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Final Report

FINAL REPORT ON PROCESSING OF SIX AGCarb/INTREMOLD III CYLINDERS

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REFERENCES

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2.	Davis H. O./Brown J. A. to L. M. Swope "Nozzle Extension- Documentation of Dimensional and Weight Change Data for AGCarb Liner/Intremold III Cylinders" Aerojet Liquid Rocket Company, Materials Engineering Department Report No. ME-71-162 Dec. 9, 1971.
3.	Davis H. O./Brown J. A. "Nozzle Extension- Documentation of Carbon Yield and Other Properties of Candidate Binder Systems for AGCarb Composites" Aerojet Liquid Rocket Company, Materials Engineering Department Report ME-72-122, April 1972.
4.	Crow, Edwin L., et al, <u>Statistics Manual</u> Dover Publications, Inc. New York, 1960.

I. INTRODUCTION

A. BACKGROUND

AGCarb 101, a two-dimensional (2D) reinforced laminated fibrous graphite composite was proposed for use as the material for fabricating the Nerva nozzle extension, due to its excellent combination of high temperature properties. However, in the flange attachment region the calculated stress level of 2500 psi is about double the shear strength level of the 2D composite. A higher cross ply tensile strength than available in the 2D AGCarb was also required. Intremold III, a three-dimensional composite was selected to provide the required interlaminar shear strength and generally more isotropic properties in the attachment flange. This approach necessitated development of a 3D to 2D composite joint. Carbon-to-carbon bonding of the two materials and integral mechanical interlocking were two techniques investigated.

The joining of one fibrous graphite composite to another to obtain optimum strengths at the interfaces required investigation of different joint designs. A total of 12 designs were made. Six cylinders approximately 8-in. dia, x 16 to 18 in. in length and approximately 0.5 in. wall thickness were fabricated. Each of the six cylinders contained a center section of Intremold III with laminated 2-D AGCarb design on either end, utilizing rosette (spiral) and shingle layup fabrication. The design, details of construction and processing up to and including first graphitization are contained in references 1 and 2.

All materials (the raw materials are listed below) and treatments of materials utilized in fabrication of the AGCarb/Intremold III cylinders are applicable to fabrication of a full scale Nerva nozzle extension.

1. AGCarb

AGCarb is the Aerojet-General Corporation designation for fiber-reinforced carbonaceous matrix composite materials fabricated in accordance with processes specified by AGC. Although there are variations depending upon process parameters, AGCarb consists of a graphite fabric, WCA from Union Carbide, impregnated with a carbonaceous filled USP 39 resin from U.S. Polymeric Chemicals Incorporated.

2. Intremold III

Intremold III used in this investigation contained WYB 125, Union Carbide graphite yarn and USP 39 resin. Three helically and three orthogonally wound cylinders were made. These starting materials received the layup, cure, carbonization and graphitization related in reference 1 and 2, plus the additional densification and stabilization processing covered in this report.

3. Densification Binder

The densification binder (impregnant) used to reimpregnate the cylinders of AGCarb/Intremold III was a mixture of 15V carbon pitch and Indene. Indene C_9H_8 is a product of Henley Corp. and 15V is a product of Allied Chemical Corporation. The reinforcements, resins and binder plus the processing covered in this report identify a unique "AGCarb" composite. To date a number identification has not been assigned.

B. SCOPE OF INVESTIGATION

The data presented in this report was taken from the process history and physical changes of the AGCarb/Intremold III cylinders after the first graphitization through the final graphitization. A flow chart of the complete history of process steps is shown in Figure 1.

Of the processing steps shown in Figure 1, four reimpregnation (densification) steps, four cure cycles, four carbonization cycles and a final graphitization cycle are covered in this report. The source for the summarized data was the Process Identification Number (PIN) log and the Process Definition Documents (PDD). These documents, at present, are on file in individual fabrication history logs with ANSC Quality Assurance.

The dimensional measurements on the six cylinders identified as PIN 2001, 2002, 2003, 2004, 2005 and 2006 were taken at the same location as reported in Reference 2. The same template patterns were used for locating reported I.D., O.D., and length measurements. Processing information, including viscosity, specific gravity, and temperature of pitch were taken from the laboratory data sheets. Mixture ratios, additions to the original mix, evacuation times, vacuum readings, and pressure/time cycles were also recorded on the laboratory data sheets. All of these sheets are filed by PIN in the fabrication history logs.

II. SUMMARY/CONCLUSIONS

A. SUMMARY

The process history and physical changes occurring during processing of six AGCarb Liner/Intremold III cylinders for the NERVA SNP-1 Program were documented and recorded in process history logs. The cylinder dimensions were approximately 8 inch inside diameter, 0.5 inch thick wall, and 16 inch length. They contained Intremold III helically or orthogonally wound center sections integrally joined to AGCarb (two dimensional) rosette or shingle lay-up construction sections. Twelve transition joint designs were used to join the AGCarb to the Intremold III sections. Construction and processing through first graphitization is discussed in References 1 and 2. This report includes the documentation from initial graphitization through final regraphitization and covers the process steps impregnation, cure, carbonization and regraphitization.

Each cylinder was impregnated, cured and carbonized four times. All cylinders received a final regraphitization at 5000°F. A mixture of 15V/Indene was used as the impregnant (binder). The mixture ratio was 75 percent by weight 15-V carbon pitch and 25 percent by weight Indene (C_9H_9). Indene, a clear, light liquid, was used as a solvent and to reduce the viscosity of the impregnant mixture. After evacuation to 27" Hg minimum, the cylinders were pressure impregnated at 200-225 psi. The pitch was cured at temperatures up to 500°F in circulating air and carbonized up to 1550°F in a flowing argon atmosphere. Graphitization up to 5000°F was also accomplished in an argon atmosphere. The six cylinders were processed in two groups of three each.

Group 1 included cylinders 2001, 2002, and 2003 and Group 2 included cylinders 2004, 2005, and 2006. Group 2 cylinders received more uniform processing which was reflected in reduced variability in weight pick-up and carbon yield. Weight and dimensional changes were recorded upon completion of each significant step in the processing.

The process variables of impregnant mixture viscosity, specific gravity, and temperature, before and after impregnation, were evaluated in relation to percent weight pick-up, volatile loss and final carbon yield. Linear regression analyses were made to show the percent of variable "y" explained by variable "x". In general the cause/effect relationships were as expected. Some relationships appeared to be non-linear but were not analyzed as such. Correlation coefficients, r , were calculated and many exceeded the critical absolute values at the 95% significance level. Various combinations of variables had high correlation coefficients just below the 95% significance level, indicative of other relationships which could be used for predicting process effects relating to impregnating fibrous graphite composites. Significant negative correlations were observed between cylinder specific gravity and percent weight pick-up during impregnation. A specific gravity goal of 1.40 was established for the cylinders after regraphitization and was achieved with final specific gravities ranging from 1.40 to 1.45. Basic process variable effects were somewhat obscured by the variation in cylinder starting specific gravity. The difference in relative volumes of AGCarb and Intremold fibrous graphite within individual cylinders contributed some difficulty in explaining weight pick-up and carbon yield variability. Variables which had measurable effects

on cylinder properties were viscosity, temperature, evacuation time, removal temperature, and technique for removing excess pitch. Location in the cure oven was also a variable with cylinders near the hotter oven walls tending to bubble or blister more than those centrally located.

Cylinder O.D., I.D. and length shrinkage was, with few exceptions, less than 1% from the initial cured through final regraphitization cycles. The orthogonally wound cylinders shrank more on the O.D. and had less increase in inside diameter than the helically wound cylinders. The dimensional changes in the helical cylinders is believed to be a function of matrix shrinkage and scissoring of the helix yarns while the movement of the orthogonal cylinders is primarily a function of resin shrinkage. As expected, the length of the helically wound cylinders decreased more than the orthogonally wound cylinders. Although the precision of the dimensional measurements was not high, the overall change in cylinder dimensions tended to be in the same general direction between graphitization and regraphitization cycles. The fibrous graphite was not totally stable after initial graphitization. Small changes in cylinder O.D., I.D., and length were recorded between graphitization and regraphitization.

Cylinder dimensional changes induced no delaminations or separations which could be detected by alcohol penetrant tests or radiographic inspection. The overlay hoop windings of PIN 2001 did delaminate, but the delaminations were in a circumferential filament wound area of a joint and not in the AGCarb liner or Intremold III. The Intremold III sections were readily permeable to isopropyl alcohol while the AGCarb rosette and shingle sections were not.

B. CONCLUSIONS

1. The specific gravity goal of 1.40 minimum for the AGCarb Liner/Intremold III combinations was achieved with four pressure impregnations of 15V/Indene.
2. The changes in length, O.D. and I.D. dimensions for the AGCarb Liner/Intremold III cylinders were less than one percent from as-cured dimensions to regraphitization dimensions.
3. The differential strain (resulting from shrinkage) between AGCarb Liner and Intremold III produced no visible separations or breaking of the carbon to carbon bonding between the two materials.
4. The helically wound cylinders tended to increase in outside diameter more than the orthogonally wound cylinders.
5. The net change in wall thickness was greater for the AGCarb Liner material than for the Intremold III material.
6. The AGCarb rosette construction shrinks more on the O.D. than either the AGCarb shingle, Intremold III helical or Intremold III orthogonal constructions.
7. It was demonstrated that two types of fibrous graphite construction can be integrated in structural shapes in the uncured state and subsequently processed to produce fully graphitized, dense components. Strengths of transition joints between differing basic construction types have not been determined to date.

8. The carbon yield of the 15V/Indene 75/25 impregnant mixture is approximately 40 percent, but varied from 20 to 78 in the cylinders.

9. The predicting equations and/or regression relationships between variables derived for the subject cylinders apply generally to the impregnation of fibrous graphite; however, curve slopes and/or constants would be different for structures produced using only AGCarb Liner or Intremold III constructions.

10. Processing results and relationships generally followed expected trends; however, overall tighter control of processing limits produced more uniform results.

III. TECHNICAL DISCUSSION

A. DENSIFICATION BINDER (IMPREGNANT)

A mixture ratio of 75/25 15V/Indene was used to impregnate the six cylinders. The selection was made based on the results of the binder investigation of the Nerva Program, Contract SNP-1, M-6 Plan.

The 15-V pitch is characterized as follows:

Softening Point	90 - 95°C (194 - 203°F)
Specific Gravity (25°C)	1.26 - 1.32
Coking Value	
7 min. 950°C	26% min.
10 min. 700°C	30% min.
Ash Percent	0.25% max.

Indene (C_9H_8) is a clear light liquid. The average assay of the Indene technical is 90-92% pure. The viscosity at room temperature is less than 1 when measured on the Brookfield viscosimeter #1 spindle at 10 rpm. It readily mixes with the 15-V pitch after the pitch is liquid; however, initial mixing is enhanced by agitation.

Viscosity, specific gravity and carbon yield data are given for 15-V Pitch and 15-V Pitch and Indene mixtures in reference 3. However, the viscosity vs. temperature curves shown in Ref. 3 do not show the effects of aging and additions of new mix and Indene. The 15-V/Indene mixtures used for the impregnation of the AGCarb/Intremold III cylinders were used up to five times. Each time the impregnant was exposed to heat up to 200 to 300°F for periods of 4 to 12 hours. In addition the mixture was exposed to preheat temperatures of approximately 200°F for 12 to 16 hours (overnight warmup). Addition of both fresh pitch and Indene were made as needed to replace mixture lost in impregnation and from evaporation during the heating and evacuation cycles.

The characteristic aging effects for the 15-V/Indene mixture after the third impregnation is shown in Figure 2. The viscosity as a function of temperature from 180°F to 280°F is shown for the mix prior to impregnation and after impregnation. Viscosity differences are much greater at the lower temperatures. For example, the before viscosity at 200°F is 40 to 50 cps and the after viscosity is 1000 cps at 200°F. Based on the changes in viscosity and specific gravity, it appears that aging consists of primarily a loss of Indene and some low molecular weight hydrocarbons.

The amount or degree of polymerization was not determined. The carbon yield after 5 impregnations was approximately 40 percent. The same yield was obtained from a fresh mixture of 15V/Indene. A high degree of polymerization would be expected to increase the carbon yield. The molecular weight distribution of the new batch vs. the aged batch would possibly be different at the same viscosity. The effects of aging are compounded by the additions of 15V and Indene.

B. PROCESSING - IMPREGNATION, CURE, CARBONIZATION AND REGRAPHITIZATION

Each of the cylinders was processed four times through the impregnation, cure and carbonization. The impregnation was accomplished in a sinter-seal impregnator, serial No. B-1055 manufactured by National Sinter-Seal Div., N.B. Newcomb Co., Inc. Cure was in an air circulating oven and carbonization was accomplished in a welded retort in a resistance heated furnace. The cylinders were impregnated individually, but were cured and carbonized in groups of three. PIN 2001, 2002, 2003 were processed together as the first group. PIN's 2004, 2005 and 2006 were the second group. After the fourth carbonization the cylinders were regraphitized to complete the process.

1. Impregnation Processing

The impregnation process was accomplished within the following parameters and limits.

a. Mixture Ratio (15-V-75% by weight/Indene-25% by weight)

Additions to the mixture were made in the same ratio; however, if viscosity was above 50 cps at $250 \pm 25^{\circ}\text{F}$, Indene was added to lower viscosity below 50 cps. Viscosity was measured at temperature with Brookfield

Viscosimeter RVF #1 Spindle at 10 rpm. Specific gravity was not used as a controlling parameter; however, it was measured with a hydrometer at approximately the same temperature that the viscosity was measured.

b. Heating Cycle

The mixture was heated overnight in an air circulating oven with a vent. The oven was set at approximately 200°F for overnight heating and raised to 300°F two to three hours prior to planned use of the pitch mixture. Viscosity and temperature were checked and additions made until temperature was between 225 to 275°F and viscosity was below 50 cps.

