the results of process trials conducted with IM7-GP-12K/3K and IM7-GP-12K/8HM unidirectional prepreg tapes to evaluate vacuum debulking and other parameters on the reduction of voids in electron beam cured laminates.

KEY WORDS: Electron Beam Curing, Hand Lay-Up, Laminates

1. INTRODUCTION

Electron beam curing of composites is a non-autoclave process (1,2,3) and consolidation pressure during cure has therefore been limited to that which may be provided by vacuum bagging the part, or by the application of direct mechanical force (weights, spring-loaded tooling, shrink tape, etc.) Tooling to apply mechanical force tends to be limited to simple geometric shapes and may also block (shadow) the electron beam's path to the composite during exposure. Shrink tape is limited to shapes that have simple, round (or at least convex) surfaces such as cylinders.

Vacuum bagging has the advantage that it is readily adapted to most complex three-dimensional parts. However, the applied consolidation pressure during cure is limited to roughly atmospheric pressure. In an autoclave, the high external pressure (typically 0.34-0.68 MPa) limits the growth of voids in the laminate caused by the expansion of trapped gases (air, moisture and other volatiles). The lower consolidation pressure of the vacuum bag process (0.07-0.10 MPa) cannot be relied on to collapse the expansion of volatiles, leading to higher void contents in the electron

beam cured laminate. Preventive measures are therefore needed to reduce the factors that cause voids.

One opportunity to reduce voids in the cured laminate is to minimize air entrapment between plies during lay-up. This can be partly accomplished by mechanically squeezing out the air between plies as each layer is added to the laminate (stack). Vacuum debulking, in which the laminate is bagged and allowed to dwell under vacuum for a specified period of time, may further eliminate/minimize entrapped air.

Trials were conducted as part of this study to evaluate vacuum debulking and other process parameters on the void content of electron beam cured laminates. The trials were conducted in three phases. In the first phase, the test lay-up consisted of 24-ply thick unidirectional ($[0]_{24}$) laminates prepared from IM7-GP-12K/3K unidirectional prepreg tape. The intent of this phase was to characterize the sensitivity of laminate void content to a wide range of processing parameters. The second phase of process trials attempted to bracket the minimum times and temperatures for an effective elevated temperature debulking process. The test matrix was expanded to include both IM7-GP-12K/3K and IM7-GP-12K/8HM unidirectional prepreg tapes, and the constructions consisted of 8-ply thick unidirectional ($[0]_8$) and bi-directional ($[0/90]_{2S}$) laminates. In the third and last phase, 16-ply thick unidirectional ($[0]_{16}$) IM7-GP-12K/3K laminates were vacuum debulked at still higher elevated temperatures to assess the impact of lowered resin viscosity during debulking on laminate void content. Perforated and non-perforated release films were incorporated in these experiments to control resin flow from the laminate.

All laminates were electron beam cured after lay-up, and their fiber and void contents were measured. The results and general conclusions regarding the sensitivity of laminate void content to a range of lay-up parameters are reported in this paper.

2. MATERIALS

Both the IM7-GP-12K/3K and IM7-GP-12K/8HM unidirectional prepreg tapes used in this study were manufactured by YLA, Incorporated. Prepregging specifications for both tapes had nominal fiber areal weights of 145 g/m^2 and resin contents of 34 ± 3 weight percent. The 3K and 8HM resin systems used in this work were electron beam curable cationic epoxy resins developed by the Oak Ridge National Laboratory (4,5).

3. EXPERIMENTAL AND RESULTS

The basic lay-up process was identical for all test laminates prepared during all three phases of this study. Overhead facility lights were turned off to minimize the exposure of the prepreg to direct light during the lay-up process. (That is because electron beam curable cationic epoxy resins are photosensitive and exposure to light may inadvertently advance the cure of the prepreg resin.)

All laminate plies were applied by hand one ply at a time. A rubber-sanding block was used to squeegee out air by pressing the block with hand pressure over the ply release paper and in the

direction of the unidirectional fiber in one continuous motion. The direction of the squeegee motion initiated from the middle of the lay-up and ended out toward its perimeter, with the intent to always work the entrapped air toward an open pathway (the edge) out of the laminate. After working the prepreg with the rubber-sanding block, the release paper was removed and the second ply was applied.

