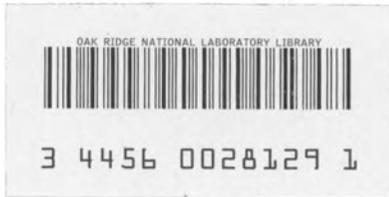


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Fabrication of Fiber-Reinforced Ceramic Composites by Chemical Vapor Infiltration

A. J. Caputo
W. J. Lackey

*Fossil
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FABRICATION OF FIBER-REINFORCED CERAMIC COMPOSITES
BY CHEMICAL VAPOR INFILTRATION

A. J. Caputo and W. J. Lackey

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NOTICE

This document contains information of a preliminary nature. It is subject to revision or correction and therefore does not represent a final report.

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FABRICATION OF FIBER-REINFORCED CERAMIC COMPOSITES
BY CHEMICAL VAPOR INFILTRATION*

A. J. Caputo and W. J. Lackey

ABSTRACT

Ceramic fiber-ceramic matrix composites are of interest because of their potentially higher strength and toughness. A vacuum forming technique was used to fabricate low-density structures from SiC fibers. These fibrous structures were then infiltrated by chemical vapor deposition. Matrices of Si_3N_4 and SiC have been deposited.

INTRODUCTION

Increasingly, ceramic composites are coming of age. The primary reason for the current interest in composites is their improved toughness and strength. Fiber composites are currently in the limelight as a result of exceptionally high toughness values recently achieved for glass ceramics¹⁻³ reinforced with silicon carbide fibers, high strengths achieved by German researchers with SiC or carbon fibers infiltrated with a chemically vapor deposited SiC matrix,⁴ and unpublished French work on a composite of SiC fibers with a chemically vapor deposited SiC matrix. Since use of ceramics is frequently restricted because of inadequate toughness, any progress towards toughening ceramics is potentially important.

Clearly, fiber-reinforced ceramics have the potential for improving tensile strength as well as toughness. However, a generic problem that must be overcome is that normal ceramic fabrication processes tend to mechanically and chemically damage the fibers when they are consolidated within a ceramic matrix. For example, the fibers may be broken by a pressing operation, or the high sintering temperature required to densify the ceramic matrix may damage the surface of the fibers or cause them to react chemically with the matrix. The objective of this work is to form

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the matrix by a comparatively low-stress low-temperature chemical vapor deposition (CVD) infiltration process, which will avoid the pitfalls of conventional ceramic processing.

PRIOR WORK

In extensive prior work, chemical vapor deposition was used to form the matrix of a fiber-reinforced ceramic composite, as summarized in Table 1. The earliest work was with carbon fibers and a carbon matrix, but considerable work has been performed with matrices of SiC, Si₃N₄, B₄C, BN, and TiB₂. It is well established that such composites have excellent mechanical properties, but without exception, processing times required for CVD infiltration have been extremely long (several weeks). Our goal is to reduce processing times to several hours so that product costs are reduced sufficiently to make the use of these materials practical for many industrial applications.

Table 1. Ceramic composites prepared by chemical vapor infiltration

Matrix material	Reinforcing material	Reference
SiC, BN, carbon	Carbon spheres	5
Carbon	Graphite fibers plus spheres	5
Chromium carbide	Nickel spheres	5
Carbon	Carbon fibers	6,7
SiC	Graphite fibers	4,8
SiC	SiC fibers	4,8
B ₄ C	Si ₃ N ₄ fibers	8
SiC, TiB ₂ , Al ₂ O ₃	Al ₂ O ₃ fibers	8
SiC, TiB ₂ , B ₄ C, TiC, carbon	Filament-wound carbon fibers	9,10
SiC	Carbon-coated carbon	11
BN	BN and SiO ₂ fibers	12
ZrC	Carbon fibers	13
TiC	Graphite fibers	14

The CVD infiltration work previously referenced and the general level of understanding of the thermodynamics¹⁵⁻⁷ and kinetics¹⁸ of the CVD process are of valuable assistance in optimizing the infiltration process. For example, infiltration, as opposed to formation of an undesirable surface coating is clearly aided by low deposition temperatures.⁴ That is, the process should be rate limited by a chemical reaction rather than by diffusion through the pores of the preform or by diffusion through a stagnant gas boundary layer.¹⁹ Other process conditions that cause deposition to be reaction rate limited are reduced pressure, dilution of reactants with low-molecular-weight inert gases, and an endothermic deposition reaction that has a high activation energy.

