

THERMOPLASTIC COMPOSITE PROCESSING USING SUPERPLASTIC FORMED MAGNESIUM BLADDERS

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ABSTRACT

This paper discusses a method of manufacturing thermoplastic composite parts using superplastic formed AZ31 magnesium bladders. This method superplastically forms magnesium against the composite part at temperatures lower than traditional superplastic forming temperatures and above thermoplastic composite melting temperatures to consolidate the thermoplastic composite. Historically, superplastic forming has been completed with pressurized argon gas. An investigation into the use of nitrogen gas was completed and the resulting parts were evaluated. The part configurations are evaluated for attenuation, surface roughness, and ply locations on a gentle contoured single sided tool surface with single use bladders. It was found that the use of nitrogen as compared to argon as a bladder pressurization gas had a negative impact on the part quality.

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1. INTRODUCTION

The RAPid High Performance Molding (RAPM) for Small Parts program has investigated and demonstrated the ability to fabricate PEEK thermoplastic preforms by applying compaction pressure with a superplastic formed magnesium bladder [4] [5] [6]. Previously, these parts were used in a secondary stamp forming processing step. The part trials completed for this paper consolidated PEKK thermoplastic composite material from loose plies to a net shape composite part using an AZ31 Magnesium bladder on a low curvature single sided tool. With this process change, nitrogen was used as a pressurization gas to improve compatibility with automation equipment. The part surface quality, ply locations, and consolidation quality were evaluated for the parts.

1.1 Audience

This paper was written for those experienced in thermoplastic composite part fabrication. A new processing method for parts fabricated in a press is discussed. The paper discusses process modification and parameters of importance and evaluates the resulting parts. For those familiar with thermoplastic composite processing this paper will provide an overview of the superplastic formed AZ31 magnesium bladder processing of parts.

1.2 Background on Magnesium Bladder Processing

The RAPM program discussed an alternative to match metal tooling for thermoplastic part fabrication. This single sided tooling approach has lower tooling requirements. Historically, bladder materials have been unable to withstand the processing temperatures with the elongation

needed for processing. As a result alternative bladder materials were evaluated. Superplastic forming of AZ31 magnesium has been used in automotive industries, the material is relatively low cost, and the supply base exists. For this reason it was chosen as a bladder material candidate earlier in the program. At the beginning of the program the bladders were processed with argon with successful preform fabrication. To be more compatible with current automation equipment design for use with nitrogen, the processing gas was changed. This also had the additional benefit of nitrogen gas being lower cost.

1.3 Tooling and Part Geometry

The parts were fabricated from a PEKK thermoplastic polymer matrix with unidirectional carbon fiber reinforcement. The parts had a balanced and symmetric layup. The tooling is shown in Figure 1. The tooling was manufactured from Invar 42 to best match the thermal expansion of the composite material. The tool sat with the tool CMD-TP-RAPM-003-501 tool inside of a pixilated active heating and cooling clamping fixture capable of applying 150 ton clamping force and heating to over 400°C fabricated by Surface Generation Ltd [4] (CF-OaO-RAPM-001). The tool has a gentle contour to manufacture a part 40.6 cm x 40.6 cm (16 inches x 16 inches) and 1.3cm (0.5 inches) in height.

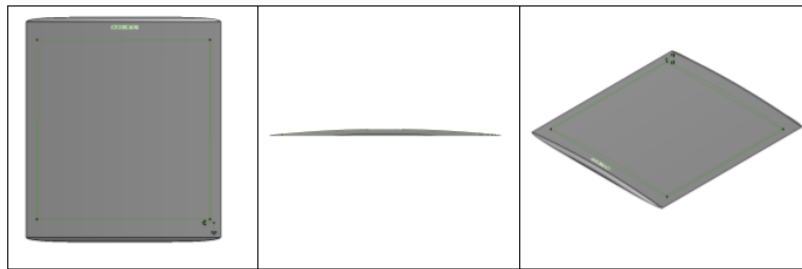


Figure 1 - CMD-TP-RAPM-017-501 top view (left), front view (middle), isometric view (right)

Three part geometries were evaluated with varying thickness and ply drops. The parts are shown in Figure 2. The three different Geometries (flat, ply drop, and picture frame) have 32 plies in their thick regions and 16 plies in their thin regions. Between the two regions are a 20:1 ply drop ratio.

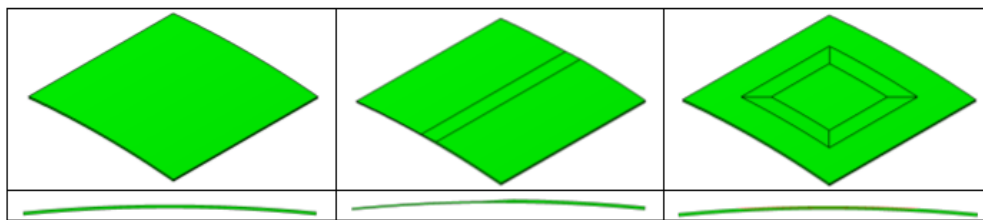


Figure 2 - Part geometry 1 (left), geometry 2 (middle), and geometry 3 (right)

In this paper the part name will reference the part geometry (G), and Serial Number (N). The full part name would be TP-RAPM-017-G-N but the part names will be referenced as 017-G-N as shorthand where G is the geometry and N is the serial number for the specific geometry.