After the pitch mixture reached the required temperature and viscosity, it was placed in a metal container in the impregnator. (In some instances, the heating was accomplished in the metal container.) The container was wrapped with a Ramaflex heater blanket type F-1479-1, Rama Corporation, San Jacinto, Calif. The blanket was insulated with asbestos wraps and taped to the container. Efforts were made to cover the heater blanket completely in case of pitch spillage or overflow. After the container was placed in the impregnator, the thermistor (control) was turned to the "On" position. The temperature controller for the blanket was set at $350 \pm 25^\circ\text{F}$. For some of the final impregnations, the controller was set at a lower temperature or turned "Off" approximately 30 minutes prior to end of pressurization cycle. This was to reduce run-off and bubbling as the cylinders were removed from the impregnant. The changes and their effects are discussed in more detail below.

c. Preparation of Cylinder for Impregnation

The cylinders were weighed and measured prior to each impregnation. The data are recorded in Table I through VI under column headed weight of part.

The cylinders were impregnated individually; however, preparation for each followed approximately the same sequencing. After weighing the cylinders were attached with wire to the hydraulic vertical lowering ram in the lid of the impregnator. The extension of the ram and wire was checked each time to insure sufficient length for immersion of the cylinder into the mixture when the ram was fully extended. A metal weight was also attached to the cylinder to insure complete immersion.

The impregnator lid was closed and locked into position with the hydraulic arm in the retracted position. The cylinder was directly above the heated pitch mixture prior to starting the evacuation cycle so that it could be lowered into the solution after the evacuation procedure was completed.

d. Evacuation

With the cylinder above the hot pitch mixture, the impregnation chamber was evacuated from three to five minutes to a vacuum gage reading of 25 to 29 in. Hg.

The cylinder was then lowered into the pitch mixture. Vacuum was maintained for 0.5 to 5 minutes with the cylinder immersed in the pitch solution prior to pressurization.

e. Pressurization

After the cylinder was immersed, the impregnation chamber was pressurized with nitrogen to 200 to 225 psi. Pressure was maintained for 2 ± 0.25 hours. Pressure and temperature were checked approximately every 15 minutes until the end of the pressure/temperature cycle. At the end of cycle, pressure was released to one atmosphere and the impregnator was opened after raising the cylinder out of the pitch mixture.

2. Curing Cycle

Immediately after removal from the impregnator, and prior to cure the cylinder was wiped with a cloth to remove surface excess material. The cylinder was weighed and the value was recorded on the Impregnation History Sheet. The weight history for each cylinder and cycle have been summarized in Tables I through VI. The weight of cylinder and impregnant is shown in column WP+I. The weight of the impregnant, WI, and percent by weight of part pick-up, % pI is also shown in Tables I through VI for each impregnation cycle.

The cylinders were reweighed after cure and this data appear in the column WP+CuI, weight of part plus cured impregnant weight. The volatiles (weight loss) and the cured impregnant weight are also shown. Percentage figures are also given for volatiles in Tables I through VI.

3. Carbonization

After weights were taken, the cylinders, in groups of three, were placed in a stainless steel (AISI 347) retort. Each cylinder was individually wrapped in graphite cloth and/or carbon felt. An inert atmosphere was

maintained during the carbonization cycle. Argon, 99.9999% pure, was used. Chopped pieces of .045-in. dia. titanium (Ti 55A) wire getter was placed in the inlet tube of the retort prior to each carbonization cycle, to remove any moisture or oxygen in the argon.

The standard three-day carbonization cycle per ANSC Specification 90293, was used for converting the pitch to carbon. The cycle includes a six-hour hold at 1550°F. A flow of argon through the retort at 10 to 20 CFH is maintained during both the heatup and cooldown phase of the carbonization cycle. Cooldown rate did not exceed 100°F per hour. The instructions and precautions given to technicians for the carbonization cycle are listed in Appendix I.

After each carbonization the cylinders were weighed and measured. The weights are recorded in Tables I through VI. The percent carbon yield, cumulative percent carbon were calculated based on the weight of carbon and are shown in Tables I through VI for each of the four impregnations, cure, carbonization cycles.

The specific gravity after each carbonization was determined by water displacement and corrected for water absorbed by reweighing the wet cylinder immediately. The water absorb is subtracted from weight in H₂O prior to calculating specific gravity.

C. REGRAPHITIZATION

After the fourth and final carbonization, each of the cylinders were regraphitized at 5000°F. The actual graphitization cycles for the cylinders are filed in the individual PIN folders. The weights after graphitization are

shown in Tables I through VI. A full set of I.D. and O.D. measurements at each section and position were taken after the regraphitization.

D. DISCUSSION OF RESULTS

The goal of the densification processing was to achieve a minimum of 1.40 sp. g. in as few impregnations as possible. Three conditions controlled the number of impregnations. They were (1) specific gravity of cylinder after first graphitization (2) percent pick-up of impregnant and (3) carbon yield of the impregnant. The first two are a function of the porosity of the cylinder. Items (2) and (3) are a function of the process variations. The porosity of the cylinders was not measured; therefore, the discussion of results and effects of variables is limited primarily to process variables.

1. Specific Gravity - Cylinder

The specific gravity for each cylinder after the first graphitization, after each carbonization and after regraphitization is shown in Tables I through VI. The beginning specific gravities were somewhat different for the six cylinders and the final (after regraphitization) specific gravities ranged between 1.40 and 1.46. The bar graph in Figure 3 show the comparative beginning and final specific gravities. In general, the second batch of cylinders showed a greater increase in specific gravity. The specific gravity results for PIN 2004, 2005 and 2006 are consistent with the more uniform processing of the second batch of cylinders --discussed below. However, to ascertain the specific gravity differences, if any, related to the porosity of the Intremold III sections, the percent by weight and volume of AGCarb in each cylinder would have to be evaluated as a possible contributor to the variation. The general qualitative differences in volume are shown in Figure 1, reference 1. For example

PIN 2006 and 2006 had the lowest starting density and they have the greater percent by volume of Intremold III based on the cross section lay-out.

After the cylinders are sectioned the differences in specific gravity of AGCarb and Intremold III can be readily ascertained.

2. Weight Pick-up of Impregnant

It was anticipated that percent weight pick-up of impregnant based on the weight of the cylinder should correlate negatively with the specific gravity. (See further discussion and correlation coefficients for this and other relations of process variables in Statistical Analysis.) The trend observed was a negative correlation, i.e. the lower specific gravity cylinders had higher percentage weight pick-up. Variations in drainage, blistering during cure also affected the pick-up percentages.

The percent weight pick-up by cylinder for each impregnation is shown in Figure 4. There was a large variation in percent pick-up between cylinders; however, the trend indicates a sharp decrease in percent pick-up after the first impregnation and a lesser subsequent decrease.

The second impregnation for PIN 2001 and the third impregnation for PIN 2003 did not follow the general trend for decreasing weight pick-up with each successive impregnation. The 2nd impregnation percent weight pick-up for PIN 2001 was lower than the 3rd. The reason for the low pick-up for the second impregnation could not be established.

The 3rd percent weight pick-up for 2003 was lower than the fourth. Ten pounds of Indene were added to the mix just prior to the 3rd impregnation of PIN 2003. These are two examples of the effect of variations in

weight pick up. Better control and uniformity were achieved for the 2nd batch of cylinders, PIN 2004, 2005 and 2006.

The following is a list of variables which were factors affecting percent weight pick-up. However, all were not quantitatively measured in this investigation. Those measured in this investigation are discussed further in the Statistical Analysis Section. Some have been isolated in other studies for the M-6 Program, reference 3.

- (1) Porosity of Cylinder (not quantitatively defined)
- (2) Evacuation time for cylinder above solution and immersed in solution
- (3) Viscosity and specific gravity of solution
- (4) Temperature of mixture viscosity and specific gravity when cylinder was removed from mixture.
- (5) Pressure when cylinder is removed from mixture
- (6) Specific Gravity of cylinder
- (7) Temperature of cylinder when excess is removed by cloth wiping
- (8) Technique for removing excess pitch

In addition the cured weight retained is significantly affected by the condensation of volatiles on the cylinders. (This was previously recorded for flat test panels in the M-6 program.) The condensation occurs during evacuation of the chamber prior to immersing the cylinder. If pitch was introduced from an outside reservoir after evacuation was completed, less time would be allowed for condensation to occur. For the second batch of cylinders the time

above solution was reduced as much as possible to reduce variation in cured weight pick up caused by condensation of Indene and other low molecular weight hydrocarbons on the cylinders.

3. Carbon Yield - Binder

In relation to number of impregnations required, the weight retained after each impregnation, cure and carbonization was the controlling variable. The weights retained (carbon yields) are shown in Table I through VI. In general the percent carbon yield based on weight of impregnant was higher for the initial impregnation. One exception was PIN 2001 which had a higher yield (based on weight of impregnant pick-up) for the 2nd impregnation. However, the pick-up was very low for the 2nd impregnation of PIN 2001. Impregnation number one for PIN 2002 and number three for PIN 2003 also had low weight pick-up and comparatively high carbon yield. As can be seen in Figure 5, the reproducibility for weight pick-up and carbon yield was much better for the 2nd set of cylinders PIN 2004, 2005, 2006 processed.

The erratic results for PIN 2001, 2002, and 2003, shown in Figure 5 are a result of several inconsistencies in processing. The cause-effect relationship cannot be individually assignable; however, the following differences for the 1st and 2nd batch of cylinders existed.

	<u>1st Batch</u> <u>Pin 2001, 2002, 2003</u>	<u>2nd Batch</u> <u>Pin 2004, 2005, 2006</u>
Evacuation Time		
a. Above solution -	up to 5 min.	up to 3.5 min.
b. Immersed -	up to 5 min.	up to 0.5 min.
Blanket Thermistor Setting	350°F	350°F
Time Blanket on	2 hrs.	1-1/2 hrs.
Pressure	200 - 225	200 - 225
Temperature of Pitch after Cycle	up to 295°F	up to 250°F
Method of Removing Excess Pitch	Wipe with cloth - some bleeding observed.	Wipe edges and surface lightly - very little bleeding observed.
Cure Cycle in Oven	Open to direct hot air from oven.	Place Al foil between oven wall and cylinder

These changes were made after reviewing the laboratory data sheets for the first batch of cylinders and as a result of observation made during the impregnation processing.

In addition to the variations discussed above there is a difference in percentage by weight and by volume of AGCarb and Intremold III between the various cylinders. The difference by weight is not measurable; however, the difference by volume can be seen in the cross section of Figure 1, reference 2. Furthermore, there were undetermined differences in the density of the Intremold III sections which contributed to variations in impregnant pick up as well as the initial and final specific gravity of the cylinders.

4. Impregnant Process Variations

In addition to the variations between cylinders and in the impregnation process technique; there were variations in the impregnant (binder) mixture. Before impregnation and after impregnation viscosity curves are shown in Figure 2 for 15V Pitch/Indene mixture. The curve for after impregnation is typical; however, the amounts of Indene added, amounts of fresh mix added, heating times and temperatures as well as number of times reused would affect the curve. The mixture after impregnation generally required additions of Indene and/or replacement 15V/Indene mixture. The viscosity curve in Figure 2 for the before mixture represents a fresh batch and the after curve represents a batch (plus additions) after three impregnations. Prior to reuse, Indene had to be added to bring the viscosity into the required limits, 40 - 50 cps. The viscosity over the temperature range shown in Figure 2 was not determined after each use; however, the temperature, viscosity and specific gravity for the mix prior to the impregnation of each cylinder is shown in Table VII. Two batches of 15V/Indene were used. The first batch of 15V/Indene was used five times (with additions). This included the 4 impregnations of 2001, 2002 and 2003 plus the first impregnation of PIN 2004, 2005 and 2006. The second batch was used three times, the 2nd, 3rd and 4th impregnations of PIN 2004, 2005 and 2006.

5. Dimensional Changes of Cylinders

The initial processing of the AGCarb Liner/Intremold III Cylinders was discussed in reference 1 and 2. Dimensional changes from Cure through Graphitization were recorded in reference 2. The major changes in

I.D., O.D. and length measurements occurred during the processing up to and including the first graphitization. However, a continual check on dimensional changes was made during the impregnation, cure, carbonization and a final regraphitization processing.

In general, shrinkage and/or distortion have been related to delaminations in fibrous graphite composites: The combination of AGCarb in rosette and shingle lay-up with Intremold III in both orthogonal and helically filament round cylinders was suspect for non-uniform dimensional changes even after first graphitization. Therefore, a large number of measurements were taken at the end of each carbonization or graphitization process cycle. Tables VIII, IX, X and XI are examples of the number of measurements taken for the outside diameters of each cylinder and the calculations made to ascertain the change in O.D. at each point. Tables VIII and IX contain measurements and calculations for PIN 2001 and Tables X and XI for PIN 2002. The location of measurements are shown in Reference 2 in Table VIII and Figure 1. The type of fibrous graphite structure at the specific location is also identified in the tables of this report for ease of reference.

The measurements and calculations were repeated for the remaining four cylinders, but are not included in this report. In addition, inside diameter measurements and changes were recorded and are on file with the process history logs. However, the percent change is recorded in this report for all measurements including length and these are discussed below.

Templates were used to assist in repeating the location of measurements. However, there was still some variation in location which could not be eliminated. In addition, a somewhat variable thickness in the carbonized pitch on the O.D. and I.D. contributed to measurement error.

