Project #: E-18-675
Center #: 10/24-6-R8150-0A0

Contract #: 0099-DG09-01
Prime #: F33615-94-C-4437

Subprojects?: N
Main project #: •

Project unit: MSE
Project director(s): STARR T L EDEML

Sponsor/division names: MATERIALS SCIENCES CORP
Sponsor/division codes: 212

Award period: 940601 to 941031 (performance) 941031 (reports)

Sponsor amount
Contract value 15,080.00
Funded 15,080.00

Cost sharing amount
New this change 0.00
Total to date 15,080.00

Does subcontracting plan apply?: N

Title: REVIEW OF GAS PERMEABILITY ESTIMATION METHODS

PROJECT ADMINISTRATION DATA

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Security class (U,C,S,TS) : U
Defense priority rating : N/A
Equipment title vests with: Sponsor
NONE PROPOSED OR ANTICIPATED.

Administrative comments -
INITIATION OF PROJECT E-18-675.

ONR resident rep. is ACO (Y/N): N
N/A supplemental sheet
GIT
GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 12/01/94

Project No. E-18-675

Center No. 10/24-6-R8150-0A0

Project Director STARR T L

School/Lab MSE

Sponsor MATERIALS SCIENCES CORP/FT. WASHINGTON, PA

Contract/Grant No. 0099-DG09-01

Contract Entity GTRC

Prime Contract No. F33615-94-C-4437

Title REVIEW OF GAS PERMEABILITY ESTIMATION METHODS

Effective Completion Date 941031 (Performance) 941031 (Reports)

Closeout Actions Required:  

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Comments

Subproject Under Main Project No.

Continues Project No.

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NOTE: Final Patent Questionnaire sent to PDPI.
REVIEW OF GAS PERMEABILITY ESTIMATION METHODS

Phase I Final Report

November 1994

Prepare by

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Prepared for

Materials Science Corporation
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Under a Phase I SBIR for

United States Air Force
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INTRODUCTION

For forced flow chemical vapor infiltration (CVI) the gas permeability of the preform and the partially dense composite is a critical factor in the success of the process. The gas permeability of the material controls the pattern of reactant flow through the preform and sets the limit for the ultimate achievable density.

The process simulation code, GTCVI, was developed as a general purpose model of the CVI process and can be used for process design and optimization. Accurate simulation of the densification process for a specific preform requires good estimates of the transport properties. Experimental measurements of the gas permeability of square-weave cloth layup preforms was a key factor in the successful validation of the GTCVI code for this preform.

In order to allow more general use of GTCVI it is desirable to add transport property estimation routines that are based on the fiber architecture and the constitutive properties of fiber and matrix materials. In addition to broadening the effective use of GTCVI for different preforms, this modification will allow use of the model for selecting and designing fiber architectures by linking changes in such factors as fiber spacing and filament count to densification performance. The Phase I effort toward developing a more general gas permeability model consisted of a review of previously reported estimation methods. Implementation of such a model will be performed in Phase II.
REVIEW OF TRANSPORT MODEL ESTIMATION METHODS

Previous efforts to estimate gas transport properties and their evolution during CVI densification have been limited. It is important to note that estimation of the initial properties of the preform is not sufficient for our application. The transport properties of the material change as density increases and a suitable model must track this change. In particular, the limiting behavior near full density is very important as this determines to a great extent the final microstructure of the composite.

Early work by Naslain and co-workers\textsuperscript{2,3} modeled the preform as a collection of cylindrical, monosized pores and estimated the effective diffusion coefficient for the material as the pores filled during CVI. Convective flow of gas was not considered although this simple microstructure would allow a relatively straightforward extension of the model to include gas permeability. This simple microstructure, however, is not a good representation of real woven preforms and cannot provide quantitative estimates of transport properties.

For randomly oriented, short fiber preforms Starr\textsuperscript{4} proposed a structure model based on an orthogonal array of cylinders and used this model to estimate surface area and gas permeability during CVI. This microstructure model is more realistic in that it includes a distribution of pore sizes. Developed for chopped fiber preforms the model does not apply to anisotropic woven materials.