1.4 Manufacturing Equipment and Method

The parts were manufactured on the CF-OaO-RAPM-001 tool clamping fixture with and without the accompanying Pressclave pressure controller. The tooling was placed inside the CMD-TP-RAPM-003 picture frame underneath an AZ31 magnesium bladder. The CF-OaO-RAPM-001 clamping fixture allows for the rapid heating and cooling of the tool face using 180 independent heating and cooling zones. The CMD-TP-RAPM-003-501 picture frame tool allows for the pressurization of the magnesium bladder at elevated temperatures against the thermoplastic part using pressurized gas to superplastic form the bladder against the composite at melt temperatures. This superplastic forming of the bladder is what provides the necessary compaction force against the thermoplastic part.

Traditionally superplastic forming of AZ31 Magnesium is completed at temperatures above 400°C however, at lower temperatures, magnesium will form at low strain rates. This manufacturing method takes advantage of the low temperature forming to debulk, compact and consolidate the thermoplastic composite. The bladders apply compaction pressure to the thermoplastic composite during the heat up. As the composite material reached its melt temperature pressure was increased against the composite. During the cool down, pressure was maintained to ensure consolidation.

1.5 Superplastic Forming Simulation

Prior to manufacturing of the composite parts the following finite element analysis was used to predict material thinning and pressure requirements for the superplastic forming (SPF) of an AZ31 magnesium sheet. The SPF process makes use of extensive microstructural deformation mechanisms. The complexity of these mechanisms makes development of accurate constitutive material models difficult. LS-Dyna is frequently used to perform analysis of SPF processes. LS-Dyna is an advanced general-purpose finite element program capable of simulating complex real world problems. LS-Dyna code models a highly nonlinear, transient dynamic finite element analysis using explicit time integration [2]. The large deformations and material non-linearity of the SPF process requires a solver that it set up for such mechanisms.

2. EXPERIMENTATION

2.1 Magnesium Super Plastic Forming Simulation

The geometry of the model is shown in Figure 3. This is a model of the picture frame tool that the consolidation tool sits inside of. The CMD-TP-RAPM-017-501 tool was located inside the boundary of the picture frame beneath the magnesium sheet. The consolidation tool was removed for a conservative value which would require the magnesium to elongate more in the simulation than in application. The model consists of two components, a stainless steel tool and magnesium sheet. Each component was defined with its own set of properties and material models. The surfaces of each component were used to generate a mesh using uniform thickness shell elements of the type ELFORM 16. ELFORM 16 assigns a fully integrated shell element based on Reissner-Mindlin kinematic assumption. [1] This element type was selected due to its balance in compatibility with implicit simulations without being relatively computationally expensive.

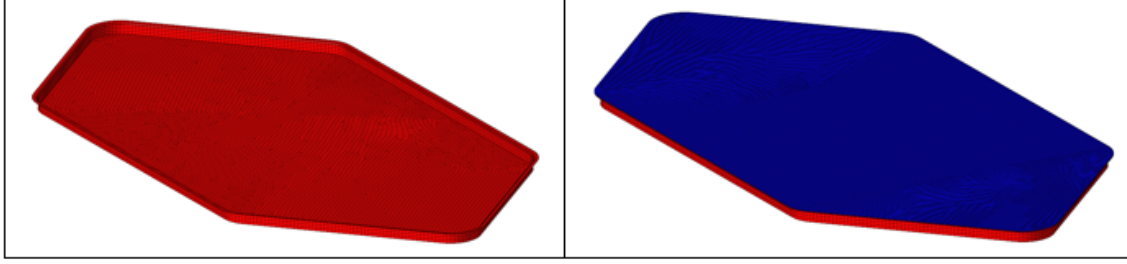


Figure 3 - Forming tool for the superplastic forming simulation (left), forming simulation with the magnesium sheet (right)

The material model for the magnesium sheet was based on constitutive equations developed by Taleff et al [3]. The paper derives strain rate equation and temperature dependent modulus for Mg AZ31 forming at 450°C. Equation 1 shows the strain rate and Equation 2 shows the dynamic unrelaxed temperature dependent Young's modulus.

$$\dot{\epsilon} = 7.494 \times 10^8 \left(\frac{\sigma}{E}\right)^{1.57} \exp\left(\frac{-Q_1}{RT}\right) + 2.517 \times 10^{24} \left(\frac{\sigma}{E}\right)^{5.1} \exp\left(\frac{-Q_2}{RT}\right) \quad [1]$$

$$E(T) = 48,700 - 8.59T - 0.0195T^2 \quad [2]$$

Where:

$$Q_1 = 91 \frac{kJ}{mole} \quad Q_2 = 138 \frac{kJ}{mole} \quad \dot{\epsilon} = \text{Strain Rate}$$

$E = \text{Youngs Modulus}$ $\sigma = \text{Flow Stress}$ $R = \text{Ideal Gas Constant for Air}$
 $T = \text{Temperature (K)}$

MATL 64 in LS-Dyna is a material model that implements properties for a strain rate sensitive elasto-plastic material with power law hardening [3]. The constitutive relationship is of the form is shown in Equation 3.

$$\sigma = k\epsilon^m \dot{\epsilon}^n \quad [3]$$

Where:

$$\sigma = \text{Flow Stress} \quad \epsilon = \text{Strain} \quad \dot{\epsilon} = \text{Strain Rate} \quad k = \text{Strength Coefficient}$$

$$m = \text{Strain Rate Sensitivity Exponent} \quad n = \text{Strain Hardening Exponent}$$

Solving Equations 1 & 2 for a given temperature and strain rate yields the corresponding flow stress. Once a flow stress is determined, the corresponding k, m, and n coefficients were estimated by fitting the curve to the true stress vs true strain curve [3]. Using these two data sets k, m, and n coefficients were selected to best match the curve for a given flow stress/strain rate pair. By selecting coefficients of: k=85, m=0.225, and n=0.005, the following curve fits were generated for 400°C.