There were trends observed in the diametral changes related to the material and/or type of construction. It is believed that a mandrel is necessary to prevent excessive movement and control dimensions, particularly ovality. Use of the graphite mandrels, reference 2, for post cure, first carbonization, graphitization and regraphitization, reduced the changes which would have occurred if the cylinders had been freestanding during these process steps.

Primarily comparisons are made for cylinders and construction for the following conditions.

- Original cure measurements versus final (regraphitization) measurements
- Fourth impregnation carbonization measurements versus regraphitization measurements
- First graphitization and regraphitization

a. Outside Diameters

The percent changes between the following process steps are shown in Table XII, XIII, XIV, XV, XVI, and XVII for cylinders PIN 2001, 2002, 2003, 2004, 2005 and 2006 respectively:

	<u>From Completion of</u>	<u>To Completion of</u>
(1)	First graphitization	Carbonization after 2nd Impregnation
(2)	Carbonization after 2nd Impregnation	Carbonization after 3rd Impregnation
(3)	Carbonization after 3rd Impregnation	Carbonization after 4th Impregnation
(4)	First Graphitization	Carbonization after 4th Impregnation
(5)	Carbonization after 4th Impregnation	Regraphitization
(6)	First Graphitization	Regraphitization
(7)	Original Cure	Regraphitization

For the first three comparisons only one diameter at each section was compared. For comparisons (4) through (7) above, four diameters at each section were compared.

With very few exceptions, the percent change was less than one percent. Overall the percent changes were rather uniform regardless of location or section of the cylinder being measured. Furthermore, there were only small differences between cylinders.

Both shrinkage and expansion occurred as evidenced by both negative and positive percent change, (the limitations of measurements are discussed above). Some of the measurements indicate out-of-roundness or a change in roundness; however, the outside surfaces were rough. If any distortion occurred, it was less than one percent of the diameter.

(1) Graphitization to Regraphitization

Of primary interest was the change from Graphitization to Regraphitization. As shown by Figure 6, the Helix cylinders 2001, 2003 and 2005 increased in diameter more than the orthogonal cylinders 2002, 2004 and 2005 between original graphitization and regraphitization. The movement of the AGCarb liner material on either end of the Intremold III tended to move in the same general direction. For example, the orthogonal cylinders had more negative percent Δ O.D.'s than the helix cylinders.

The etiology of this phenomenon is not certain. The shrinkage which does occur is in the pitch, matrix. Strain produced by this shrinkage will be a result of stress from the matrix material. The matrix material bonds or adheres to the reinforcement if it is exposed, but more likely to the carbonaceous matrix already present. The helix wound cylinders are less restricted in the radial and axial direction because of the scissoring action of the helically wound yarns. This movement can take place without observable damage to the composite. However, any movement possibly damages the matrix and/or matrix to reinforcement bond. An increase in diameter should have produced a decrease in length if scissoring occurred unless there was slippage at the joints. The change in lengths is discussed in more detail later. Briefly PIN 2005 showed a comparatively large reduction in length. PIN 2001 had practically no shrinkage. The length measurements for 2003 were either misplaced or not taken after first graphitization.

(2) Original Cure to Regraphitization

The comparison of percent change in outside diameters from cure to regraphitization is somewhat arbitrary because of the pitch retained on the outside surfaces of the cylinders. There were both increases and decreases in outside diameters of the cylinders. The percent change and average at each location is shown in Tables XII through XVII. The average change for the four positions at each section is shown as a bar graph in Figure 7. Again the orthogonal cylinders showed less increase in diameter. All average percent changes were negative with the exception of Section B in 2002 and Section C in 2006. Section C had both positive and negative changes; therefore the average of $+0.01\%$ is not meaningful. Section B of 2002, $+0.21\%$ is in a rosette/Intremold joint area.

The trend for orthogonal cylinders is for a decrease in outside diameter; however, the maximum average decrease was -0.17% . The maximum shrinkage in O.D. for the AGCarb material was -0.88 percent in the A Section of 2004. Section A is a rosette section.

Helically wound sections increased in diameter up to 0.86 percent for cylinder 2003. As stated above, the helix cylinders can scissor around the radial pins to relieve shrinkage stress. The orthogonal cylinders are more restrained and shrinkage occurs in wall thickness with a resulting decrease in outside diameter.

b. Inside Diameters

The measurements after process step completion are the same for inside diameters as for outside diameters listed above. The percent change for the six cylinders are listed in Table XVIII through XXIII. The

percent changes from first graphitization through fourth (final) carbonization were negative in almost every instance. This decrease in diameter is mostly from pitch buildup on the I.D. surface and does not represent any significant movement of the cylinders.

(1) Graphitization to Regraphitization

There were both increases and decreases in inside diameters. The percent changes tended to be more positive for helically wound cylinders indicating larger increases in inside diameters. The same was true for the outside diameters of helix cylinders discussed above. The helically wound cylinders are freer to move because of the possibility of scissoring of the hoop yarns between the radial pins. Again there was neither increase nor decrease greater than one percent.

The average percent change for each section of the cylinders including the joints, Intremold III and AGCarb sections are shown in Figure 8. In general a decrease in inside diameter corresponded to a decrease in outside diameter; however, there were many contradictions.

(2) Original Cure to Regraphitization

Figure 9 shows the average percent change from original cure dimensions to regraphitization for all sections of the six cylinders. The values are also listed in Tables XVIII through XXIII. With three exceptions all percent changes were positive with increases in inside diameter up to .84 percent. The three sections with negative percent change were from cylinder 2004 in the AGCarb rosette or AGCarb rosette/orthogonal Intremold III sections. These values are within 0.10 percent.

The pattern trends for movement of helix versus orthogonal were again apparent. The helically wound cylinders showed greater increases in percent change than did the orthogonal wound cylinders. Secondly the greater increase in percent change of inside diameters corresponded to more positive percent increases in outside diameters. Again, there are many contradictions.

c. Change in Length

The percent change in length at four locations, 0°, 90°, 180° and 270°, for all the cylinders are shown in Table XXIV. The length measurements for (1) original cure (2) first graphitization (3) 4th impregnation carbonization and (4) regraphitization are recorded and percent change between process steps was calculated from the recorded values. The average changes were negative showing a decrease in length between process step except for from "fourth impregnation carbonization" to "regraphitization". For this cycle PINs 2001, 2002, 2003 and 2006 were positive.

These increases in length between fourth impregnation carbonization, although small, are opposite from the expected. Additional shrinkage occurs during graphitization of the carbonized pitch matrix; therefore a decrease in length was anticipated. Cylinder 2005 did shrink as expected; and the average percent change was a negative .15 percent.

All cylinders had a decrease in length from "original cure" to "regraphitization"; however, cylinder 2005, a helically wound cylinder had the largest decrease of -0.92 percent. The next largest decrease was also a helix cylinder. PIN 2001 had a decrease of -.29 percent. Results from flat

plates of AGCarb liner material show practically no change in length or width; therefore, it is assumed that the changes in lengths are primarily a function of the Intremold III. The helical wound cylinders would be expected to experience greater length changes than orthogonal cylinders. PIN 2003 was an exception.

d. Change in Wall Thickness

The change in wall thickness was not measured; however, a review of the I.D. and O.D. measurements show a decrease in wall thickness with few exceptions. The wall thickness of PIN 2003 in the Intremold III helical wound section apparently increased in thickness. PIN 2005 at Section D, helically wound, also had a slight increase in wall thickness. Changes in wall thickness of 1 to 4 percent have been measured on flat plates of AGCarb. Based on the comparative O.D. and I.D. measurements it appears that changes greater than 4% and up to 10% may have occurred for the cylinders.

e. Inspection

Each cylinder was alcohol penetrant inspected after regraphitization.

Cylinders had been previously checked after Post Cure, Carbonization and Graphitization. Results are recorded in PIN Process History logs. Briefly PIN 2006 had delaminations for almost 360° around the cylinder .050 to .130 from I.D. after original graphitization in the IM shingle section. The depth could not be ascertained. The AGCarb liner Joint Design XVI of PIN 2006 was damaged on the sharp edge after carbonization to a depth of approximately .250" from inside diameter extending approximately 3".

Both the delamination and damage were sealed with pitch and/or removed during subsequent machining. A small indication from the damaged area in the rosette section is still visible. However, with this exception there were no delaminations or separations observed during alcohol penetrant check except on the circumferential filament windings of PIN 2001. The results of radiographic inspection were also negative as to delaminations and/or separations. There were; however, some questionable areas. Radiographic prints are on file.

The Intremold sections were porous. In an attempt to compare the relative porosity, alcohol permeability tests were conducted. The time for 10 cc of isopropyl alcohol to penetrate from I.D. to O.D. was the gage used for comparison of Intremold and AGCarb sections. The results are contained in Table XXV. There was no observable penetration of the AGCarb with 10 cc of isopropyl alcohol after 4th impregnation carbonization or after regraphitization. There was no penetration of the Intremold III material of cylinders 2004, 2005 and 2006 after fourth impregnation carbonization. (PIN 2001, 2002 and 2003 were not checked). On the other hand all Intremold III sections were penetrable by isopropyl alcohol after regraphitization. The time varied from 5 seconds to 30 seconds.

PIN 2006 orthogonal and PIN 2003 were the least permeable. That is, more time was required for the alcohol to appear on the outside of the cylinder. These cylinders also had a greater spread in times to penetrate than the other cylinders. It is anticipated that these results would have related to specific gravity of the Intremold III. Since the cylinders were not tested this could not be verified.

f. Machining

After regraphitization and final measurements were taken, the cylinders were machined in accordance with applicable drawing. Some modifications were made to fit parts as fabricated. The nominal wall thickness was approximately .400 - .500.

The cylinders were machined with standard carbide cutting tools on an engine lathe. The cylinders were held to male molds for machining the outside diameters. They were chucked on O.D. to machine to inside diamensions. Each cylinder was faced to length with the carbide cutting tool. There were no machining discrepancies noted and apparently no damage to Intremold III or AGCarb material.

E. STATISTICAL AND GRAPHITTICAL ANALYSIS

A statistical analysis of some of the measured cylinder data and process variables was conducted. Statistical analysis was used to verify and/or show which variables could be expressed as a function of another variable. These results were not treated rigorously, but used for trend indications and to show cause/effect relationships. After completion of the statistical analysis, additional graphs were made to depict the relationship between variables. The graphs and statistical analysis are supplemental to the preceding discussion of the results.

Primarily Linear Regression analysis was used in comparing variable "y" with another variable "x"; however, selected variables were analyzed using

Multiple Linear Regression analysis. For linear regression analysis those combinations which would be expected to interact were analyzed and the correlation coefficient r was calculated. Most of relationships identified were obvious and regression of variable "y" upon variable "x" was expected. A 5% significance level was used to test the computed values of r . If the computed value of r exceeded the critical value from Table 7, reference 4, the null hypothesis that the population of "x's" and "y's" had zero correlation was rejected. However, some of the correlation coefficients which were lower than the critical value at the 95% significance level show trends and should be investigated further.

The line of best fit was not computed for the linear regression lines shown in the graphs but were estimated from the plotted data points.

The results of both the Linear Regression and Multiple Linear Regression analysis are discussed in the following paragraphs.

1. Linear Regression Analysis

a. Correlation Matrix #1

The computed correlation coefficients r are shown in Figure 10 for the following variables:

- (1) Starting specific gravity of cylinder
- (2) Final specific gravity of cylinder
- (3) Percent weight pick up first impregnation based on weight of cylinder
- (4) First carbonization specific gravity
- (5) Second carbonization specific gravity

- (6) Third carbonization specific gravity
- (7) Fourth carbonization specific gravity
- (8) Percent weight pick up second impregnation based on weight of cylinder
- (9) Percent weight pick up third impregnation based on weight of cylinder
- (10) Percent weight pick up fourth impregnation based on weight of cylinder

All combinations compared have correlation coefficients r listed in the correlation matrix, Figure 10. The correlation matrix is symmetric; however, each correlation coefficient has been listed only once. Those combinations without correlation coefficient in the matrix were not compared. After reviewing the data, the computation for the combinations not shown were not considered necessary.

There were no significant positive correlation coefficients for the combinations "tested" in Figure 10. A positive r indicates that the "y" variable increases with an increase in the "x" variable.

The significant negative correlation coefficients were between the following cylinder properties and/or process parameters.

- (1) Starting Specific Gravity of Cylinder vs. Percent weight pick up first impregnation based on weight of cylinder.
- (2) Starting Specific Gravity of Cylinder vs. First Carbonization Specific Gravity.
- (3) Starting Specific Gravity of Cylinder vs. Second Carbonization Specific Gravity
- (4) Starting Specific Gravity of Cylinder vs. Percent Weight pick up second impregnation based weight of cylinder.

- (5) Percent weight pick up first impregnation based on weight of cylinder vs. first carbonization specific gravity, (cause for this relationship is a third variable-starting specific gravity of cylinder).

The variable listed on the left is most likely the causal variable.

There were other trends indicated by the relatively high values for some of the computed correlation coefficients which did not exceed the selected critical values at the 95% significance level.

Some of these however, are merely related and the cause for both variations is due to a third variable or some variation not measured.

Of the variables measured and compared in correlation Matrix #1, the starting specific gravity is of primary importance in its effect upon (1) percent pick-up as well as (2) specific gravity after further processing. An estimated regression line of specific gravity (initial) versus percent pick-up is shown in Figure 11. The first and second impregnations for the six cylinders are shown. The slope has decreased for the second impregnation.

Figure 12 shows the regression line for the percent pick-up (2nd impregnation) versus specific gravity after the first carbonization cycle. The correlation coefficient between these two variables was relatively high at $-.633$ and just below the critical value. The relationship is apparent and useful for predicting percent pick-up. However, the specific gravity represents a

combination of AGCarb Liner and Intremold III which have different specific gravities, pore size and pore distribution. A better relationship for specific gravity versus percent pick-up could be established for either material individually.