Vacuum debulking was accomplished by placing the samples within a pre-fabricated vacuum bag tool. Elevated temperature debulking was accomplished by placing the vacuum bag tool in a preheated convection oven followed by bringing the samples to the desired temperature for the specified amount of time.

After lay-up, the laminates were sandwiched tightly between 1.7-mm thick aluminum plates that had been lined with a layer of release film. The samples were then shipped to the Acsion Facility in Pinawa, Manitoba, Canada where they were vacuum bagged and electron beam cured using Acsion's 10-MeV, 1-kW Industrial Processing Linear Accelerator (IMPELA). Samples prepared from the IM7-GP-12K/3K prepreg were cured with a total electron beam dose of 150 kGy, while samples prepared from the IM7-GP-12K/8HM prepreg were cured with a total dose of 200 kGy. Unless otherwise specified by the process matrix, the dose per pass (dose increment) for both materials was 25 kGy/pass.

After cure, the bagging materials and aluminum plate tools were removed. The laminates were evaluated for composition (fiber and void content) using an acid digestion method based on ASTM D3171 "Standard Test Methods for Constituent Content of Composite Materials". Composition measurements in Phases 1 and 2 were performed by YLA Incorporated with three samples per laminate. Composition measurements in Phase 3 were performed by Cincinnati Testing Laboratory (CTL) with five samples per laminate.

Additional details and descriptions of processing procedures used to accomplish this debulking study are provided in sections 3.1-3.3.

3.1 Phase 1 Process Trials The first phase of this debulking study was designed around the parameters of a lay-up process that was originated by YLA Incorporated, but with the added intent to characterize the sensitivity of laminate void content to a range of variations to this procedure. Test lay-ups prepared in the first phase of this debulking study all consisted of 24-ply thick unidirectional ($[0]_{24}$) laminates prepared from IM7-GP-12K/3K unidirectional prepreg tape.

In the baseline lay-up process, the laminate stack is vacuum debulked after the application of every single ply at ambient temperature for 15 minutes. The debulking materials consists of one layer of Teflon coated glass fabric (Airtech Release Ease 234 TFP) and/or perforated A4000 release plastic film followed by a layer of breather cloth that are laid over the laminate stack. The lay-up is then vacuum debulked in a Zip-Vac tool at room temperature for the time specified by the test matrix.

After the last ply has been added, the entire stack is given a 1-hour vacuum debulking cycle at an elevated temperature sufficient to promote resin flow from the stack, which for the 3K resin, is $\sim 70^{\circ}\text{C}$. The lay-up scheme for the final heat debulk is detailed in Figure 1. One layer of Teflon

coated glass fabric (Airtech Release Ease 234 TFP) followed by layers of Airtech Release Ply C bleeder cloth are placed on both sides of the stack to absorb the resin. The number of plies of bleeder cloth is dependant on the stack thickness, the initial resin content of the prepreg and the desired final resin content in the laminate.

For these trials, the baseline bleed-out process was conducted using nominally 2-3 layers of Release Ply C bleeder cloth for every 4-6 plies of prepreg. The quantity of bleeder cloth was divided so that roughly half of the layers would be placed on the top of the stack and half on the bottom. Unless otherwise specified, bleed-out was therefore symmetrical i.e. from both faces of the laminate stack. (For example, the elevated temperature debulking of a 24-ply stack was conducted with 4 plies of bleeder cloth on both sides of the laminate, or a total of 8 plies of bleeder cloth.)

The lay-up of the laminate stack with Teflon coated glass fabric and Release Ply C bleeder cloth was laid onto a layer of A4000 release film in a vacuum bag tool. A layer of perforated A4000 release film followed by a 0.64-cm thick aluminum caul plate overwrapped with a layer of non-perforated release plastic film was laid on top, followed by a layer of breather cloth. The stack was then vacuum debulked in an oven for the time and temperature specified by the test matrix. For process trials which specified a non-bleed elevated temperature debulking cycle, a layer of A4000 release film was substituted for the Teflon coated glass fabric and the bleeder cloth layers.