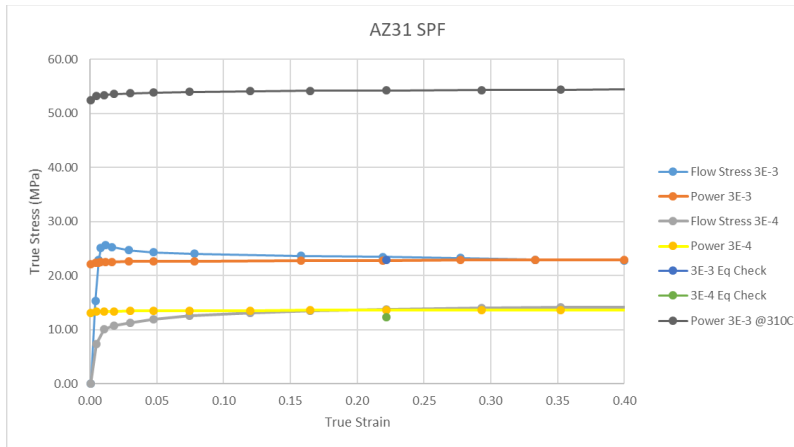


Figure 4 - Power law curve fits vs test data from material models for simulation of superplastic Mg alloy sheet forming [3]

Figure 4 shows that the curve fits for 400 °C were a sufficient representation of the AZ31 alloy up to 0.4 true strain. Unfortunately, no data was available for the parameters of interest for our FEA. The temperature of interest was 310 °C, and the fully formed geometry would likely require true strains greater than 0.5. This material model was unverified in this temperature range but was used in testing prior to part fabrication as the best available approximation. Since the material model is unverified one of the recommendations to gain further confidence in these results would be to perform ASTM E2448 superplastic tensile testing to verify the constitutive equations used here are representative. The tool properties (density, modulus, Poisson's, etc.) were taken from design handbooks, and assigned MATL 20 [2]. However they were of little relevance as the part was assigned to be perfectly rigid and fixed in space. The remainder of the model was created using the following parameters. All nodes on the perimeter of the sheet were fixed from displacement and rotations. All surfaces on the sheet were applied a tabular pressure loading vs time as seen in Table 1.

Table 1 - Pressure schedule for the forming simulation

Time (s)	Pressure bar (psi)
0	0 (0)
5	2.07 (30)
30	9.65 (140)
300	9.65 (140)
330	0 (0)

The sheet was assigned contact properties that included: Static friction coefficient = 0.4, Dynamic friction coefficient = 0.2, Contact stiffness = 0.1, Viscous damping coefficient = 10%.

2.2 Part Processing

Each part was fabricated with a new 1.6 mm (0.063 inch) thick AZ31 Magnesium bladder. The ply kits were cut using a drag blade Gerber cutter and ultrasonically welded together following

collation. These ply kits were then placed within the scribe line of the cure tool inside the clamping fixture. The ply kit prior to consolidation is shown in Figure 5.



Figure 5 - Ply kit on the consolidation tool prior to fabrication

The tool was closed and the part was heated to 385 ± 15 °C. During the heating of the part various pressurization cycles were tested. Following the heating the part was cooled below 220 °C, pressure was released, and the part was extracted from the clamping fixture. To validate the part temperatures, a thermal profile was completed by measure the tool face in every independent heating channel and adjusting set points until all the tool face channels were within the 385 ± 15 °C temperature band. The thermal profile was validated by imbedding thermocouples into the thermoplastic ply kit. The thermocouples were imbedded in the locations shown and produced the thermal validation plot in Figure 6.