Figure 13 shows all impregnation cycles for percent pick-up based on weight of part versus specific gravity prior to impregnation. The scatter about the regression lines is much less for the 2nd group of cylinders. Again, this is indicative of the closer control of processing the second group of cylinders.

b. Correlation Matrix #2

The computer program used to determine correlation coefficients did not allow for all variables in one computation. The second set of variables compared is listed in the legend of Figure 14. Figure 14 also contains correlation Matrix #2. The sample size for #2 was 13; therefore, the critical absolute value at the 95% significance level was $\pm .533$.

There was only one negative correlation coefficient which exceeded the $-.533$ value. There was a significant correlation ($-.786$) between temperature of impregnant after impregnation cycle and percent volatiles in impregnant. In other words, the lower temperature of impregnant at cylinder removal time produces a higher volatile content in the impregnant contained in the cylinder pores. It is evident that a relative "hot" pitch would evolve more volatiles upon exposure of the cylinder to the atmosphere. (Note: There was a time lapse between removal of cylinder from impregnant and weighing; therefore, the comparatively hot cylinders will lose more volatiles between

removal and weighing.) The individual data points are plotted in Figure 15 and a linear regression line is estimated for this relationship.

Cylinders removed at the lower temperatures lost more volatiles during cure. The viscosity after impregnation is also a factor in drainage and volatile loss. The optimum temperature for removal was not established; however, a temperature between 220°F and 260°F is desirable. Above 265°F, bubbling or blistering may occur as well as excessive drainage. Below 200°F, the excess pitch is difficult to remove.

Other correlation trends were indicated. The following combinations had relatively high negative correlation coefficients:

- (a) Viscosity of impregnant after impregnation cycle versus percent pick-up of impregnant based on weight of part prior to impregnation cycle.
 $r = -.500$
- (b) Temperature of impregnant after impregnation cycle versus viscosity of impregnant after impregnation cycle. $r = -.434$

The variable listed on the left in the above comparison is likely the causal factor.

A positive correlation coefficient of .627 was obtained for temperature of impregnant after impregnation cycle versus percent carbon yield based on weight of impregnant. This relationship is a corollary to the negative relationship between (temperature of impregnant after impregnation)

versus (Percent Volatiles in impregnant). That is, volatiles were lost if the cylinder was removed relatively hot; therefore, the pick-up weight recorded contained less volatiles and naturally had a higher carbon yield based on impregnant weight.

c. Correlation Matrix #3

The third set of variables analyzed are shown in the legend of Figure 16, Correlation Matrix #3. All of these variables also appeared in Correlation Matrix #2. They are repeated in this analysis with a larger sample size, $n = 24$. The sample size was smaller for Matrix #2 because the after removal process data was not recorded for all cycles. The following combinations had positive correlation coefficients which exceeded the critical value ($\pm .404$) at a 95% significance level:

- (1) Viscosity of impregnant prior to impregnation versus specific gravity of impregnant prior to impregnation cycle. $r = .727$
- (2) Percent carbon yield based on weight of cured impregnant versus percent carbon yield based on weight of impregnant. $r = .907$

The high correlation coefficient for combination (2) indicates that the differential loss of volatiles and pitch from drainage between cylinders was quite small. It is assumed that the approximately 20 percent unexplained variation is a result of differential drainage and/or volatile loss. The individual data points and line of best fit by group for this relation is shown in Figure 17.

The data represents two separate batches of 15V/Indene and many additions of Indene and/or pitch and Indene. In addition the surface area of impregnant exposed in relation to the volume of fresh impregnant is progressively more with each impregnation. There was also more impregnant (percent by weight) actually on the surface of the cylinders for the later impregnations. This was brushed off after cure and does not appear in the carbon yield calculations. The brushing contributes to the carbon yield variability. All data analyzed in Matrix #3 was treated the same regardless of the number of the impregnation cycle. The prior impregnations were treated as having zero effect. It is obvious from the data that the converse is true. Furthermore, less variation in the process variables analyzed in Matrix #3 would be expected for one batch of pitch without additions of Indene.

A significant negative correlation was obtained between percent volatiles in impregnant based on weight loss during cure versus percent carbon yield based on weight of impregnant. An r of $-.530$ was computed. The higher carbon yield was obtained from the impregnant with lower volatile content prior to starting cure. The relationship is shown graphically in Figure 18.

One additional linear regression analysis (not shown in the matrix) was made. Specific gravity of the cylinders from both groups prior to each impregnation was compared to percent pick-up of impregnant based on weight of part prior to impregnation cycle. As expected a significant negative correlation coefficient was obtained. The value was $-.453$, which exceeds

the $\pm .404$ at 95% confidence level, $n = 24$. The variation for percent pick-up for the first batch of cylinders was affected by the wider process variations. Therefore, a much higher correlation coefficient would be expected with less process variation.

2. Multiple Linear Regression

A cursory analysis was made to ascertain if predicting equations could be found using from 2 to 4 independent processing variables. As expected, the viscosity of 15V/Indene mixture is predictable from the specific gravity of mixture and temperature of the mixture.

$$y = -977.967 + 958.627 A - .326 B$$

The regression estimate or predicting equation is as follows:

$$y = b_0 + b_1 A + b_2 B$$

y = predicted viscosity in cps

b_0, b_1 & b_2 = best estimates of constants

A = Specific Gravity

B = Temperature °F

The index of determination for the linear regression of the (viscosity) upon the variables of (specific gravity) and (temperature °F) is 0.697. This is significant at the 1% level. The equation could be refined and improved with the analysis of more data.

The average and standard deviation for viscosity, specific gravity and temperature of the 15V/Indene mixture for all impregnations is shown in Table VII. Values for each impregnation are also tabulated.

Other variables analyzed included (1) y - percent carbon yield as a function of the independent variables A - viscosity, B - specific gravity and C - temperature and (2) y - percent pick-up as a function of A - viscosity, B - specific gravity and C - temperature.

Without further search and calculation neither of the above equations could be used based on the Multiple Linear Regression analysis. It is likely that other relationships exist. A tightening of upper and lower limits for process variables, as well as more uniformity in the samples being impregnated would aid in establishing and verifying these relationships. More rigorous statistical analysis techniques would produce additional relationships for predicting equations. Collecting of additional data should be proceeded by a selection of an experimental design of process variables and cylinder properties to reduce possible interaction effects.

As a part of the linear regression analysis, means, ($\bar{X}$) and standard deviations, (S.D.) for the major process variables and cylinder properties were computed. These are listed in Table XXVI. These values are presented to show the variation for a particular property and/or process.

F. RECOMMENDATIONS

1. The maximum width of fabric which can be used without producing wrinkles should be determined for rosette and shingle lay-ups on a full diameter cone.
2. In conjunction with the first recommendation a full diameter short (6") demonstration cone or cylinder should be fabricated and processed through the first graphitization cycle to verify the scalability of the shrinkage and distortion data contained in this report.
3. The data contained in this report, reference 1 and reference 2 should be correlated with the physical and mechanical properties of the cylinders including the strength of the various joint designs.
4. The AGCarb, transition joints and Intremold III should be cross sectioned, polished and examined at 100X magnification or greater to establish uniformity of the composites.
5. Additional studies of process variations should be conducted to establish optimum impregnation, cure and carbonization cycles.
6. A technique and/or facility should be devised for conducting the evacuation of the fibrous graphite component separate from the impregnant.

TABLE I

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGCarb LINER/INTREMOLD III CYLINDER PIN 2001
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	Wt. of Part W·P	Wt. of Part + Impregnant W·P+I	Wt. of Impregnant W·I	% Impregnant Based on Weight of Part %pI	Wt. of Part + Impregnant Wt. W·P+Cu·I	Wt. of Cured Impregnant W·Cu·I	Wt. of Vols. W·V	% Vols. in Impregnant %iV	Wt. of Part + Carbon W·P+C	Wt. of Carbon W·C	% Carbon Based on Part Wt. %pC	Cum- ulative %pC	% Carbon Based on Impregnant Wt. %iC	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) ¹ After Carb.
First	5000	6,630.0	1630	32.6	6407	1407	223	13.7	5932	932	18.6	18.6	57.2	66.2	1.30
Second	5909	6345	436	7.4	6300	391	45	10.3	6193	284	4.8	23.9	65.1	72.6	1.37
Third	6193	6995	802	13.0	6822	629	173	21.6	6509	316	5.1	30.2	39.4	65.4	1.442
Fourth	6509	7109	600	9.2	6992	483	117	19.5	6728	219	3.4	34.6	36.5	45.3	1.454
Regraphitization									6682			33.6			1.45

LEGEND

Symbols for weight change and percent weight change Table I through VI.

P = Cylinder (Part)
I = Impregnant (Binder)
Cu = Cured
V = Volatiles - Drainage during cure
included in all volatile calculations
C = Carbon - Weight retained after carbon-
ization cycle

W = Weight in grams
%p = Percent based on cylinder weight
%i = Percent based on impregnant weight

¹ Specific Gravity was determined after carbonization and graphitization by water displacement and corrected by subtracting weight of water pick-up from weight of cylinder in water.

TABLE II

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGCarb LINER/INTREMOLD III CYLINDER PIN 2002
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	W·P	W·P+I	W·I	%I	W·P+Cu·I	W·Cu·I	W·V	%IV	W·P+C	W·C	%C	Cum- ulative %C	%IC	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) ¹ After Carb.
First	3846	4790	944	24.5	4708	862	82	8.7	4586	740	19.2	19.2	78.4	85.9	1.312
Second	4545	5028	483	10.6	4966	421	62	12.8	4801	256	5.6	24.8	53.0	60.8	1.351
Third	4801	5070	269	5.6	5000	199	269	74.0	4908	107	2.2	27.6	39.7	53.8	1.375
Fourth	4908	5380	472	9.6	5241	333	139	29.4	5068	160	3.3	31.8	33.9	48.0	1.415
<u>Regraphitization</u>									5030			33.8			1.400

TABLE III

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGCarb LINER/INTREMOLD III CYLINDER PIN 2003
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	W·P	W·P+I	W·I	%I	W·P+Cu·I	W·Cu·I	W·V	%IV	W·P+C	W·C	%C	Cum- ulative %C	%IC	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) ¹ After Carb.
First	3301.0	4500.0	1199	36.3	4343	1042	157	13.1	3948	647	19.6	19.6	54.0	62.1	1.252
Second	3948	4705	757	19.2	4595	647	110	14.5	4296	348	8.8	30.1	46.0	53.8	1.369
Third	4296	4432	136	3.2	4405	109	27	19.9	4383	87	2.0	32.8	64.0	79.8	1.390
Fourth	4383	4885	502	11.5					4538	155	3.5	37.5	30.9		1.442
<u>Regraphitization</u>									4531			37.2			1.415

TABLE IV

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGCarb LINER/INTREMOLD III CYLINDER PIN 2004
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	W•P	W•P+I	W•I	%pI	W•P+Cu•I	W•Cu•I	W•V	%iV	W•P+C	W•C	%pC	Cum- ulative %pC	%iC	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) ¹ After Carb.
First	5101	6930	1829	35.9	6536	1435	394	21.5	6044	943	18.5	18.5	51.6	65.7	1.260
Second	6052	7238	1186	19.6	6883	831	355	29.9	6528	476	7.9	28.0	40.1	57.3	1.351
Third	6528	7511	983	15.1	7117	589	394	40.1	6825	297	4.5	33.8	30.2	50.4	1.401
Fourth	6825	7555	730	10.1	7345	520	209.5	28.6	7050	225	3.3	38.2	30.8	43.3	1.392
<u>Regraphitisation</u>									6937			36.0			1.448

TABLE V

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGCarb LINER/INTREMOLD III CYLINDER PIN 2005
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	W•P	W•P+I	W•I	%pI	W•P+Cu•I	W•Cu•I	W•V	%iV	W•P+C	W•C	%pC	Cum- ulative %pC	%iC	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) ¹ After Carb.
First	4744	6685	1941	40.9	6295	1551	390	20.1	5807	1068	22.4	22.4	54.8	68.5	1.241
Second	5807	7019	1212	20.9	6635	828	384	31.7	6280	473	8.1	32.4	39.0	57.1	1.329
Third	6280	7232	952	15.2	6911	631	321	33.7	6575	295	4.7	38.6	31.0	46.8	1.410
Fourth	6575	7342	767	11.7	7058	483	784	37.0	6745	110	2.6	42.2	22.2	35.2	1.419
<u>Regraphitisation</u>									6652			40.2			1.441

TABLE VI

CHANGES IN WEIGHTS AND SPECIFIC GRAVITY FOR AGCarb LINER/INTREMOLD III CYLINDER PIN 2006
THROUGH FOUR IMPREGNATION, CURE AND CARBONIZATION CYCLES AND A REGRAPHITIZATION CYCLE

Number of Impregnation, Cure and Carbonization Cycle	W.P	W.P+I	W.I	%I	W.P+Cu+I	W.Cu+I	W.V	%IV	W.P+C	W.C	%PC	Cumulative %PC	%IC	%C Based on Impregnant Weight After Cure	Specific Gravity (Corrected) ¹ After Carb.
First	4136	5950	1814	43.9	5452	1316	498	27.5	5042	906	21.9	21.4	49.9	68.8	1.241
Second	5042	6201	1159	23.0	5770	728	431	37.2	5470	428	8.5	32.3	36.9	58.8	1.313
Third	5174	6500	1026	18.7	6055	581	445	43.4	5765	291	5.3	39.4	28.4	56.6	1.360
Fourth	5765	6615	850	14.7	6330	565	285	33.5	5941	176	3.1	43.7	20.7	31.2	1.380
<u>Regraphitization</u>									5875			42.0			1.426