For one of the process trials in Phase 1, the elevated temperature debulking was accomplished under pressure using a heated platen press instead of in the oven. However, this laminate was vacuum bagged using the same procedure and materials described above for a non-bleed process.

Table 1 is the summary of the test samples prepared and their relevant processing data for this phase of the debulking study. The process parameters investigated included:

- Debulking temperature (ambient versus elevated temperature)
- The number of plies (stack height) prior to ambient temperature debulking
- Stack height prior to elevated temperature debulking
- Time of ambient temperature debulking cycle
- The effect of additional external pressure applied to the laminate during elevated temperature debulking cycle
- The effect of resin bleed-out during debulking versus a non-bleed process
- Additional debulking *after* shipment to the accelerator facility (to determine if the laminate appreciably “relaxes” or separates during transit time)
- The consequence of delaying cure for a period of time (several weeks) after debulking versus lay up of the laminate at the accelerator facility followed by cure immediately thereafter
- Area (size) of laminate
- Operator differences (due to the individual conducting lay-up)
- The effect of electron beam dose per pass (dose increment) on void growth during cure
- The laminate location sampled for composition (edge versus center of laminate)

Composition results indicated that the factor that yielded the lowest void contents in these trials was the elevated temperature debulking step. The baseline process (one elevated debulking cycle upon completion of lay-up) yielded laminates with void contents on the order of 2.6 to 2.9 volume percent. Lower void contents (1.5 to 1.8 volume percent) were achieved by performing elevated temperature vacuum debulking at regular intervals (every 4 plies) during lay-up of the laminate stack (numbers 9 and 10). Not surprisingly due to the higher number of bleed-out cycles, these laminates also had higher fiber contents (~64 volume percent versus 58-60 volume percent).

An *additional* (non-bleed) elevated temperature vacuum debulking cycle conducted at the accelerator facility and immediately prior to cure (number 4) also yielded lower voids (1.8 volume percent).

The data indicate that vacuum debulking at ambient temperature has some benefit in lowering void content for thick laminates, but the contribution is not as effective as it is for the elevated temperature debulking step. Lower void levels were achieved for laminates vacuum debulked after the application of every ply and for 15 minutes intervals at ambient temperature (baseline process) versus laminates debulked only after every 2, 4, or 24 plies, or for only 5 minutes (numbers 2, 3, 12, and 8). However, the laminates that were debulked every four plies at elevated temperature still had lower void levels than the laminates receiving only one elevated temperature vacuum debulking cycle at the conclusion of their 24-ply lay-up.

A non-bleed heat debulking process (number 5) did not measurably increase the void level above that of laminates debulked with forced resin bleed-out into bleeder cloth. Sample number 5's void level was also 3 volume percent, although its fiber content was significantly lower (57.6 volume percent) than the fiber contents of laminates undergoing resin bleed-out during debulking.

The lowest void level (0.9 volume percent) was obtained by conducting the elevated temperature vacuum debulking step under high pressure (0.68 MPa applied using a heated platen press) and using a non-bleed process (number 11). After debulking, pressure was maintained on the laminate as it cooled back to ambient temperature. The intent of this process was to "pre-consolidate" the laminate with autoclave-magnitude pressures prior to cure. This sample also had a fiber content of only 60.8 volume percent, indicating that forcing high resin bleed-out is not necessarily the only route for producing low void laminates. Although this process yielded the lowest void content, methods for making it practical (cost-effective) in the manufacture of large-scale composite hardware are not readily available.

For comparable samples, no correlation was observed between electron beam dose per pass (dose increment) and void level for these data. The void level for the laminate cured at 5 kGy/pass (number 6) was 2.9 volume percent. A second laminate cured at 5 kGy/pass (number 11) had a significantly lower void level but, as described earlier, this is attributed to the preconsolidation under high pressure in the heated platen press during the elevated temperature. A significantly higher void level (3.7 volume percent) was measured for a comparable laminate cured at 50 kGy/pass (number 7). Although this void level was one of the highest measured in this study,

other laminates cured with the 25 kGy dose/pass and similar lay-up/debulking schemes also had void levels above 3 volume percent.