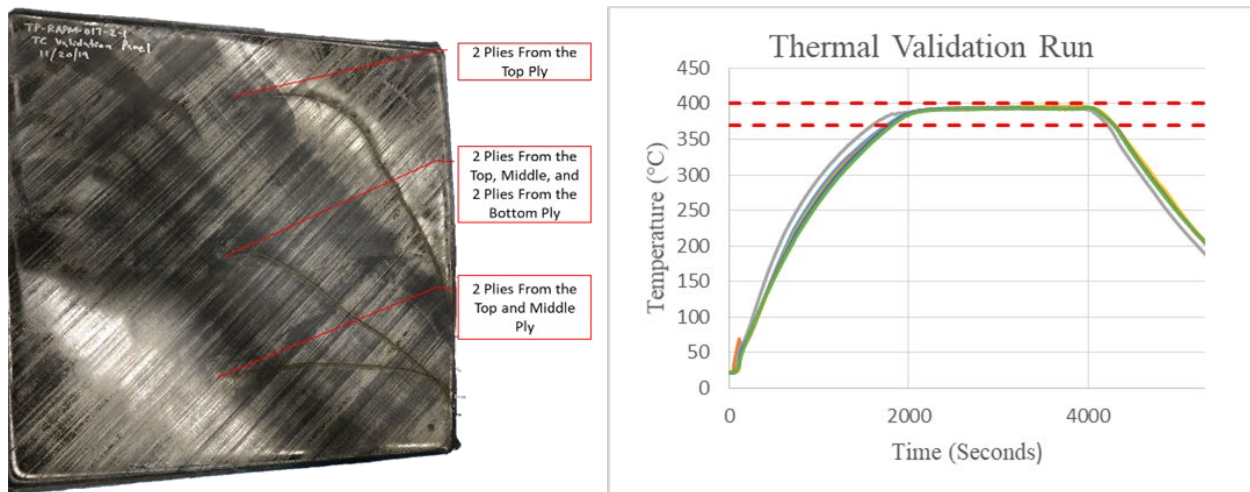


Figure 6 - Thermal profile article thermocouple locations (left), Imbedded part thermocouples in the thermal validation run (right)

2.3 Pressurization

It has been demonstrated that using pressurized argon inside the magnesium bladder will provide a satisfactory surface to the resulting composite parts. There was interest in using a lower cost

nitrogen gas as a pressurization method. As of December 2019 nitrogen costs approximately 2/3 the cost of argon gas. Additionally, argon gas reduces the lifespan of the seals used inside of the automated pressurization equipment (Pressclave), which was designed for use with nitrogen gas. The following pressure cycles were tested with either nitrogen or argon. The control was part TP-RAPM-017-1-1. This part used argon for pressurization, applied an initial pressure of 1.38 bar (20 psi) at room temperature and ramped pressure to 9.65 bar (140 psi) after the lagging part thermocouple reached the melt temperature. The part was then cooled at 10 °C/minute under pressure until the part thermocouple was below 220 °C, then the part was extracted from the clamping fixture.

While evaluating the use of nitrogen gas as the pressurization method, processing steps were altered based on part quality observations. Iterations to the pressurization schedule as well as timing of pressurization were conducted. Initial pressure was changed from 1.38 bar (20 psi) to 2.07 bar (30 psi), 3.45 bar (50 psi), and 4.83 bar (70 psi) to determine the optimal compaction pressure with nitrogen. Pressurization schedules were tuned from 0.70 bar (10 psi) per minute to 0.70 bar per 15 seconds when ramping to the maximum compaction pressure of 9.65 bar (140 psi). The timing of maximum pressurization was evaluated to investigate the part quality from the application of pressure just prior to the polymer melt and just after the polymer melt. All parts were cooled down at the same rate of 10 °C/minute to below 220 °C before removal. A total of 10 parts were fabrication in 3 different geometries throughout the process optimization process. These parts were evaluated with combinations of NDI, surface roughness, dimensional tolerance and destructive inspection.

3. RESULTS

3.1 Simulation Results

The first checks of the FEM output was that the material was flowing at speeds appropriate to the constitutive models. The strain rate target for the process was 3.0E-3, by sampling the largest element deformations over time was produced. Figure 7 shows that all of the elements sampled move at strain rates appropriate to the process over the first 20 seconds. After 20 seconds some of the elements contact the tool and cease to displace further. The pressure ramp rate could potentially be increased without causing the sheet to form too quickly.

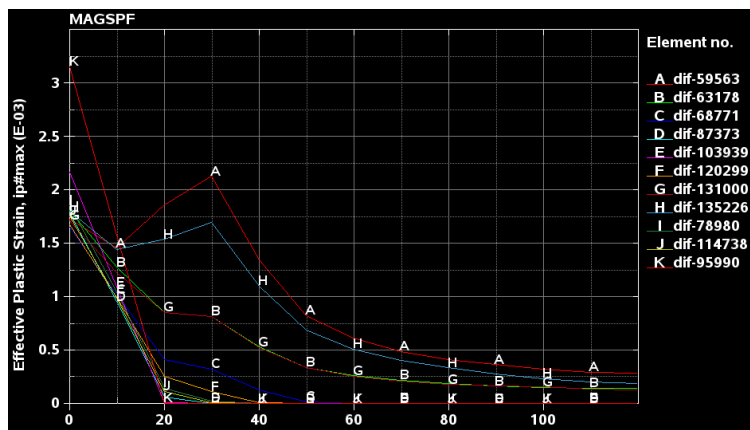


Figure 7 - Elemental strain rate vs time

One of the primary concerns with the lower temperature on forming the AZ31 sheet was the need to apply compaction pressure to the thermoplastic part before the melt temperature of the composite at 310 °C. It was expected that the part was considered to be under full pressure when the magnesium bladder was in contact with the bottom of the picture frame. This ignores the actual consolidation tool and provides a conservative estimate. The part being processed is approximately 18” across. Figure 8 shows the approximate time to contact an 18” span on the tool. Based on this analysis it was shown that the magnesium would contact an 18” wide span on the tool after 30 seconds if pressure was introduced when the tool hit 310 °C. This full coverage time can potentially be reduced by applying pressure earlier in the processing cycle as the part is heated to its melt temperature.