TABLE VII

VISCOSITY, SPECIFIC GRAVITY AND TEMPERATURE OF IMPREGNANT
PRIOR TO IMPREGNATION CYCLES °F

AGCarb/Intremold III Cylinders

<u>Number of Impregnation Cycle</u>	<u>Pin No.</u>	<u>Viscosity CPS</u>	<u>Specific Gravity</u>	<u>Temperature °F</u>
FIRST	2001	48	1.144	225
	2002	41	1.154	265
	2003	48	1.152	250
	2004	52	1.160	250
	2005	41	1.162	260
	2006	56	1.164	250
SECOND	2001	43	1.154	265
	2002	49	1.154	250
	2003	55	1.160	285
	2004	29	1.140	235
	2005	28	1.143	230
	2006	39	1.140	250
THIRD	2001	39	1.138	250
	2002	34	1.144	248
	2003	39	1.142	252
	2004	69	1.156	220
	2005	26	1.140	255
	2006	46	1.156	255
FOURTH	2001	44	1.162	265
	2002	56	1.162	258
	2003	43	1.166	262
	2004	23	1.139	265
	2005	55	1.156	235
	2006	26	1.138	270
	Avg.	42.875	1.151	252.083
	Standard Deviation	--	0.010	14.981

TABLE III

OUTSIDE DIAMETER MEASUREMENTS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2001

Outside Diameter Location		Material	Post Process Step Measurement in Inches					Regraph.
Section	Position		Orig. Cure	First Graph	2nd Imp. Carb.	3rd Imp. Carb.	4th Imp. Carb.	
A	D1	Intremold III Helix	8.792	8.813	8.814	8.815	8.810	8.815
	D2		8.801	8.810			8.809	8.830
	D3		8.805	8.827			8.793	8.837
	D4		8.773	8.795			8.812	8.807
B	D1	FM 5228 Shingle	9.158	9.145	9.141	9.134	9.125	9.142
	D2		9.148	9.130			9.138	9.140
	D3		9.152	9.136			9.136	9.140
	D4		9.156	9.146			9.136	9.160
C	D1	Intremold III Helix	8.847	8.826	8.829	8.820	8.836	8.870
	D2		8.835	8.812			8.855	8.879
	D3		8.821	8.847			8.843	8.857
	D4		8.854	8.843			8.860	8.879
D	D1	Joint II Intremold III + FM 5228 Shingle	9.299	9.224	9.222	9.216	9.214	9.232
	D2		9.224	9.222			9.205	9.225
	D3		9.234	9.240			9.230	9.237
	D4		9.230	9.228			9.215	9.262
E	D1	FM 5228 Shingle	9.155	9.133	9.124	9.120	9.122	9.128
	D2		9.154	9.131			9.119	9.149
	D3		9.155	9.144			9.131	9.118
	D4		9.153	9.123			9.120	9.158

TABLE IX

CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III PIN 2001

			Changes in Outside Diameters Between Process Steps						
Outside Diameter Location		Material	O.D. Graph to 2nd Imp. Carb.	O.D. 2nd to 3rd Imp. Carb.	O.D. 3rd to 4th Imp. Carb.	O.D. 1st Graph to 4th Imp. Carb.	O.D. 4th Imp. Carb. to Regraph.	O.D. 1st Graph to Regraph	O.D. Orig. Cure to Regraph
Section	Position								
A	D1	Intremold III Helix	+.001	+.001	-.005	-.003	+.0035	+.034	+.0353
	D2						+.0321	+.020	+.029
	D3						+.004	+.010	+.032
	D4						-.005	+.012	+.034
B	D1	FM 5228 Shingle	-.004	-.003	-.009	-.020	+.017	-.003	-.016
	D2						+.002	+.010	-.008
	D3						+.004	+.004	-.012
	D4						+.024	+.014	+.004
C	D1	Intremold III Helix	+.003	+.001	+.006	+.010	+.034	+.044	+.023
	D2						+.024	+.067	+.044
	D3						+.014	+.010	+.036
	D4						+.073	+.036	+.025
D	D1	Joint II Intremold III + FM 5228 Shingle	-.002	+.004	-.002	-.010	+.018	+.008	+.003
	D2						+.020	+.003	+.001
	D3						+.007	-.003	+.003
	D4						+.047	+.034	+.032
E	D1	FM 5228 Shingle	-.009	-.004	+.002	-.011	+.006	-.005	-.027
	D2						+.030	+.018	-.005
	D3						-.0313	-.026	-.035
	D4						+.038	+.035	+.005

TABLE X

OUTSIDE DIAMETER MEASUREMENTS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2002

Outside Diameter Location		Material	Post Process Step Measurements in Inches					Regraph.
Section	Position		Orig. Cure	First Graph	2nd Imp. Carb.	3rd Imp. Carb.	4th Imp. Carb.	
A	D1	FM 5228	9.198	9.180	9.146	9.156	9.117	9.153
	D2	Rosette	9.178	9.144			9.129	9.090
	D3		9.210	9.180			9.146	9.755
	D4		9.182	9.145			9.132	9.107
B	D1	JTXVI	8.859	8.927	8.924	8.930	8.933	8.962
	D2	FM 5228	8.920	8.911			8.898	8.896
	D3	Rosette	8.941	8.929			8.911	8.930
	D4	Intremold III Ortho	8.898	8.862			8.885	8.894
C	D1	Intremold III Ortho	8.781	8.785	8.824	8.832	8.769	8.761
	D2		8.848	8.824			8.829	8.819
	D3		8.838	8.892			8.830	8.827
	D4		8.839	8.827			8.823	8.822
D	D1	Intremold III Ortho	8.822	8.817	8.818	8.824	8.806	8.805
	D2		8.843	8.826			8.818	8.820
	D3		8.845	8.835			8.827	8.838
	D4		8.851	8.835			8.833	8.837
E	D1	Intremold III Ortho	8.841	8.842	8.840	8.848	8.8821	8.813
	D2		8.830	8.838			8.814	8.843
	D3		8.838	8.821			8.834	8.826
	D4		8.852	8.842			8.836	8.840
F	D1	JT VII	8.822	8.822	8.821	8.827	8.831	8.797
	D2	FM 5228-Rosette	8.816	8.840			8.819	8.811
	D3	+ Intremold III	8.826	8.810			8.806	8.815
	D4	Ortho	8.839	8.838			8.825	8.844
G	D1	FM 5228	9.153	9.148	9.148	9.154	9.119	9.124
	D2	Rosette	9.193	9.168			9.172	9.177
	D3		9.155	9.127			9.103	9.143
	D4		9.233	9.206			9.181	9.165

TABLE XI

CHANGE IN OUTSIDE DIAMETER OF AGCarb LINER/INTREMOLD III PIN 2002

			Changes in Outside Diameters between Process Steps						
Outside Diameter Location		Material	Δ O.D. Graph. to 2nd Imp. Carb.	Δ O.D. 2nd to 3rd Imp. Carb.	Δ O.D. 3rd to 4th Imp. Carb.	Δ O.D. 1st Graph. to 4th Imp. Carb.	Δ O.D. 4th Imp. Carb. to Regraph.	Δ O.D. 1st Graph. to Regraph.	Δ O.D. Orig. Cure to Regraph.
Section	Position								
A	D1	FM 5228	-.039	+.010	-.039	-.063	+.036	-.027	-.045
	D2	Rosette					-.039	-.054	-.088
	D3						+.077	-.005	-.035
	D4						-.025	-.038	-.075
B	D1	JTXVI	-.003	+.006	+.003	+.006	+.029	+.035	+.103
	D2	FM 5228					-.002	-.015	-.024
	D3	Rosette					+.019	+.001	-.011
	D4	Intremold III Ortho					+ .09	+.034	-.004
C	D1	Intremold III	+.039	+.008	-.063	-.016	-.008	-.024	-.020
	D2	Ortho					-.010	-.005	-.029
	D3						-.003	-.015	-.011
	D4						-.001	-.005	-.017
D	D1	Intremold III	+.001	+.006	-.024	-.017	+.005	-.013	-.017
	D2	Ortho					+.002	-.006	-.023
	D3						+.011	+.003	-.007
	D4						+.004	+.002	-.014
E	D1	Intremold III	-.002	+.008	-.027	-.021	-.008	-.029	-.028
	D2	Ortho					+.029	+.005	+.013
	D3						-.008	+.005	-.012
	D4						+.004	-.002	-.012
F	D1	JT VII	-.001	+.006	+.004	+.009	-.034	-.025	-.025
	D2	FM 5228-Rosette					-.008	-.029	-.005
	D3	+ Intremold III					-.009	+.005	-.011
	D4	Ortho					+.019	+.006	+.005
G	D1	FM 5228	.000	+.006	-.035	-.029	+.055	-.024	-.029
	D2	Rosette					+.005	+.009	-.016
	D3						+.040	+.016	-.012
	D4						-.016	-.041	-.068

TABLE XII

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III
CYLINDER 2001

			Percentage Δ O.D. Between Process Steps						
Section	Outside Diameter Location Position	Material	% Δ O.D. Graph to 2nd Imp. Carb.	% Δ O.D. 2nd to 3rd Imp. Carb.	% Δ O.D. 3rd to 4th Imp. Carb.	% Δ O.D. 1st Graph to 4th Imp. Carb.	% Δ O.D. 4th Imp. Carb. to Regraph	% Δ O.D. 1st Graph to Regraph	% Δ O.D. Orig. Cure to Regraph
A	D1	Intremold III Helix	+.01	+.01	-.06	-.03	+.43	+.36	+.60
	D2						+.24	+.23	+.33
	D3						+.50	+.11	+.36
	D4						-.06	+.14	+.39
							Avg.	+.24	+.42
B	D1	FM 5228 Shingle	-.04	-.08	-.10	-.22	+.19	-.03	-.17
	D2						+.02	+.11	-.09
	D3						+.04	+.04	-.13
	D4						+.26	+.15	+.04
							Avg.	+.13	-.09
C	D1	Intremold III Helix	+.03	+.01	+.07	+.11	+.38	+.50	+.26
	D2						+.27	+.76	+.50
	D3						+.16	+.11	+.41
	D4						+.83	+.41	+.28
							Avg.	+.41	+.36
D	D1	Joint II Intremold III + FM 5228 Shingle	-.02	+.04	-.02	-.11	+.20	+.09	+.03
	D2						+.22	+.03	+.01
	D3						+.08	-.03	+.03
	D4						+.51	+.37	+.35
							Avg.	+.25	+.11
E	D1	FM 5228 Shingle	-.10	-.04	+.02	-.12	+.06	-.05	-.29
	D2						+.33	+.20	-.05
	D3						-.14	-.28	-.40
	D4						+.42	+.38	+.05
							Avg.	+.17	-.20

TABLE XIII

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2002

Outside Diameter Location			Percentage ΔO.D. between Process Steps						
			% ΔO.D. Graph. to 2nd Imp. Carb.	% ΔO.D. 2nd to 3rd Imp. Carb.	% ΔO.D. 3rd to 4th Imp. Carb.	% ΔO.D. 1st Graph. to 4th Imp. Carb.	% ΔO.D. 4th Imp. to Regraph.	% ΔO.D. 1st Graph. to Regraph.	% ΔO.D. Orig. Cure to Regraph.
Section	Position	Material							
A	D1	FM 5228	-.37	+.11	-.43	-.69	+.39	-.29	-.49
	D2	Rosette					+.43	-.59	-.96
	D3						+.30	-.05	-.38
	D4						+.27	-.42	-.82
						Avg.	+.03	-.34	-.66
B	D1	JTXVI	-.03	+.05	-.03	+.07	+.32	+.39	+1.16 ⁽¹⁾
	D2	FM 5228					+.02	-.17	-.27
	D3	Rosette					+.21	+.01	-.12
	D4	Intremold III					+.10	+.36	-.04
		Ortho				Avg.	+.15	-.15	+.21
C	D1	Intremold III	+.44	+.09	-.71	-.18	-.09	-.03	-.23
	D2	Ortho					-.11	-.06	-.33
	D3						-.03	-.17	-.12
	D4						+.01	-.06	-.19
						Avg.	+.00	-.14	-.12
D	D1	Intremold III	+.01	+.07	-.27	-.19	+.00	-.15	-.19
	D2	Ortho					+.02	-.07	-.26
	D3						+.12	+.03	-.08
	D4						+.05	+.02	-.16
						Avg.	+.06	-.04	-.17
E	D1	Intremold III	-.02	+.09	-.31	-.24	-.09	-.33	-.32
	D2	Ortho					+.33	+.06	+.15
	D3						-.09	+.09	-.14
	D4						+.05	-.02	-.14
						Avg.	+.05	-.05	-.03
F	D1	JT VII	-.01	+.07	+.05	+.10	-.39	-.28	-.39
	D2	FM 5228-Rosette					-.04	-.33	-.06
	D3	+ Intremold III					-.10	+.06	-.12
	D4	Ortho					+.22	+.07	+.06
						Avg.	-.09	-.12	-.13
G	D1	FM 5228	.000	+.07	-.38	-.35	+.06	-.26	-.32
	D2	Rosette					+.06	+.10	-.17
	D3						+.44	+.18	-.13
	D4						-.17	-.45	-.74
						Avg.	+.10	-.11	-.34