3.2 Phase 2 Process Trials The second phase of process trials attempted to bracket the minimum time and temperature for the elevated temperature debulking process to effectively reduce voids. The test matrix was expanded to include both IM7-GP-12K/3K and IM7-GP-12K/8HM unidirectional prepregs, and the constructions consisted of 8-ply thick unidirectional ($[0]_8$) and bi-directional ($[0/90]_{2s}$) laminates. The initial hand lay-up of the laminates was the same as in Phase 1. The ambient temperature debulking step was eliminated. Instead, elevated temperature vacuum debulking was conducted after the first four-ply increment to the laminate, and again after application of the last four plies.

For the initial four-ply lay-up, a layer of porous Teflon coated glass fabric (Airtech Release Ease 234 TFP) was placed over both sides of the stack, followed by one layer of Airtech Release Ply C. The stack was laid onto a layer of A4000 release film in a vacuum bag tool. A 0.64-um thick aluminum caul plate overwrapped with a layer of non-perforated release film were laid on top, followed by a layer of breather cloth. The stack was vacuum debulked in an oven for the time and temperature specified by the test matrix. For the first stack only, the initial debulk bleed-out was from both sides of the laminate.

After debulking, the next four-ply increment was added to the stack one ply at a time. The second elevated temperature vacuum debulk was conducted similarly to the first cycle with the exception that the bleeder materials were applied to the new composite layers, and resin bleed-out was intended to be from the top face of newly added plies only. The layer of A4000 release plastic placed beneath the stack and adjacent to the opposite face of the previously debulked plies prevented/minimized any additional bleed-out from this portion of the laminate.

Tables 2 and 3 summarize respectively the relevant fabrication data and composition results for the IM7-GP-12K/3K and IM7-GP-12K/8HM laminates prepared and evaluated as part of the second phase of this debulking study. Measurements made of the total resin weight loss (bleed-out) for the laminates as a result of elevated temperature debulking are included as well.

The data for the IM7-GP-12K/3K laminates are comparable to results obtained in the Phase 1 debulking studies for the same prepreg system. Lower voids were obtained more consistently using the higher debulking temperature (71°C). At 60°C, lower voids were obtained for long(er) debulking times of 16 hours. Half of the laminates debulked at 60°C for 2 hours or less had higher void levels (greater than 2.5 volume percent). Void contents measured for unidirectional and cross-ply laminates debulked under the same conditions were comparable.

For the IM7-GP-12K/8HM resin laminates, void levels comparable to those obtained for the 3K prepreg (less than 2 volume percent) were obtained with debulking cycles of 16 hours at 60°C and 1 hour at 71°C. For unidirectional and cross-ply laminates debulked under these same conditions, the void levels were comparable.

The data show that debulking the IM7-GP-12K/8HM laminates longer (2 hours) at 71°C and at higher temperatures (82°C) did not consistently translate to lower voids. Although the cross-ply

laminates still had void levels well below 2 volume percent, the unidirectional laminates actually had voids greater than 2.5 volume percent. The reason for this is unknown and further investigation is required in this area.

The resin bleed-out data summarized in Tables 2 and 3 showed that total resin bleed-out resulting from this elevated temperature debulking process was on the order of 4.5-7.0 weight percent for the 3K resin laminates and 5.3-8.6 weight percent for the 8HM resin laminates. No correlation was deduced between the amount of resin bleed-out and the resulting void level in the cured laminate. The unidirectional lay-ups for both resin systems tended to show higher resin bleed-outs than their bi-directional counterparts. This is probably because interference between the rigid 0° and 90° ply fibers in the bi-directional laminates minimized the amount of compression (“squeezing”) possible between layers.

3.3 Phase 3 Process Trials The data from Phase 2 suggested that a greatly reduced resin viscosity will facilitate the escape of air and volatiles from the laminate during vacuum debulking. In the third and last phase, 16-ply thick unidirectional ([0]₁₆) IM7-GP-12K/3K laminates were vacuum debulked at higher (100°C) elevated temperatures to assess the impact on void content. Process experience with the 3K resin system indicates that the resin can tolerate several hours at 100°C with no advancement of cure or other adverse effects on properties (6).