Yu and Sotirchos\textsuperscript{5} proposed a generalized pore model for gas-solid reactions based on an interconnected network of cylindrical
pores with a distribution of diameters. Percolation theory is used to predict creation of trapped porosity as density increases and the smaller pores are filled. This feature is an important advance since residual, inaccessible porosity is always present in "fully" infiltrated CVI processed composites. This approach has been tested using Monte Carlo simulation$^6$ and applied to CVI processing$^7,8,9,10,11$. While this approach has many desirable features, the structure model employed does not relate well to the anisotropic, woven fiber structures commonly used for CVI preforms.

For cloth layup preforms Starr$^{12}$ used a semi-empirical approach relating the anisotropic gas permeability and diffusion coefficient to the contrasting properties of the cloth-containing regions and the regions between cloth layers. While introducing anisotropy as a model feature, this approach does not relate properties directly to microstructure but relies on experimental measurements to provide model parameters.

Also for woven cloth preforms, Chung et al.$^{13,14}$ employed a model that explicitly includes such structural features as tow and cloth spacing and the number and size of filaments in the tows. This model includes diffusional transport only and cannot be used to estimate gas permeability. Also, this model does not include percolation effects and the creation of inaccessible porosity.

Ongoing work by Starr$^{15}$ aims at developing a transport property model that relates to the real, physical microstructure of the preform and that includes the anisotropy and residual porosity observed in CVI processing. This model is described below.
Figure 1. Node-bond model represents structure of porosity in woven fiber preform.

NODE-BOND MODEL FOR GAS PERMEABILITY OF CVI COMPOSITES

A general model for the microstructure and gas permeability of woven fiber preforms and composites is based on a node-bond model. In this model the porosity is represented as a network of spherical "nodes" connected by cylindrical "bonds" (Figure 1). The network geometry is directly related to the regular structure of the woven preform. Nodes correspond to the large pores created at tow crossing points while bonds correspond to the smaller channels that connect nodes to their nearest neighbors. The spatial arrangement of the nodes and their coordination number (number of nearest neighbors) is fixed by the weave architecture i.e. tow spacing and weave or braid pattern. Values of the node and bond diameters exhibit a distribution of values around an average which is set to match the overall volume fraction of intertow porosity.
As density increases the smaller bonds close and the fraction of open bonds approaches the percolation limit. The gas permeability of this network is calculated assuming Poiseuille flow through the cylindrical bonds with the given distribution of diameters. Densification is simulated by incrementally reducing the bond and node dimensions and recalculating the network pore volume, surface area and permeability. As densification proceeds the smaller bonds close (Figure 2), and as the fraction of open bonds approaches the percolation limit the accessible pore fraction and the network permeability both vanish exponentially.

The model can be used to estimate the permeability of a square-weave, cloth layup preform and composite. This estimate is shown in Figure 3 where \( K_z \) is perpendicular to the cloth. The preform structure assumes 15 \( \mu \)m fibers with 500 filaments/tow, tow spacing of 0.15 cm in the cloth (X and Y directions) and a cloth
**Figure 3.** Estimated gas permeability of a square-weave, cloth layup preform and composite as function of density.

A spacing of 0.03 cm. These parameters yield an overall fiber loading of 40%. It is assumed that the 60% porosity includes 40% porosity within the tow and 20% porosity between the tows. The overall form of the estimation is reasonable. It shows an order of magnitude higher permeability parallel to the cloth layers and a sharp drop in both values as the density approaches approximately 93% full density.

**CONCLUSIONS: PHASE I**

Development of microstructure evolution and gas permeability models for CVI densification of fiber preforms has shown steady progress over the past decade. A node-bond model now being developed at Georgia Tech relates directly to preform fiber architecture and exhibits realistic values of gas permeability for anisotropic preform structures.
REFERENCES