(1) Questionable number - average not meaningful

TABLE XIV

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2003

			Percentage Δ O.D. Between Process Steps						
Outside Diameter Location			% Δ O.D. Graph. to 2nd Imp. Carb.	% Δ O.D. 2nd to 3rd Imp Carb.	% Δ O.D. 2nd to 4th Imp. Carb.	% Δ O.D. 1st Graph. to 4th Imp. Carb.	% Δ O.D. 4th Imp. to Regraph.	% Δ O.D. 1st Graph. to Regraph.	% Δ O.D. Orig. Cure to Regraph.
Section	Position	Material							
A	D1	FM 5228	-.11	-	-.17	-.28	+.19	-.09	-.31
	D2	Rosette				-.13	-.01	-.14	-.44
	D3					-.04	+.17	+.14	-.09
	D4					-.28	+.31	+.02	-.23
					Avg.	-.21	+.17	-.02	-.27
B	D1	JT-V	-.02	-	-.13	-.66	+.77	+.10	+.61
	D2	Intremold III				+.13	+.41	+.54	+.45
	D3	Helix FM 5228				-.11	+.55	+.43	+.62
	D4	Rosette				-.44	+.72	+.27	+.48
					Avg.	-.27	+.61	+.34	+.54
C	D1	Intremold III	0.00	-	-.11	-.11	+.68	+.57	+.92
	D2	Helix				+.48	+.17	+.65	+1.02
	D3					+.25	+.27	+.52	+.86
	D4					+.01	+.39	+.40	+.63
					Avg.	+.16	+.38	+.54	+.86
D	D1	Intremold III	+.03	-	-.49	-.45	+.90	+.44	+.17
	D2	Helix				+.11	+.78	+.98	+.67
	D3					+.01	+.44	+.45	+.70
	D4					-.15	+1.07	+.92	+.64
					Avg.	-.12	+.80	+.60	+.55
E	D1	Intremold III	-.02	-	-.60	-.62	+.42	-.20	+.12
	D2	Helix				+.01	+.26	+.27	0.00
	D3					-.23	+.24	+.01	+.06
	D4					-.14	+.32	+.18	+.18
					Avg.	-.25	+.31	+.07	+.09
F	D1	JT VIII	-.07	-	-.14	-.21	-.05	-.25	-.57
	D2	Intremold III				+.19	-.36	-.17	-.70
	D3	Helix FM 5228				-.28	-.40	-.68	-.98
	D4	Rosette				+.23	-.29	-.06	-.81
					Avg.	-.02	-.28	-.29	-.77
G	D1	FM 5228	-.03	-	-.18	-.22	+.20	-.01	-.06
	D2	Rosette				+.30	+.16	+.46	+.29
	D3					-.07	-.10	-.17	-.21
	D4					+.21	+.23	+.43	+.21
					Avg.	+.06	+.12	+.18	+.06

TABLE XV

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2004

			Percentage Δ O.D. Between Process Steps						
Outside Diameter Location		Material	% Δ O.D. Graph. to 2nd Imp. Carb.	% Δ O.D. 2nd to 3rd Imp. Carb.	% Δ O.D. 3rd to 4th Imp. Carb.	% Δ O.D. 1st Graph. to 4th Imp. Carb.	% Δ O.D. 4th Imp. to Carb. Regraph.	% Δ O.D. 1st Graph. to Regraph.	% Δ O.D. Orig. Cure to Regraph.
Section	Position								
A	D1	JT XVIII	+.58	-.63	+.09	+.04	-.66	-.62	-.91
	D2	FM 5228 Rosette				+.05	-.45	-.40	-.83
	D3	Intremold III				+.16	-.47	-.32	-.70
	D4	Ortho				-.17	-.58	-.75	-1.09
						Avg. +.02	-.54	-.52	-.88
B	D1	JT XVIII	-.07	+.01	-.21	-.27	-.08	-.36	-.48
	D2	FM 5228 - Rosette				-.11	-.39	-.49	-.62
	D3	Intremold III				+.04	-.27	-.23	-.48
	D4	Ortho				-.23	-.22	-.45	-.66
						Avg. -.14	-.24	-.38	-.56
C	D1	Intremold III	+.11	0.00	-.03	+.08	-.30	-.22	-.28
	D2	Ortho				+.02	+.35	+.37	+.17
	D3					-.07	+.35	+.28	+.15
	D4					+.08	-.21	-.13	-.25
						Avg. +.03	+.05	+.08	-.05
D	D1	Intremold III	+.03	-.20	+.30	+.17	-.13	+.03	-.09
	D2	Ortho				+.07	+.25	+.31	+.19
	D3					+.08	-.02	+.06	-.06
	D4					+.28	-.38	-.10	-.30
						Avg. +.15	-.07	+.08	-.07
E	D1	JT XIX	+.09	-.17	+.12	+.03	-.49	-.45	-.69
	D2	FM 5228-Rosette				-.15	+.11	-.04	-.012
	D3	Intremold III				+.02	+.04	-.02	-.26
	D4	Ortho				+.30	-.33	-.03	-.64
						Avg. +.05	-.19	-.14	-.43
F	D1	FM 5228	+.22	-.45	-.05	-.28	-.56	-.84	-.83
	D2	Rosette				-.24	-.37	-.61	-.87
	D3					+.14	-.75	-.62	-.88
	D4					+.09	-.24	-.14	-.30
						Avg. -.07	-.48	-.55	-.72

TABLE XVI

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2005

			Percentage O.D. Between Process Steps						
Outside Diameter Location		Material	% Δ O.D. Graph to 2nd Imp. Carb.	% Δ O.D. 2nd to 3rd Imp. Carb.	% Δ O.D. 3rd to 4th Imp. Carb.	% Δ O.D. 1st Graph. to 4th Imp. Carb.	% Δ O.D. 4th Imp. to Regraph.	% Δ O.D. 1st Graph. to Regraph.	% Δ O.D. Orig. Cure to Regraph.
Section	Position								
A	D1	FM 5228 Rosette	-.35	+.37	-.18	-.16	+.10	-.06	+.15
	D2					-.09	+.20	+.11	-.12
	D3					-.25	+.45	+.20	+.29
	D4					-.09	+.02	-.07	-.03
						Avg.	-.15	+.19	+.05
B	D1	JT XX-FM 5228 Rosette Intremold III Helix	-.18	+.43	-.24	0.00	-.16	-.16	-.15
	D2					+.06	-.09	-.03	-.27
	D3					-.07	+.16	+.09	-.02
	D4					-.09	-.19	-.29	-.39
						Avg.	-.03	-.07	-.010
C	D1	Intremold III Helix	+.03	-.02	0.00	+.01	+.12	+.13	+.39
	D2					+.09	+.13	+.22	+.04
	D3					+.13	+.48	+.62	+.53
	D4					+.02	+.08	+.10	+.12
						Avg.	+.06	+.20	+.27
D	D1	Intremold III Helix	+.03	+.05	+.11	+.13	+.16	+.30	+.62
	D2					+.32	+.51	+.83	+.49
	D3					+.13	+.27	+.41	+.39
	D4					+.14	+.12	+.26	+.32
						Avg.	+.18	+.27	+.45
E	D1	JT XXIII FM 5228 Rosette Intremold III Helix	-.02	-.01	+.05	+.02	-.05	-.03	+.20
	D2					+.02	-.03	-.01	-.18
	D3					-.11	-.27	+.16	+.31
	D4					+.11	-.21	-.09	-.06
						Avg.	+.01	-.14	+.01
F	D1	FM 5228 Rosette	-.04	-.06	-.02	-.12	+.08	-.04	+.03
	D2					+.01	-.10	-.09	-.41
	D3					-.04	+.06	+.02	-.04
	D4					+.02	-.25	-.23	-.42
						Avg.	-.03	-.05	-.09

TABLE XVI

PERCENT CHANGE IN OUTSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2006

			Percentage Δ O.D. Between Process Steps						
Outside Diameter Location			% Δ O.D. Graph. to 2nd Imp. Carb.	% Δ O.D. 2nd to 3rd Imp. Carb.	% Δ O.D. 3rd to 4th Imp. Carb.	% Δ O.D. 1st Graph. to 4th Imp. Carb.	% Δ O.D. 4th Imp. to Carb. Regraph.	% Δ O.D. 1st Graph. to Regraph.	% Δ O.D. Orig. Curo to Regraph.
Section	Position	Material							
A	D1	JT IM FM 5228	+06	-20	+03	-11	-55	-65	-69
	D2	Rosette				-05	+34	+28	-01
	D3	Intremold III				-11	-21	-32	-55
	D4	Ortho				-07	-24	-32	-48
					Avg.	-09	-17	-25	-43
B	D1	JT IM FM 5228	-15	-12	+18	-08	-50	-59	-46
	D2	Rosette				+02	+14	+16	+03
	D3	Intremold III				-08	-04	-13	-18
	D4	Ortho				-04	-17	-21	-34
					Avg.	-05	-14	-19	-24
C	D1	Intremold III	-04	-26	-31	-61	+45	-16	-21
	D2	Ortho				-03	+61	+57	+33
	D3					-33	+49	+15	+04
	D4					-05	+14	+09	-12
					Avg.	-26	+42	+16	+01
D	D1	Intremold III	-04	-05	-03	-13	-25	-38	-34
	D2	Ortho				-02	+48	+46	+23
	D3					+01	+28	+29	
	D4					-08	+14	+07	-08
					Avg.	-06	+16	+11	-05
E	D1	JT XVI FM 5228	-07	-133	+134	-08	-43	-51	-28
	D2	Rosette				-05	-00	-05	-14
	D3	Intremold III				-11	-01	-12	-11
	D4	Ortho				-08	-05	-13	-16
					Avg.	-08	-12	-20	-17
F	D1	JT XVI FM 5228	-03	-01	-08	-12	-46	-57	-35
	D2	Rosette				-111	+104	-08	-12
	D3	Intremold III				-05	-127	-24	-22
	D4	Ortho				-118	+106	-14	-16
					Avg.	-62	+09	-26	-21

TABLE XVIII

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2001

			Percentage Δ I.D. between Process Steps						
Inside Diameter Location		Material	Δ I.D. Graph. to 2nd Imp. Carb.	Δ I.D. 2nd to 3rd Imp. Carb.	Δ I.D. 2nd to 4th Imp. Carb.	Δ I.D. 1st Graph. to 4th Imp. Carb.	Δ I.D. 4th Imp. to Carb. Regraph.	Δ I.D. 1st Graph. to Carb. Regraph.	Δ I.D. Orig. Cure to Carb. Regraph.
Section	Position								
A	D1	Intremold III	-.29	--	-.13	-.40	+ .51	+ .09	+ .93
	D2	Helix		--			+ .30	- .01	+ .79
	D3			--			+ .57	+ .09	+ .79
						Avg.	+ .46	+ .06	+ .84
B	D1	FM 5228	+ .03	--	-.13	-.10	+ .11	+ .01	+ .36
	D2	Shingle		--			+ .15	+ .03	+ .33
	D3			--			+ .28	+ .05	+ .41
						Avg.	+ .18	+ .03	+ .37
C	D1	Intremold III	-.03	--	-.23	-.25	+ .52	+ .27	+ .55
	D2	Helix		--			+ .50	+ .36	+ .59
	D3			--			+ .50	+ .25	+ .78
						Avg.	+ .51	+ .29	+ .64
D	D1	Joint II	-.06	--	-.28	-.34	+ .65	+ .30	+ .59
	D2	Intremold III		--			+ .65	+ .19	+ .51
	D3	Helix		--			+ .56	+ .20	+ .46
		FM 5228 Shingle				Avg.	+ .61	+ .23	+ .52
E	D1	FM 5228	-.09	--	-.14	-.22	+ .47	+ .24	+ .52
	D2	Shingle		--			+ .36	+ .18	+ .40
	D3			--			+ .34	+ .10	+ .37
						Avg.	+ .39	+ .17	+ .43

TABLE XIX

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2002

			Percentage Δ I.D. between Process Steps						
Inside Diameter Location		Material	Δ I.D. Graph. to 2nd Imp. Carb.	Δ I.D. 2nd to 3rd Imp. Carb.	Δ I.D. 2nd to 4th Imp. Carb.	Δ I.D. 1st Graph. to 4th Imp. Carb.	Δ I.D. 4th Imp. Carb. to Regraph.	Δ I.D. 1st Graph. to Regraph.	Δ I.D. Orig. Cure to Regraph.
Section	Position								
A	D1	FM 5228	-.29	--	+.04	-.25	+.08	-.18	+.15
	D2	Rosette		--		-.27	+.05	-.22	+.05
	D3			--		-.29	+.11	-.18	-.01
					Avg.	-.27	+.08	-.19	+.06
B	D1	JTXVI - FM 5228	-.23	--	-.20	-.43	+.32	-.11	+.18
	D2	Rosette - Intremold		--		-.34	+.15	-.19	+.15
	D3	III Ortho		--		-.44	+.25	-.19	+.24
					Avg.	-.40	+.24	-.16	+.19
C	D1	Intremold III		--	-.05	-.28	+.29	+.01	+.19
	D2	Ortho		--		-.34	+.28	-.06	+.23
	D3			--		-.29	+.24	-.05	-.05
					Avg.	-.30	+.27	-.03	+.12
D	D1	Intremold III	-.19	--	-.10	-.29	+.20	-.09	+.23
	D2	Ortho		--		-.24	+.18	-.09	+.23
	D3			--		-.28	+.20	-.08	+.31
					Avg.	-.27	+.19	-.09	+.26
E	D1	Intremold III	-.16	--	-.20	-.37	+.45	+.08	+.28
	D2	Ortho		--		-.33	+.38	+.05	+.29
	D3			--		-.44	+.47	+.03	+.28
					Avg.	-.38	+.43	+.05	+.28
F	D1	JTVII - FM 5228	-.20	--	-.15	-.36	+.29	-.06	+.14
	D2	Rosette - Intremold		--		-.38	+.29	-.09	+.11
	D3	III Ortho		--		-.41	+.25	-.15	+.15
					Avg.	-.38	+.28	-.10	+.13
G	D1	FM 5228	-.22	--	-.09	-.30	+.15	-.15	+.14
	D2	Rosette		--		-.33	+.18	-.15	+.13
	D3			--		-.36	+.20	-.15	+.18
					Avg.	-.33	+.18	-.15	+.15