The laminates were prepared and debulked using the same materials and procedures described in sections 3.2 for conducting elevated temperature debulking at 4-ply stack intervals. To prevent any additional diffusion of resin from the laminate and beyond the bleeder plies, layers of perforated and non-perforated release films were incorporated proximate to the bleeder plies and the breather cloth. These debulking material locations are notated in Table 4. No aluminum caul plate was used for debulking the Phase 3 laminates. Debulking time at 100°C was reduced to 30 minutes.

Table 4 summarizes the relevant fabrication data and composition results for the IM7-GP-12K/3K laminates that were prepared and evaluated as part of this phase of the debulking study. Measurements made of the total resin weight loss (bleed-out) for the laminates as a result of elevated temperature debulking are included as well. For comparison, a laminate was also prepared using a process similar to the Number 10 debulking method described in section 3.1 with the exception that no caul plate was used during the 71°C elevated temperature debulking steps.

The results summarized in Table 4 show that the void levels for all laminates debulked at 100°C is less than 1 volume percent. Results for the Number 1 process are slightly higher (1.1 volume percent). It is interesting that the Number 1 case in Phase 3 had slightly lower voids compared to the Number 10 case in Phase 1 which had voids of 1.8 percent. One difference is that the Phase 3 laminates were 16 plies thick instead of the 24 plies in Phase 1. Another reason for this difference may lie with the fact that no caul plate was used in Phase 3, perhaps increasing the surface area for air to escape from the laminate stack.

Visual observations are that the caul plate should not be necessary with the intermittent debulking process because air is taken out gradually from the laminate every 4 plies instead of in

one single step at the end of the lay-up. It should therefore be easier to maintain surface control during debulking without the use of a caul plate. This is advantageous for fabricating curvilinear and other complex shaped parts.

No differences were deduced between the various debulking materials and their impact on resin diffusion from the laminate. Total resin weight loss as a result of elevated temperature debulking is on the order of 10 weight percent for all of the laminates, including the baseline sample that was debulked at 71°C. Differences in the void contents of the laminates are minor and may not be statistically significant.

4. CONCLUSIONS

Process trials were conducted to evaluate vacuum debulking and other process parameters on the void content of electron beam cured laminates. This study was conducted in three phases. The test lay-ups in the first phase consisted of 24-ply thick unidirectional ($[0]_{24}$) laminates prepared from IM7-GP-12K/3K unidirectional prepreg tape. Laminates in the second phase were prepared from both IM7-GP-12K/3K and IM7-GP-12K/8HM unidirectional prepreg tapes, and included 8-ply thick unidirectional ($[0]_8$) and bi-directional ($[0/90]_{2S}$) constructions. The third phase was conducted with 16-ply thick unidirectional ($[0]_{16}$) laminates prepared from IM7-GP-12K/3K unidirectional prepreg tape.

Composition results indicated that the factor that consistently yielded the lowest void contents for the 3K resin laminates was elevated temperature debulking at regular intervals during lay-up of the laminate stack. Vacuum debulking at ambient temperature has some benefit in lowering void content for thick laminates, but the contribution is not as effective as it is for the elevated temperature debulking step. In these trials, IM7-GP-12K/3K void levels were reduced to the 1-2 volume percent level with elevated temperature debulking at 71°C applied after the lay-up of every 4 plies. This contrasts with void levels measured as high as 2-4 volume percent for laminates processed without intermittent heat debulking during lay-up.

Results indicate that there is a combination of minimum time-and-temperature required to minimize voids with elevated temperature debulking. For the 3K resin laminates, low voids could be achieved with lower debulking temperatures provided that the debulking time was also increased.

Elevated temperature debulking also yielded comparable void levels (less than 2 volume percent) for laminates prepared from IM7-GP-12K/8HM prepreg. However, increasing debulking times and temperatures did not consistently translate to lower void levels for 8HM resin laminates in this study, and in some cases, actually produced greater voids. The reason for this is unknown and additional study is required to understand and optimize the laminate debulking process for 8HM resin prepreps.

Excellent results were obtained with IM7-GP-12K/3K laminates that were vacuum debulked at 100°C for 30 minutes after the lay-up of every 4 plies. These results are consistent with the observation that reducing resin viscosity enhances the removal of volatiles and air during vacuum debulking.

