PROCESSING SCALE-UP STUDIES ON CARBON-CARBON COMPOSITES

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Introduction

Larger carbon/carbon components will be required for future applications. Procedures are being developed for processing carbon/carbon composites up to (20 cm x 20 cm x 30 cm) which exhibit the same favorable performance characteristics as has been obtained with smaller composites (10 cm x 10 cm x 20 cm). The scale-up processes being studied on this program include an initial chemical vapor deposition of carbon within a woven carbon preform and the subsequent impregnation, carbonization and graphitization of this composite. Mechanical and thermal test results are being obtained.

Chemical Vapor Deposition (CVD)

The woven graphite preforms are rigidized using a CVD infiltration procedure which consists of a heat-treatment to clean the carbon fiber bundles followed by a CVD infiltration which coats the fibers and rigidizes the preform. For 10 cm x 10 cm x 20 cm preforms, the infiltration time required is 80 hours. Process parameters were developed to infiltrate the 20 cm x 20 cm x 30 cm preforms to achieve similar density gradients and fiber coating thicknesses in these larger preforms as was previously achieved in 10 cm x 10 cm cross-section preforms. The major changes to the process included a slightly lower deposition pressure, an increased deposition time, and a redesign of the preform support. Figure 1 shows the typical coating thicknesses on T-50 fibers, and Figure 2 shows the coating thickness on individual fibers as a function of location within the preform.

Densification

The densification process consists of an impregnation/encapsulation of the CVD infiltrated preform, followed by a high pressure (100 MPa) carbonization at 650°C, and then a graphitization at 2750°C. These steps are repeated for five cycles. The density increase in these five cycles is typically 0.98-1.03 gms/cm^3 (from 0.86 to 1.88 gm/cm^3). The major change which had to be made to the standard process was a "pre-impregnation" step which is required for the first impregnation. This process consists of applying a pressure up to 3 MPa on the liquid pitch (~ 230°C) during the encapsulation operation. This added step is to insure the pitch has penetrated into the core of the preform before the pitch is allowed to solidify. Figure 3 compares the impregnation process used for the larger billets to the process used for 10 cm x 10 cm cross-section billets.

In addition to scaling up the process for larger carbon/carbon composites, a secondary objective is to establish if the standard process is acceptable for an alternate weave construction. The standard size preforms and larger preforms are all orthogonal weaves. The alternate construction is fabricated from stacks of woven graphite cloth which are pierced on 1.3 mm centers perpendicular to the cloth. Carbon fibers are then inserted into these sites. It was found that the standard process can be used to densify the alternate weave construction preforms without any changes. These types of preforms were densified to bulk densities of ~ 1.95 gm/cm^3. Figure 4 illustrates the densification behavior of the different types and sizes of billets processed on this program.

Characterization

The characterization of these carbon/carbon composites consists of detailed NDE at three stages, namely (1) of the as-received preform, (2) after the CVD infiltration, and (3) after final densification. The NDE consists of radiography, radiometric density, ultrasonic velocity measurements, visual inspection, layer spacing counts and fiber deficiency maps.

Many of the composites have also been destructively tested to determine mechanical/thermal properties and physical properties as a function of location within the billet. Table 1 lists typical properties for these carbon/carbon composites.

Conclusions

Process conditions have been established for fabricating carbon/carbon composites in sizes up to 20 cm x 20 cm x 30 cm. The larger sizes require additional CVD infiltration time and modified conditions to achieve similar density gradients and fiber coating thicknesses as was achieved with smaller billets. The densification process appears to be insensitive to sizes up to 20 cm x 20 cm x 30 cm. The only modification required was to apply a pressure of 3 MPa on the pitch before allowing it to cool during the initial pitch encapsulation process.

Acknowledgments

This work was sponsored by the Manufacturing Technology Division of the Air Force Materials Laboratory. Mr. Harry P. Materne was AFML Project Engineer. The author is Program Manager and Principal Investigator. The suggestions and assistance of Dr. E. Stover, Dr. J. Gebhardt, Mr. W. Mueller, Mr. J. Roetling and Mr. J. D'Andrea of the General Electric Co; Dr. D. VanWagoner of Southern Research Institute, and Dr. Gary Weber of Union Carbide Corp. (Nuclear Division), is gratefully acknowledged.

TABLE 1

TYPICAL PROPERTIES OF 223 CARBON/CARBONS

Density, gm/cm^3	1.880
Open Porosity, % (air)	6.5
Tensile Strength, ksi (RT)	
"X" Direction	20.
"Z" Direction	44.
Tensile Modulus, 10^6 psi (RT)	
"X" Direction	8.
"Z" Direction	14.
Strain, % (RT)	
"X" Direction	.25
"Z" Direction	.32
Compressive Strength, ksi (RT)	
"X" Direction	16.
"Z" Direction	22.
Thermal Expansion ($\Delta L/L \times 10^3$)	
"X" Direction: RT to 4000°F	3.43
"Z" Direction: RT to 4000°F	3.19

FIG. 1. Typical CVD Coatings on T50 Fibers. Left--Fiber at ~ 0.6 cm. from outer surface. Right--Fiber Taken at ~ 3 cm. from Outer Surface. (Original SEM @ 10,000X)