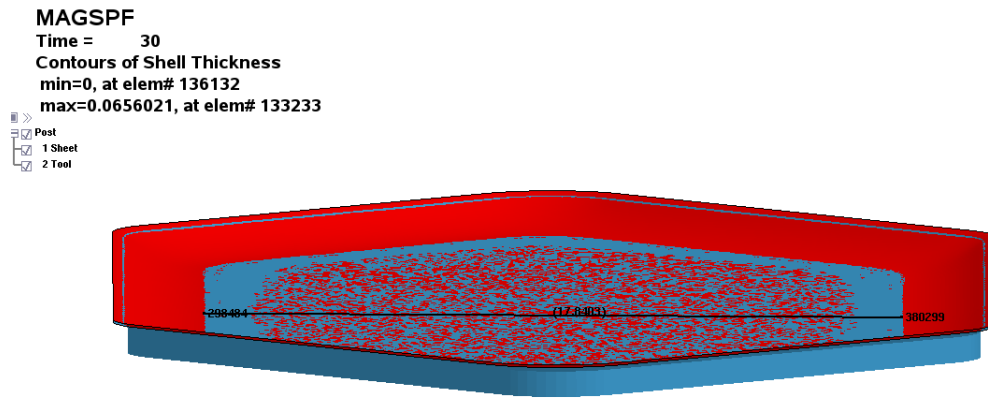


Figure 8 - Sheet contact with the tool at 30 seconds

Standard checks of the total strain (i.e. resulting shell thickness) and stresses were monitored throughout the process to look for any indications of a material failure. Results are shown in Figure 9. Over the full pressure schedule sampled the material only undergoes a strain of approximately 0.15 in the largest forming regions.

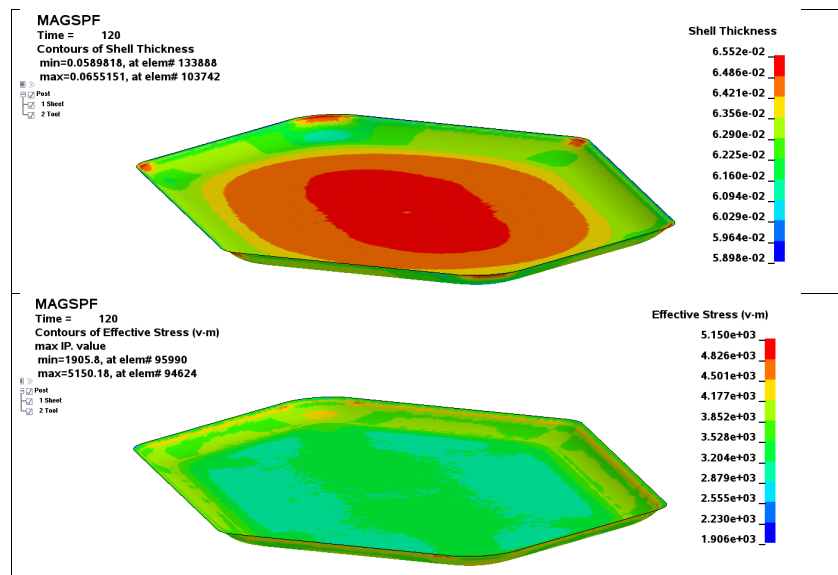


Figure 9 - Sheet thickness at steady state (top), sheet stresses as steady state (sottom)

3.2 NDI and Surface Roughness Results

The following non-destructive inspection (NDI) scans were completed with through transmission C-scans and a surface profilometer. The surface roughness was measured in 9 locations shown in Figure 10. Each location had the roughness measured in the 0°, +45°, -45°, and 90° directions. These values were reported in Table 2.

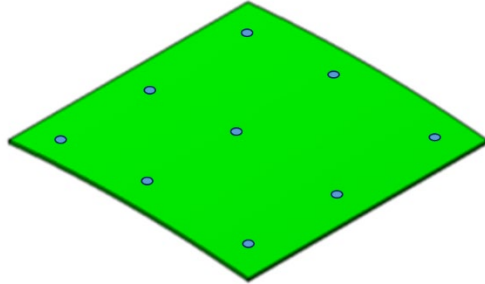


Figure 10 - Locations of surface roughness measurements

Table 2 - Surface roughness measurements

Surface Roughness (μin)	017-1-1	017-2-3	017-2-4	017-3-1	017-3-2	017-3-4
Min	24	15	23	37	39	22
Max	67	101	128	115	104	70
Standard Dev	10.23	21.51	27.97	17.80	18.36	12.89
Average	44.89	53.58	59.94	65.78	71.44	49.16

NDI scans can be seen for each of the three manufactured geometries in Figure 11, Figure 12, and Figure 13. These images were taken under 5 MHz by C-scan. Differences in the surface quality can be seen for each geometry with 017-1-1 being the argon gas control part Figure 11 (a).