5. REFERENCES

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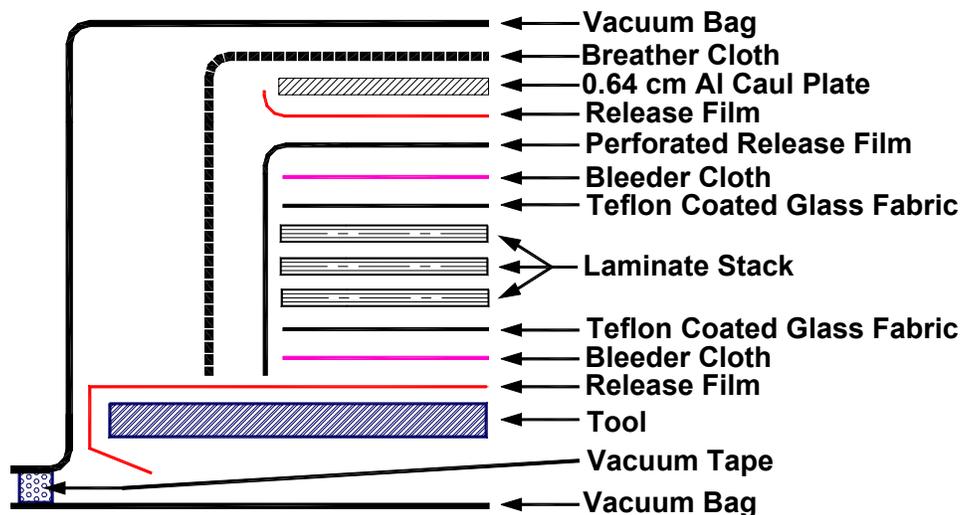


Figure 1. Bagging for Phase 1 elevated temperature debulks.

Table 1. Parametric Process Study - IM7-GP-12K/3K 24-ply Unidirectional Laminates.

Sample (process) ID	Operator	Size (cm x cm)	Vacuum Debulking Parameters						Dose* Increment (kGy/pass)	Panel Location	Composition		
			Temp (°C)	Time (min)	Stack Height (no. of plys)	Temp (°C)	Time (min)	Stack Height (no. of plys)			Density (g/cm ³)	Voids (Vol. %)	Fiber (Vol. %)
1	1	31 x 31	amb	15	4	---	---	---	25	center	1.510	2.8	57.1
	2	15 x 15	amb	15	4	---	---	---	25	edge	1.487	4.0	55.0
2	1	15 x 15	amb	15	2	71	60	24	25	edge	1.523	3.0	60.0
	2		amb	15	2	71	60	24	25	edge	1.518	3.4	59.8
3	1	15 x 15	amb	15	4	71	60	24	25	edge	1.518	3.4	60.0
	2		amb	15	4	71	60	24	25	edge	1.517	2.6	58.0
4 ¹	1	15 x 15	amb	15	4	71	60	24	25	edge	1.543	1.8	61.0
5 ²	1	15 x 15	amb	15	4	71	60	24	25	edge	1.510	3.0	57.6
6	1	15 x 15	amb	15	4	71	60	24	5	edge	1.533	2.9	61.4
7	1	15 x 15	amb	15	4	71	60	24	50	edge	1.516	3.7	60.1
Baseline	1	15 x 15	amb	15	1	71	60	24	25	edge	1.528	2.9	60.5
	2		amb	15	1	71	60	24	25	edge	1.530	2.6	60.5
8	1	15 x 15	amb	5	1	71	60	24	25	edge	1.539	3.5	63.8
9	1	15 x 15	amb	15	1	71	60	4	25	edge	1.566	1.5	64.4
10	1	15 x 15	---	---	---	71	60	4	25	edge	1.562	1.8	64.4
11 ³	1	15 x 15	amb	15	4	71	60	24	5	edge	1.554	0.9	60.8
12	1	15 x 15	amb	15	24	71	60	24	25	edge	1.498	3.8	57.3
13 ⁴	1	15 x 15	38-48	15	4	71	60	24	25	edge	1.517	3.2	59.3

* 150 kGy total dose

1. Second vacuum debulking for 1 hour at 71°C performed at Acsion prior to cure. Non-bleed process (non-permeable release plastic.) Part maintained under vacuum until cure conducted.
2. Non-bleed process (non-permeable release plastic.)
3. Elevated temperature debulk conducted under 0.68 MPa in platen press. Non-bleed process (non-permeable release plastic.)
4. Cured immediately after layup at accelerator facility

Table 2. Phase 2 debulking study - IM7-GP-12K/3K 15-cm x 15-cm laminates (8 ply).