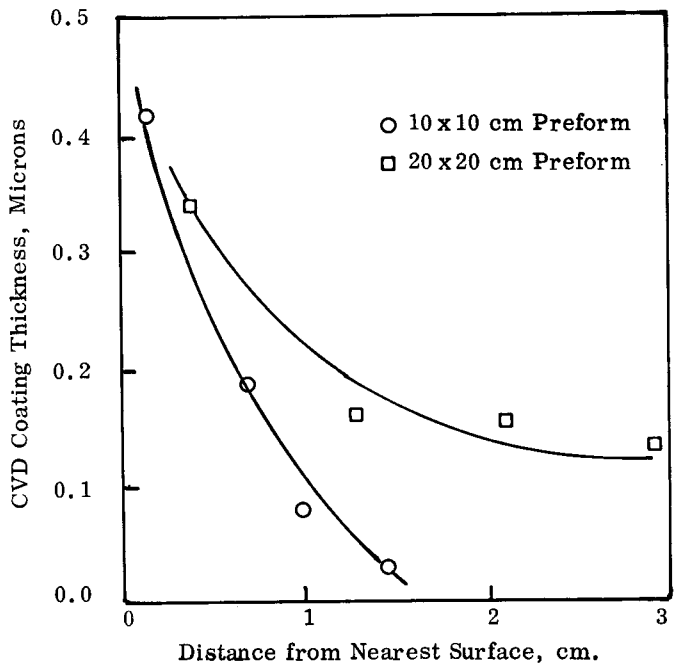

FIG. 2. Coating Thickness of Fibers at Various Depths in 10x10 cm. and 20x20 cm. Preforms. (From SEM Measurements)

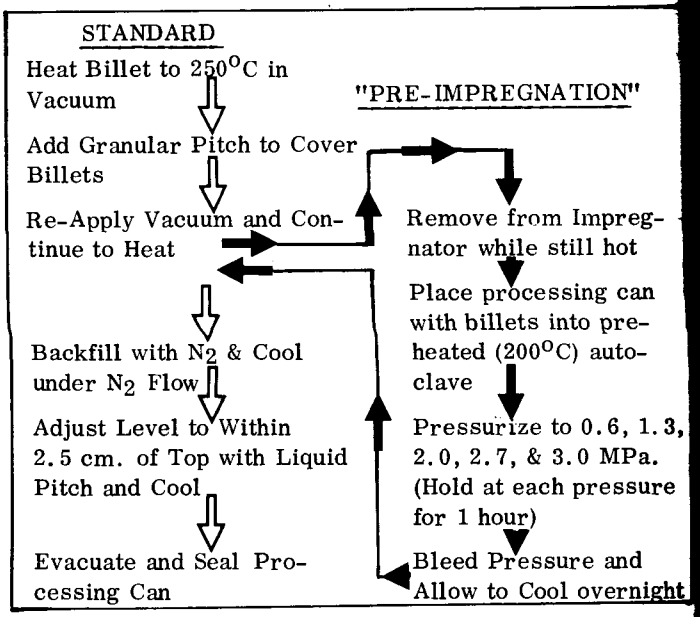

FIG. 3. Procedure for Impregnation of Preforms. Left side--Conventional Procedure. Right side--Additional Steps used in "Pre-Impregnation" Procedure of Larger Preforms.

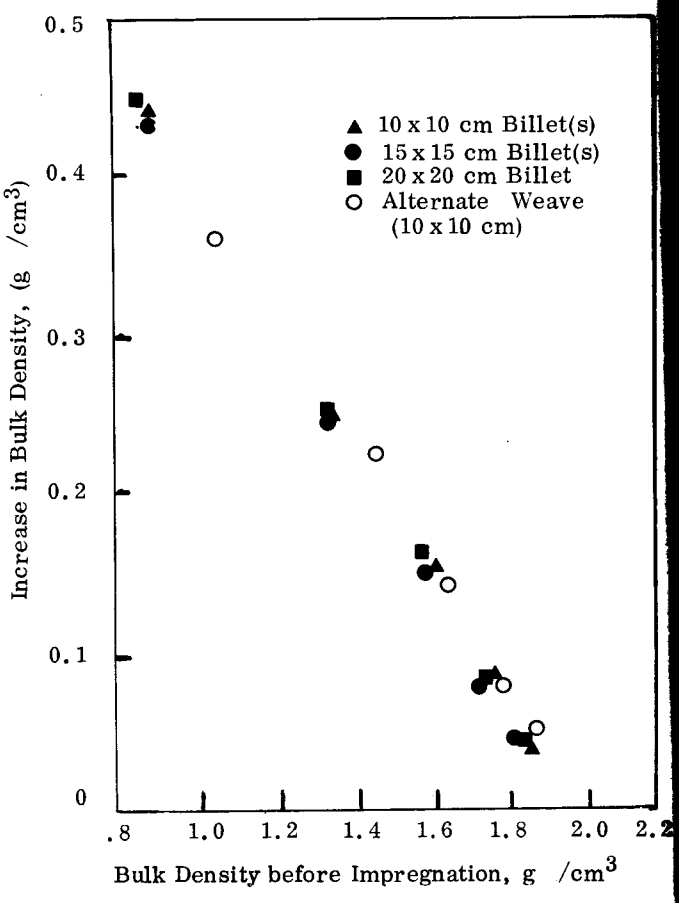

FIG. 4. Densification of 3 sizes of an Orthogonal Preform and One Alternate Weave Construction Preform. There is Little Difference between the Different Sizes. The Alternate Type was not Infiltrated Prior to Densification.