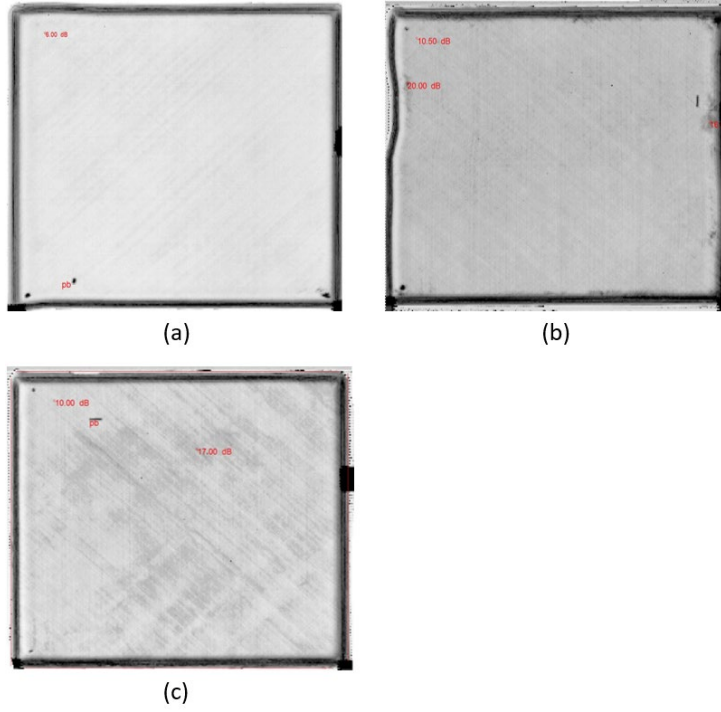


Figure 11 - NDI of flat panel geometry (a) 017-1-1, (b) 017-1-2, (c) 017-1-3

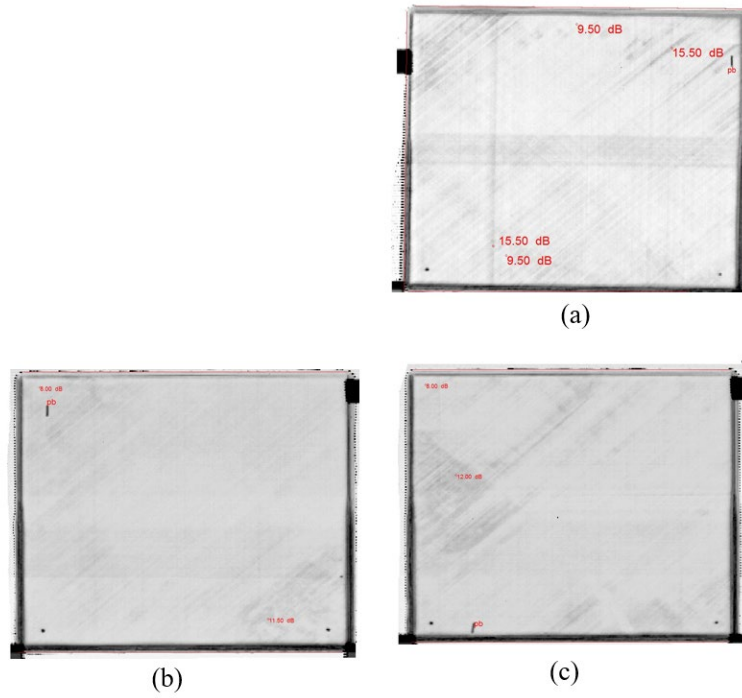


Figure 12 - NDI of ply drop panels (a) 017-2-2, (b) 017-2-3, (c) 017-2-4

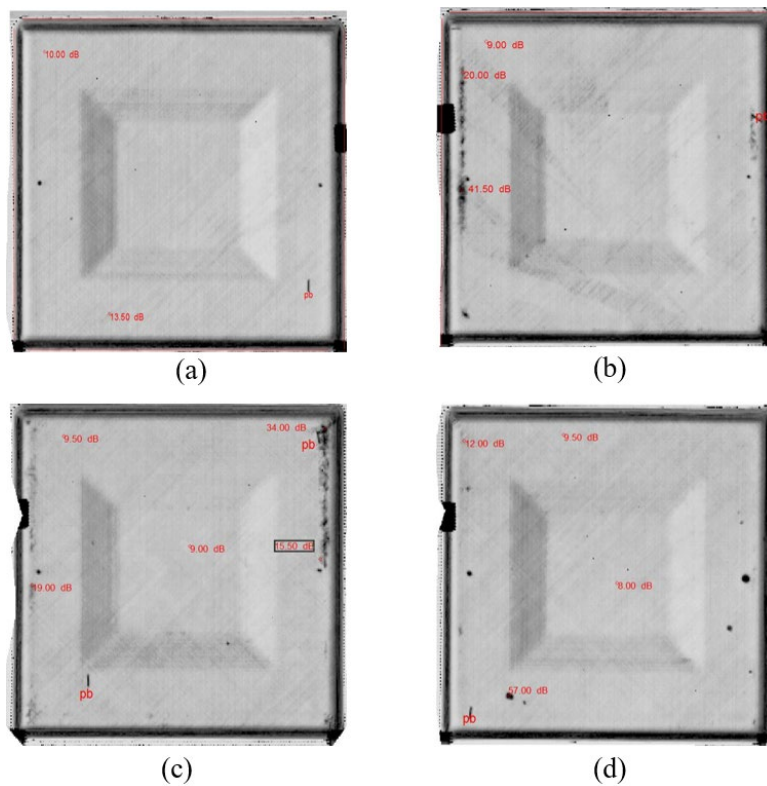


Figure 13 - NDI of picture frame panel (a) 017-3-1, (b) 017-3-2, (c) 017-3-3, (d) 017-3-4

It is expected that panel regions of 32 plies have an attenuation level of less than 20.09 dB and regions with 16 plies have an attenuation level of 14.85 dB. All of the panels have high attenuation in the two locations where the ultrasonic tack weld took place. These tack welds were excluded from evaluation. Future panels would have these welds located outside of the part net trim line. Panels 017-3-4 has good consolidation and a surface roughness that was comparable to the argon panel 017-1-1, however there are 2 dime sized delamination away from the edge. As a result the part would need to be remade.