Sample (process) ID	Lay-up	Elevated Temperature Vacuum Debulking				Density (g/cm ³)	Voids (Vol. %)	Fiber (Vol. %)
		Temp (°C)	Time (min)	Stack Height (no. of plys)	Total Bleed-out (weight %)			
3K-60-1U ¹	[0] ₈	60	60	4	6.1	1.539	1.8	61.5
3K-60-1M	[0/90] _{2s}	60	60	4	4.4	1.533	4.4	66.2
3K-60-2U	[0] ₈	60	120	4	6.9	1.539	2.7	62.5
3K-60-2M	[0/90] _{2s}	60	120	4	6.1	1.545	1.3	61.2
3K-60-16U	[0] ₈	60	960	4	7.0	1.554	0.2	60.5
3K-60-16M	[0/90] _{2s}	60	960	4	5.0	1.554	0.7	61.5
3K-71-1U	[0] ₈	71	60	4	6.9	1.542	1.4	60.8
3K-71-1M	[0/90] _{2s}	71	60	4	5.2	1.552	0.7	61.3

Laminates cured at 25 kGy/pass for a total dose of 150 kGy

1. Final debulking cycle of entire laminate (after layer no. 8) was conducted twice. During first debulking cycle, laminate was covered with non-porous layer and was therefore a non-bleed process. Second debulking cycle was repeated using standard bleeder cloth to permit bleed-out.

Table 3. Phase 2 debulking study - IM7-GP-12K/8HM 15-cm x 15-cm laminates (8 ply).

Sample (process) ID	Lay-up	Elevated Temperature Vacuum Debulking				Density (g/cm ³)	Voids (Vol. %)	Fiber (Vol. %)
		Temp (°C)	Time (min)	Stack Height (no. of plies)	Total Bleed-out (weight %)			
8HM-60-16U	[0] ₈	60	960	4	6.1	1.555	1.5	62.2
8HM-60-16M	[0/90] _{2s}	60	960	4	5.7	1.555	1.1	61.4
8HM-71-1U	[0] ₈	71	60	4	8.6	1.557	1.3	62.3
8HM-71-1M	[0/90] _{2s}	71	60	4	6.4	1.555	1.2	61.5
8HM-71-2U	[0] ₈	71	120	4	7.1	1.547	2.8	64.0
8HM-71-2M	[0/90] _{2s}	71	120	4	5.3	1.557	1.2	61.9
8HM-82-1U	[0] ₈	82	60	4	8.2	1.543	3.1	63.8
8HM-82-1M	[0/90] _{2s}	82	60	4	6.3	1.558	1.1	62.0

Laminates cured at 25 kGy/pass for a total dose of 200 kGy.

Table 4. Phase 3 debulking study - IM7-GP-12K/3K 30-cm x 30-cm unidirectional laminates (16-ply).

Sample (process) ID	Elevated Temperature Vacuum Debulking					Density (g/cm ³)	Voids (Vol. %)	Fiber (Vol. %)
	Temp (°C)	Time (min)	Stack Height (no. of plys)	Bleed-out Materials Sequence ¹	Total Bleed-out (weight %)			
1	71	60	4	bleeder/P1 ²	10.2	1.562	1.1	63.8
2	100	30	4	bleeder/P7 ³	9.6	1.562	0.9	63.6
3	100	30	4	P1/bleeder/NP ⁴	9.7	1.561	0.8	63.1
4	100	30	4	P1/bleeder/P7	9.7	1.563	0.6	63.1

Laminates cured at 25 kGy/pass for a total dose of 150 kGy

Notes:

1. Materials laid on top of laminate stack and Teflon coated glass fabric layer.
2. Airtech A4000RP1 release film - 1.1-mm diameter holes staggered at 6.4-mm hole center intervals
3. Airtech A4000RP7 release film - 0.4-mm diameter holes staggered at 76.2-mm hole center intervals
4. Airtech A4000NP (non-perforated release film)