TABLE XX

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2003

Inside Diameter Location			Percentage Δ I.D. between Process Steps						
Section	Position	Material	Δ I.D. Graph. to 2nd Imp. Carb.	Δ I.D. 2nd to 3rd Imp. Carb.	Δ I.D. 2nd to 4th Imp. Carb.	Δ I.D. 1st Graph. to 4th Imp. Carb.	Δ I.D. 4th Imp. to Regraph.	Δ I.D. 1st Graph. to Regraph.	Δ I.D. Orig. Cure to Regraph.
A	D1	FM 5228	-.04	--	-.43	-.47	+ .50	+ .03	+ .19
	D2	Rosette		--		-.48	+ .53	+ .05	+ .29
	D3			--		-.44	+ .48	+ .04	+ .28
					Avg.	-.46	+ .50	+ .04	+ .25
B	D1	JTV Intremold	-.01	--	-.28	-.29	+ .31	+ .01	+ .32
	D2	III Helix		--		-.34	+ .38	+ .04	+ .34
	D3	FM 5228 - Rosette		--		-.34	+ .46	+ .11	+ .43
					Avg.	-.32	+ .38	+ .05	+ .36
C	D1	Intremold III	-.03	--	-.39	-.42	+ .38	+ .01	+ .42
	D2	Helix		--		-.28	+ .28	.00	+ .56
	D3			--		-.34	+ .39	+ .05	+ .52
					Avg.	-.35	+ .35	+ .02	+ .50
D	D1	Intremold III	-.01	--	-.23	-.24	+ .04	-.20	+ .34
	D2	Helix		--		-.24	+ .04	-.20	+ .32
	D3			--		-.30	+ .14	-.16	+ .34
					Avg.	-.26	+ .07	-.19	+ .33
E	D1	Intremold III	-.04	--	-.18	-.22	+ .10	-.11	+ .34
	D2	Helix		--		-.25	+ .14	-.11	+ .36
	D3			--		-.34	+ .22	-.14	+ .39
					Avg.	-.27	+ .15	-.12	+ .36
F	D1	JTXVIII Intremold	-.05	--	-.24	-.29	+ .50	+ .20	+ .51
	D2	III Helix - FM 5228		--		-.34	+ .48	+ .14	+ .41
	D3	Rosette		--		-.25	+ .42	+ .16	+ .53
					Avg.	-.29	+ .47	+ .17	+ .50
G	D1	FM 5228	-.06	--	-.15	-.34	+ .32	-.02	+ .41
	D2	Rosette		--		-.39	+ .39	.00	+ .48
	D3			--		-.33	+ .31	-.03	+ .43
					Avg.	-.35	+ .34	-.02	+ .44

TABLE XXI

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2004

			Percentage Δ I.D. between Process Steps						
Inside Diameter Location			Δ I.D. Graph. to 2nd Imp. Carb.	Δ I.D. 2nd to 3rd Imp. Carb.	Δ I.D. 3rd to 4th Imp. Carb.	Δ I.D. 1st Graph. to 4th Imp. Carb.	Δ I.D. 4th Imp. Carb. to Regraph.	Δ I.D. 1st Graph. to Regraph.	Δ I.D. Orig. Cure to Regraph.
Section	Position	Material							
A	D1	JTXVIII - FM 5228	-.09	-.09	+.01	-.17	+.04	-.13	-.14
	D2	Rosette - Intremold				-.09	-.08	-.17	-.14
	D3	III Ortho				-.10	+.09	-.01	-.03
					Avg.	-.12	+.02	-.10	-.10
B	D1	JTXVIII - FM 5228	-.18	+.06	-.14	-.15	+.14	-.01	-.01
	D2	Rosette - Intremold				-.15	+.18	+.03	-.05
	D3	III Ortho				-.19	+.18	+.09	+.03
					Avg.	-.16	+.20	+.04	-.04
C	D1	Intremold III	-.56	-.04	+.05	-.54	+.28	-.27	+.10
	D2	Ortho				-.52	+.43	-.09	+.23
	D3					-.54	+.45	-.10	+.25
					Avg.	-.53	+.39	-.15	+.19
D	D1	Intremold III	-.52	-.03	+.03	-.52	+.31	-.22	+.09
	D2	Ortho				-.49	+.37	-.13	+.18
	D3					-.52	+.43	-.09	+.22
					Avg.	-.51	+.37	-.15	+.16
E	D1	JTXVIII - FM 5228	-.38	-.07	+.03	-.43	+.20	-.23	-.08
	D2	Rosette - Intremold				-.46	+.19	-.27	-.09
	D3	III Ortho				-.43	+.22	-.22	-.06
					Avg.	-.44	+.20	-.24	-.08
F	D1	FM 5228	-.14	+.07	-.10	-.17	+.11	-.05	+.01
	D2	Rosette				-.11	+.09	-.03	+.03
	D3					-.14	+.15	+.01	+.06
					Avg.	-.14	+.12	-.03	+.03

TABLE XXII

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2005

Inside Diameter Location			Percentage Δ I.D. between Process Steps						
			Δ I.D. Graph. to 2nd Imp. Carb.	Δ I.D. 2nd to 4th Imp. Carb.	Δ I.D. 3rd to 4th Imp. Carb.	Δ I.D. 1st Graph. to 4th Imp. Carb.	Δ I.D. 4th Imp. Carb. to Regraph.	Δ I.D. 1st Graph. to Regraph.	Δ I.D. Orig. Cure to Regraph.
Section	Position	Material							
A	D1	FM 5228	-.28	-.05	-.07	-.40	+.28	-.12	+.27
	D2	Rosette				-.37	+.80	+.43	+.65
	D3					-.28	+.33	+.05	+.22
					Avg.	-.35	+.47	+.12	+.38
B	D1	JTXX - FM 5228	-.26	+.21	-.16	-.21	+.20	-.01	+.12
	D2	Rosette - Intremold				-.41	+.68	+.27	+.42
	D3	III Helix				-.41	+.41	0.00	+.13
					Avg.	-.34	+.43	+.09	+.22
C	D1	Intremold III	+.17	-.15	-.07	-.05	+.37	+.32	+.48
	D2	Helix				-.37	+.70	+.33	+.50
	D3					-.34	+.22	-.12	-.02
					Avg.	-.25	+.43	+.18	+.32
D	D1	Intremold III	-.07	-.27	+.17	-.17	+.38	+.21	+.49
	D2	Helix				-.18	+.44	+.26	+.49
	D3					-.21	+.25	+.04	+.28
					Avg.	-.19	+.36	+.17	+.42
E	D1	JTXIII - FM 5228	-.38	+.09	+.06	-.23	+.30	+.06	+.39
	D2	Rosette - Intremold				-.32	+.49	+.17	+.37
	D3	III Helix				-.42	+.23	-.17	+.10
					Avg.	-.32	+.35	+.02	+.29
F	D1	FM 5228	-.42	+.20	-.11	-.33	+.27	-.06	+.25
	D2	Rosette				-.42	+.65	+.23	+.46
	D3					-.31	-.17	-.14	+.07
					Avg.	-.35	+.36	+.01	+.26

TABLE XXIII

PERCENT CHANGE IN INSIDE DIAMETERS OF AGCarb LINER/INTREMOLD III CYLINDER PIN 2006

			Percentage Δ I.D. between Process Steps						
Inside Diameter Location			Δ I.D. Graph. to 2nd Imp. Carb.	Δ I.D. 2nd to 3rd Imp. Carb.	Δ I.D. 3rd to 4th Imp. Carb.	Δ I.D. 1st Graph. to 4th Imp. Carb.	Δ I.D. 4th Imp. Carb. to Regraph	Δ I.D. 1st Graph. to Regraph.	Δ I.D. Orig. Cure to Regraph.
Section	Position	Material							
A	D1	JT IM	-.15	-.08	.00	-.23	+.34	+.11	+.38
	D2	FM 5228 - Shingle				-.11	+.27	+.15	+.38
	D3	Intremold III				-.18	+.22	+.04	+.25
		Ortho				Avg. - .17	+.27	+.10	+.34
B	D1	JT IM	-.34	-.03	-.01	-.38	+.24	-.14	-.01
	D2	FM 5228 - Rosette				-.43	+.30	-.13	+.10
	D3	Intremold III				-.37	+.24	-.13	+.05
		Ortho				Avg. - .39	+.26	-.13	+.05
C	D1	Intremold III	-.32	-.15	+.05	-.42	+.33	-.09	+.09
	D2	Ortho				-.25	+.22	-.04	+.08
	D3					-.42	+.29	-.13	-.04
						Avg. - .36	+.28	-.09	+.03
D	D1	Intremold III	-.44	-.04	+.11	-.37	+.33	-.04	+.09
	D2	Ortho				-.40	+.37	-.04	+.03
	D3					-.44	+.37	-.08	+.03
						Avg. - .40	+.36	-.05	+.05
E	D1	JTXVI - FM 5228	--	--	--	--	--	-.13	+.13
	D2	Rosette				--	--	-.19	-.06
	D3	Intremold III				--	--	-.15	+.05
		Ortho						Avg. - .16	+.04
F	D1	JTXVI - FM 5228	-.20	-.09	.00	-.29	+.32	+.03	+.38
	D2	Rosette				-.29	+.27	-.03	+.36
	D3	Intremold III				-.22	+.11	-.10	+.34
		Ortho				Avg. - .27	+.23	-.03	+.36

TABLE XXIV

CHANGE IN LENGTH OF AGCarb LINER/INTREMOLD III CYLINDERS 2001, 2002, 2003, 2004, 2005 & 2006

Cylinder Number	Material	Position of Length Measurement	Post Process Step Length Measurements in Inches				Percent Change in Length Between Process Steps			
			Orig. Cure	First Graph.	4th Imp. Carb.	Regraph.	% Δ L 1st Graph to 4th Imp. Carb.	% Δ L 4th Imp. Carb. to Regraph.	% Δ L Graph to Regraph.	% Δ L Orig. to Regraph.
2001	FM 5228 Shingle/ Intremold III Helix	0°	17.940	17.891	17.852	17.883	-.22	+.17	-.05	-.31
		90°	17.942	17.900	17.864	17.900	-.20	+.20	.00	-.23
		180°	17.955	17.890	17.871	17.890	-.11	+.11	.00	-.36
		270°	17.945	17.900	17.870	17.897	-.17	+.15	-.02	-.27
							Avg. -.18	+.16	-.02	-.29
2002	FM 5228 Rosette/ Intremold III Orthogonal	0°	16.031	15.997	15.964	15.982	-.21	+.11	-.09	-.31
		90°	16.031	16.012	15.983	15.998	-.18	+.09	-.09	-.21
		180°	16.000	16.019	16.008	16.002	-.07	-.04	-.11	+.01
		270°	16.039	16.033	15.998	16.002	-.22	+.03	-.19	-.23
							Avg. -.17	+.05	-.12	-.18
2003	FM 5228 Rosette/ Intremold III Helix	0°	16.569	-	16.590	16.630	-	+.24	-	+.37
		90°	16.649	-	16.619	16.564	-	-.33	-	-.51
		180°	16.646	-	16.569	16.612	-	+.26	-	-.20
		270°	16.614	-	16.546	16.640	-	+.57	-	+.16
							Avg. -.19	+.19	-	-.05
2004	FM 5228 Rosette/ Intremold III Orthogonal	0°	15.979	15.985	15.962	15.970	-.14	+.05	-.09	-.06
		90°	16.012	16.032	15.980	15.981	-.32	+.01	-.32	-.19
		180°	15.996	15.985	16.000	15.977	-.09	-.14	-.05	-.12
		270°	15.968	15.982	15.964	15.982	-.11	+.11	0.00	+.09
							Avg. -.17	+.01	-.12	-.07
2005	FM 5228 Rosette/ Intremold III Helix	0°	17.416	17.298	17.293	17.253	-.03	-.23	-.26	-.94
		90°	17.403	17.285	17.260	17.234	-.14	-.15	-.30	-.97
		180°	17.391	17.285	17.261	17.240	-.14	-.12	-.26	-.92
		270°	17.394	17.284	17.263	17.248	-.12	-.09	-.21	-.84
							Avg. -.11	-.15	-.26	-.92
2006	FM 5228 Rosette and Shingle/ Intremold III Orthogonal	0°	14.585	14.693	14.556	14.585	0.32	+.20	-.12	-.00
		90°	14.580	14.580	14.544	14.559	-.25	+.10	-.14	-.14
		180°	14.638	14.610	14.605	14.627	-.03	+.15	+.11	-.08
		270°	14.593	14.513	14.517	14.517	+.03	.00	+.03	-.52
							Avg. -.14	+.11	-.03	-.19

TABLE XXV

RESULTS OF ISOPROPYL ALCOHOL PERMEABILITY⁽¹⁾ AND PENETRANT TESTS ON AGCarb LINER/INTREMOLD III CYLINDERS
PIN 2001, 2002, 2003, 2004, 2005 AND 2006

Cylinder Number	Material	Circumferential Location of Alcohol Test	Axial Location of Alcohol Test	Time to Penetrate Wall I.D. to O.D. After 4th Imp. Carb., seconds	Time to Penetrate Wall I.D. to O.D. After Regraph., seconds	Alcohol Penetrant ⁽²⁾ Results After Regraph.
2001	Intremold III Helix	0°	Center of	--	11.5	Numerous intermittent delams. in O.D. roving. No other alcohol indications. Intremold sections porous.
		90°	Intremold III	--	7.4	
		180°	Area	--	10.5	
		270°	C Section	--	10.6	
	FM 5228 Shingle	0°	A Section	--	No	
		180°		--	No	
	Roving Area	0°	D Section	--	No	
		180°		--	No	
2002	Intremold III Orthogonal	0°	D Section	--	9.0	No indications of delaminations or separations; however, Intremold section was porous.
		90°		--	8.5	
		180°		--	8.3	
		270°		--	10.0	
	FM 5228 Rosette	0°	A-B Section	--	No	
		180°		--	No	
	Intremold III Helix	0°	D-E Section	--	22.0	No indications of delaminations or separations; however, variable porosity in Intremold area.
		90°		--	15.5	
		180°		--	18.1	
		270°		--	8.9	
2003	Intremold III Orthogonal	0°	C-D Section	No	9.0	No indications of delaminations; however, Intremold sections were porous.
		90°		No	12.9	
		180°		No	14.6	
		270°		No	13.2	
	FM 5228 Rosette	0°	Both A & F Sections	No	No	
		90°		No	No	
		180°		No	No	
		270°		No	No	

(1) The alcohol permeability test was accomplished by pouring 10 cc of isopropyl alcohol on the selected test area and visually checking the time to penetrate through the wall. If no penetration occurred, a (no) was recorded in the time column, otherwise the time for the first sign of wetness on the O.D. was recorded as the time to penetrate.