The panels 017-1-1, 017-3-4, 017-3-3 and 017-3-2 were processed using the same temperature and pressure cycle. These panel 017-3-3 and 017-3-2 had regions of poor consolidation mainly near the edges of the plies. Trimming 30.5mm (1.2 in) from the manufacturing edge of part removed these edge effects from the panel resulting in an acceptable part. These regions are expected to be the last regions of contact for the magnesium bladders. As a method of reducing the manufacturing excess the initial compaction pressure was increased from 1.37 bar (20 psi) to 2.07 bar (30 psi) prior to heat-up. This additional compaction force resulted in a more uniform part consolidation, especially near the edges. The increase in pressure had a negative effect on the surface roughness on the bladder side of the part. The baseline surface roughness from the argon produced panel 44.88 Ra with a max of 67 Ra. The additional pressure panel had an average Ra of 65.78 Ra and a max of 115 Ra. The surface of the resulting panel no longer had a glossy surface and was matte in appearance.

All the parts that were manufactured with nitrogen bladder pressure were loosely adhered to the magnesium bladder following consolidation. Two methods were used to attempt to improve consolidation, improve surface finish and reduce this adhesion. The first method was to sand the bladder smooth with 150 grit sand paper. Comparing 017-1-2 and 017-1-3, where 017-1-3 was fabricated with a sanded bladder, shows that the bladder surface roughness did not have an effect on the part quality. The second method was to apply pressure earlier in the processing cycle prior to the part melt. A thermal profile identified the time when the max part temperature reached the onset of melt for the PEKK material. Parts 017-2-2, 017-2-3 and 017-2-4 used this method with varying room temperature pressure application. The initial room temperature pressures that were used were 2.07 bar, 3.45 bar, and 4.83 bar (30 psi, 50 psi, and 70 psi).

Part 017-2-2 used the initial 2.07 bar (30 psi) application. The resulting part attenuation in the 16 ply region exceeded the attenuation limit. This part would have been needed to be remade. The part 017-2-3 and 017-2-4 used 3.45 bar (50 psi) and 4.83 bar (70 psi) respectively. Both panels had acceptable levels of attenuation. Part 017-2-4 had higher levels of attenuation and a rougher surface finish. It also appears that a wrinkle had formed in the ply drops down the center of the part. The higher initial pressure likely prevented the plies from sliding past each other during processing thereby trapping the wrinkle in the part.

3.3 Ply Placement and Thickness Measurements

The ply drops within the parts geometry 2 and geometry 3. The cut locations are shown in Figure 14. Geometry 2 had ply drop positions measured in sample 2-2-A while Geometry 3 had ply drops measured in location 3-3-A and 3-3-C.



Figure 14- Section cut locations for geometry 2 (left) and geometry 3 (right)

Expected distances between various plies and the actual distance is shown in Table 3. The ply drop ratio that was used was 20:1. A ply was expected to be dropped every 2.76 mm (0.109 in). These ply drops were identified from cross sections shown in Figure 15.

Table 3 Ply Drop Locations in Geometry 2 and 3

2-2-A				3-3-A				3-3-C			
Measure #	Ply Drop	Expected (mm)	Actual (mm)	Measure #	Ply Drop	Expected (mm)	Actual (mm)	Measure #	Ply Drop	Expected (mm)	Actual (mm)
1	1	2.794	1.778	1	1	2.794	2.570	1	1	2.794	1.790
3	2	2.794	2.743	4	2	2.794	2.362	2	2	2.794	2.827
4	3	2.794	3.683	5	3	2.794	1.918	3	3	2.794	4.524
5	4	2.794	3.200	6	4	2.794	1.107	4	4	2.794	3.526
6	5	2.794	2.769	7	5	2.794	2.253	5	5	2.794	3.028
7	6	2.794	3.277	8	6	2.794	2.982	6	6	2.794	2.764
8	7	2.794	0.584		7	ND	ND	7	7	2.794	3.086
	8	ND	ND		8	ND	ND	8	8	2.794	2.482
		Average	2.576			Average	2.199			Average	3.003
		Stdev	1.063			Stdev	0.641			Stdev	0.795

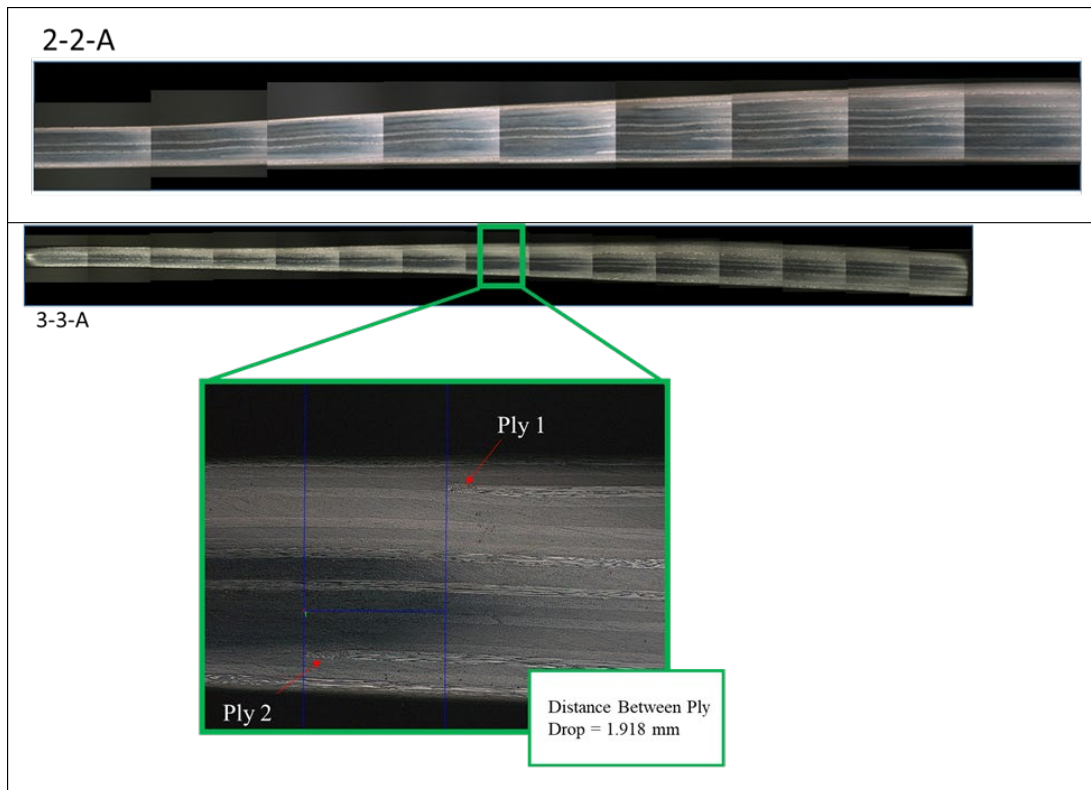


Figure 15 - Cross section of ply drops from 2-2-A (top) ply drop measurement from 3-3-A (bottom)

Bulk thickness measurements were verified in the parts 017-3-1 through 017-3-4. The average, standard deviation, minimum and maximum values are shown in Table 4. The panels have a thickness increase near the edge of the part just prior to the thinning of the laminate at the end. This thickening is occur with a 22 mm (0.87 inch) distance from the manufacturing edge of part. A cross section of thickness is shown in Figure 16.

Table 4 - Bulk thickness values and limits (mm)

	017-3-1		017-3-2		017-3-3		017-3-4		Limit	
	Outside	Inside	Outside	Inside	Outside	Inside	Outside	Inside	Outside	Inside
Avg	4.59	2.30	4.54	2.27	4.58	2.29	4.52	2.26	4.41	2.21
Stdev	0.04	0.02	0.04	0.03	0.03	0.23	0.04	0.02		
Min	4.47	2.25	4.41	2.19	4.47	2.22	4.44	2.19	4.11	2.05
Max	4.79	2.40	4.73	2.45	4.67	2.37	4.70	2.34	4.72	2.36

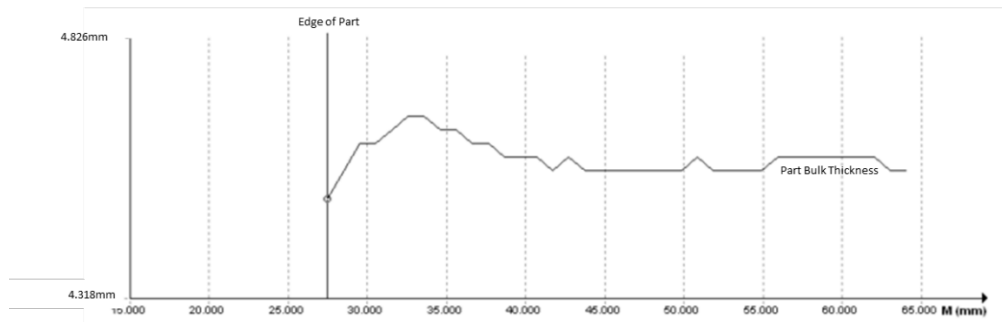


Figure 16 Laminate thickness swell near the edge of part

4. CONCLUSIONS

Panels fabricated using the argon gas show lower attenuation in NDI. The 6 dB attenuation for 32 plies of composite is lower than any other panel fabricated with the nitrogen bladder pressure. The argon bladder pressure part had lower attenuation than parts with regions of only 16 plies as well. In addition the parts fabricated with nitrogen gas have a surface roughness and a matte finish that varied more than the argon panels. Continued development of AZ31 magnesium bladder processing parameters of thermoplastic composites with argon processing gas will need to be completed. Argon processed parts produced higher quality laminates.

High attenuation levels near the edge of laminates due to low initial processing pressure typically occur in the outer 30.5 mm (1.2 in) from manufacturing edge of part. With processing parameters adjustments this edge trim could be reduced to a standard 25.4mm (1 inch) of trim to conservatively account for the edge of part thickening that occurs from the bladder use.

The pressure and temperature cycles used in part 017-1-1 and part 017-2-3 produced the parts with the best results. Both cycles applied sufficient compaction pressure to consolidate the plies without preventing the plies from being able to slide past each other preventing wrinkling.

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