(2) Alcohol penetrant test for delaminations and/or separations was made in accordance with AGC Specification 36491.

TABLE XXV (cont.)

RESULTS OF ISOPROPYL ALCOHOL PERMEABILITY⁽¹⁾ AND PENETRANT TESTS ON AGCarb LINER/INTREMOLD III CYLINDERS
2001, 2002, 2003, 2004, 2005 AND 2006

<u>Cylinder Number</u>	<u>Material</u>	<u>Circumferential Location of Alcohol Test</u>	<u>Axial Location of Alcohol Test</u>	<u>Time to Penetrate Wall I.D. to O.D. After 4th Imp. Carb., seconds</u>	<u>Time to Penetrate Wall I.D. to O.D. After Regraph., seconds</u>	<u>Alcohol Penetrant (2) Results After Regraph.</u>
2005	Intremold III Helix	0°	C-D Section	No	8.0	No indications of delaminations or separations; however, Intremold sections were porous.
		90°		No	7.9	
		180°		No	6.1	
		270°		No	5.0	
	FM 5228 Rosette	0°	A Section	No	No	
		90°		No	No	
		180°		No	No	
		270°		No	No	
2006	Intremold III Orthogonal	0°	C-D Section	No	15.1	No specific indications of delaminations observed after final graphitization; however, porosity was observed in AGCarb rosette at 270°. <u>Note:</u> The AGCarb IM shingle showed delaminations for almost 360°, .050 to .130 from I.D. after original graphitization.
		90°		No	18.1	
		180°		No	30.0	
		270°		No	25.0	
	FM 5228 Shingle	0°	A Section	No	No	
		90°		No	No	
		180°		No	No	
		270°		No	No	

(1) The alcohol permeability test was accomplished by pouring 10 cc of isopropyl alcohol on the selected test area and visually checking the time to penetrate through the wall. If no penetration occurred, a (no) was recorded in the time column, otherwise the time for the first sign of wetness on the I.D. was recorded as the time to penetrate.

(2) Alcohol penetrant test for delaminations and/or separations was made in accordance with AGC Specification 36491.

TABLE XXVI

MEANS AND STANDARD DEVIATIONS FOR MAJOR
PROCESS PARAMETERS AND CYLINDER PROPERTIES

1. Cylinder Starting Specific Gravity

Sample size $n = 6$

Mean, $\bar{x} = 1.023$

Standard Deviation S.D. = .0408

2. Cylinder Final Specific Gravity

Sample size $n = 6$

Mean, $\bar{x} = 1.431$

Standard Deviation S.D. = .0215

3. Percent Weight Pick up First Impregnation Based on
Starting Cylinder Weight

Sample size $n = 6$

Mean, $\bar{x} = 35.683$

Standard Deviation S.D. = 6.778

4. Cylinder Specific Gravity after First Impregnation
Carbonization

Sample size $n = 6$

Mean, $\bar{x} = 1.267$

Standard Deviation S.D. = .030

5. Cylinder Specific Gravity after Second Impregnation
Carbonization

Sample size $n = 6$

Mean, $\bar{x} = 1.347$

Standard Deviation S.D. = .022

6. Cylinder Specific Gravity after Third Impregnation Carbonization

Sample size $n = 6$

Mean, $\bar{x} = 1.396$

Standard Deviation S.D. .029

TABLE XXVI

7. Cylinder Specific Gravity after Fourth Impregnation Carbonization
Sample size $n = 6$
Mean, $\bar{x} = 1417$
Standard Deviation S.D. = .028
8. Percent Weight Pick up Second Impregnation Based on Cylinder Weight
Sample size $n = 6$
Mean $\bar{x} = 16.78$
Standard Deviation S.D. = 6.256
9. Percent Weight Pick up Third Impregnation Based on Cylinder Weight
Sample size $n = 6$
Mean $\bar{x} = 12.0$
10. Percent Weight Pick up Fourth Impregnation Based on Cylinder Weight
Sample size $n = 6$
Mean $\bar{x} = 11.133$
Standard Deviation S.D. = 2.016
11. 15V /Indene - Viscosity After Impregnation
Sample size $n = 13$
Mean $\bar{x} = 105.538$
12. 15V/Indene - Specific Gravity After Impregnation
Sample size $n = 13$
Mean $\bar{x} = 1.165$
Standard Deviation S.D. = .015

TABLE XXVI (cont)

13. 15V/Indene - Temperature After Impregnation

Sample size $n = 13$
Mean $\bar{x} = 251.385$

14. 15V/Indene - Temperature Before Impregnation (all impregnations)

Sample size $n = 24$
Mean $\bar{x} = 252.083$
Standard Deviation S.D. = 14.981

15. 15V/Indene Percent Volatiles Based on Weight Loss During Cure (all impregnations)

Sample size $n = 24$
Mean $\bar{x} = 25.833$
Standard Deviation S.D. = 9.994

16. 15V/Indene Percent Carbon Yield Based on Cured Impregnation Weights

Sample size $n = 24$
Mean $\bar{x} = 57.358$
Standard Deviation S.D. = 13.245

17. 15V/Indene Percent Pick up Based on Cylinder Weights (all impregnations)

Sample size $n = 24$
Mean $\bar{x} = 27.5$

18. 15V/Indene Percent Carbon Yield Based on Impregnation Weights (all impregnations)

Sample size $n = 24$
Mean $\bar{x} = 42.479$

Figure 1. AGCarb Liner/Intremold III Cylinder, Fabrication Sequence

Figure 2. Effect of Temperature on Viscosity of 15V/Indene

Figure 3. Specific Gravity of Cylinders Number 2001 Through 2006

Figure 4. 15V/Indene Impregnant Pick-up Versus Impregnation Cycle

Figure 5. Effect of Process Control on Impregnant Retention and Carbon Yield in Cylinders 2001 through 2006

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Figure 6. Average Percent Δ O.D. from Graphitization to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006

Figure 7. Average Percent Δ O.D. from Original Cure to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006.

Figure 8. Average Percent Δ I.D. from Graphitization to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006

Figure 9. Average Percent Δ I.D. from Original Cure to Regraphitization for Various Locations A through G in Cylinders 2001 through 2006

	D	E	F	H	T	W	K	Z	R	V
D	-	-.015	-.831	-.863	-.828	.401	.559	-.879	-.531	-.810
E		-	.372	-.211	.043	.762	.133	-.049	.639	-.177
F			-	-.940	-.627	-.136	-.391			
H				-			.393			
T					-			-.633		
W						-			.060	-.576
K							-			
Z								-		
R									-	
V										-

1. The critical absolute values of correlation coefficient r at a 95% confidence level are $\pm .811$

2. LEGEND

D Starting specific gravity of cylinder
 E Final specific gravity of cylinder
 F Percent weight pick up first impregnation based on cylinder weight
 H First carbonization specific gravity
 T Second carbonization specific gravity
 W Third carbonization specific gravity
 K Fourth carbonization specific gravity
 Z Percent weight pick up second impregnation based on cylinder weight
 R Percent weight pick up third impregnation based on cylinder weight
 V Percent weight pick up fourth impregnation based on cylinder weight.

Figure 10. Correlation Matrix #1 for Specific Gravity and Percent Weight Pick-up for AGCarb Liner/Intremold III Cylinders $N = 6$

Figure 11. Impregnant Pick-up Percent versus Initial Graphitization Specific Gravity

Figure 12. Impregnant Pick-up First Impregnation versus Specific Gravity After First Carbonization.

Figure 13. Specific Gravity versus Impregnant Pick-up

	D	E	F	H	T	W	K	Z	R	V
D	-	(1)	(1)	-.261			-.058		.134	
E		-	(1)		.115		-.028		-.070	-.070
F			-						-.210	
H				-		-.434	.049	.238	-.500	-.053
T					-	-.275	-.015		-.376	-.043
W						-	-.786		.390	.627
K							-			
Z								-		
R									-	
V										-

(1) See Correlation Matrix #3 Figure
A larger sample size was used for #3.

1. The critical absolute values of correlation coefficient r at a 95% confidence level are ± 0.553 .

2. LEGEND

D Viscosity of impregnant prior to impregnation cycle
E Specific gravity of impregnant prior to impregnation cycle
F Temperature of impregnant prior to impregnation cycle
H Viscosity of impregnant after impregnation cycle
T Specific gravity of impregnant after impregnation cycle
W Temperature of impregnant after impregnation cycle
K Percent volatiles in impregnant-based on weight loss during cure
Z Percent carbon yield based on weight of cured impregnant
R Percent pick-up of impregnant based on weight of part prior to impregnation cycle
V Percent carbon yield based on weight of impregnant

Figure 14. Correlation Matrix #2 for Process Variations of 15V/Indene Mixture - N = 13

Figure 15. Relation of 15V/Indene Impregnant Percent Volatiles to Temperature at the End of Impregnation Cycle.

	D	E	F	H	T	W	K			
D	-	.727	-.198	-.110	.121	-.012	.137			
E		-	.272	-.234	.045	-.152	.150			
F			-			-.065	.108			
H				-	-.530	.166	-.774			
T					-	.112	.907			
W						-	.015			
K							-			

1. The critical absolute values of correlation coefficient r at a 95% confidence level are $\pm .404$

2. LEGEND

- D Viscosity of impregnant prior to impregnation cycle
- E Specific gravity of impregnant prior to impregnation cycle
- F Temperature of impregnant prior to impregnation cycle
- H Percent volatiles in impregnant based on weight loss during cure
- T Percent carbon yield based on weight of cured impregnant
- W Percent pick-up of impregnant based on weight of part prior to impregnation cycle
- K Percent carbon yield based on weight of impregnant.

Figure 16. Correlation Matrix #3 for Process Variations of 15-V/Indene Mixture - $N = 24$

Figure 17. Carbon Yield Percent of Cured Versus Uncured 15V/Indene Impregnant.

Figure 18. Percent Volatiles Versus Percent Carbon Yield of 15V/Indene Impregnant.

APPENDIX I

INSTRUCTIONS OF SURVEILLANCE PERSONNEL DURING CARBONIZATION AGCarb LINER/INTREMOLD III CYLINDERS

(NOTIFY COGNIZANT ENGINEER IF ANY PROBLEMS OCCUR, DAY OR NIGHT)

1. Check temperature and argon flow every hour and initial recorder chart, and record time at each check.
2. Maintain argon flow at approximately 10 CFH to 20 CFH at all times; however, the flow rate should be high enough to bubble both the acid and sodium hydroxide solution. Check system for leaks if a flow rate of 30 CFH or higher is required.
3. If outlet flow system becomes stopped, change immediately to the second #2 (small outlet and unstop main (1-1/2" tubing) outlet flow. (Record temp. at which stoppage occurred).
4. Do not allow any of the tubing to droop. Liquid condensate will fill up the tube and stop flow through the system.
5. IMPORTANT. Change cams as needed on controller. This will be every 24 hours. Note: Turn off cam drive switch before changing cams. Be sure cam drive switch is back on after cam is changed.
6. Check drive arm on controller with T/C readout on circular chart. Variation should never be greater than 50°F.
7. Record temperature readout from circular chart on the recorder chart every hour.
8. Record argon bottle pressure every hour on recorder chart.
9. Change bottles of argon when pressure on gage drops below 200 psi. (The time between changes should be less than 60 seconds.) Switch to a new bottle in the twelve pack. Identify bottle being used and the bottle which has been depleted.

APPENDIX I (cont.)

10. As the outgassing starts, the bottles will show gray to green gas being evolved. Record by your initials an (O) if there is outgassing and (N) if there is no visual evidence of outgassing.
11. Use a rod to unstop the outlet line in case of a stop-up. (Maintain argon flow, although line may be stopped.)
12. Change temperature limiting control to read 100°F above maximum on cam at the time each cam is changed. Temperature limiting control is on strip chart recorder dial right hand top.
13. After outgassing has started, turn on tubing heater blankets. Use variac controller to control temperature at the temperature reading on the strip chart. There are two T/C's on the outlet tubing. Record the temperatures of the tube T/C's on the strip chart every hour.

NOTE: Do not exceed 1200°F on tapes.

14. There will be condensate in the tygon tubing. If the tube becomes stopped, remove and replace tubing. Save the tube with condensate for analysis.