Three basic types of CVD infiltration processes have been used by previous investigators, as summarized by Kotlensky.¹⁹ These are (1) an isothermal process in which the fibrous preform is positioned inside a furnace and the CVD reactant gases, as they flow through the furnace, diffuse into the preform; (2) a thermal gradient process in which infiltration, as opposed to overcoating the surface of the preform, is encouraged by the presence of a steep thermal gradient through the thickness of the preform (reactant gases flow over the cooler surface of the preform and diffuse through the preform thickness toward the heated surface); and (3) a pressure gradient technique in which reactant gases flow through an isothermal fibrous preform.

EXPERIMENTAL PROCEDURE

The CVD infiltration method used in this work is a new approach combining the thermal gradient and pressure gradient processes.* A schematic of the critical portion of the equipment, the gas injector-preform holder, is shown in Fig. 1. The fibrous preform is positioned within a furnace inside a water-cooled metal holder. The metal holder cools the bottom and side surfaces of the preform to prevent deposition there. The top of the fibrous preform is heated by the furnace. Reactant gases flow through the preform, and when the reactants are sufficiently

*Patent applied for.

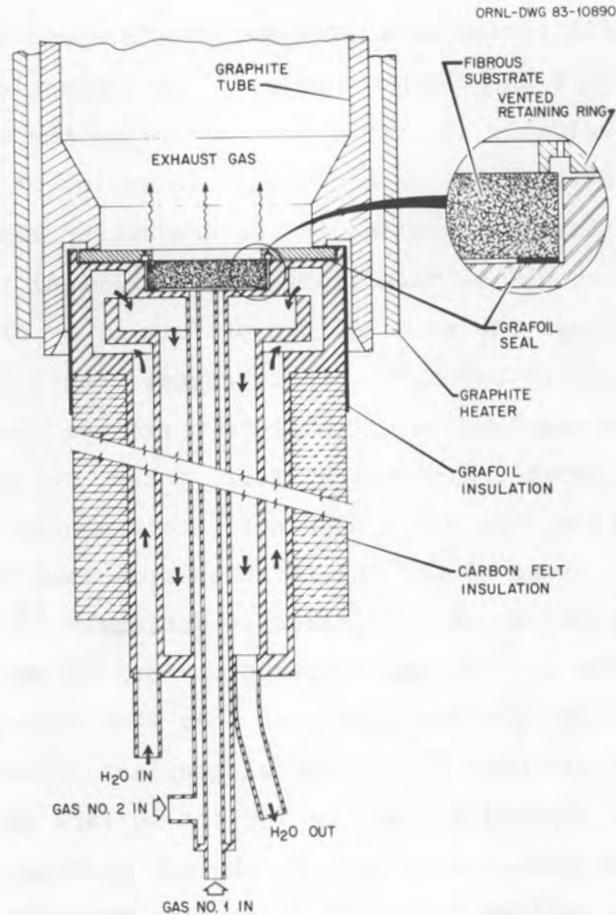


Fig. 1. Chemical vapor deposition system for infiltration of fiber-reinforced ceramic composites using thermal gradient and gas flow-through system.

heated (near the top of the preform) the chemical vapor deposition reaction occurs. As a result, the fibers are coated and surrounded by the reaction product to form the matrix of the composite. The higher the temperature, the more rapid the deposition; therefore, the infiltration occurs progressively from the top of the preform toward the bottom. The Grafoil seal forces the reactant gases to flow into the fibrous preform. When the top surface region becomes coated over, the gases flow up into the preform, then radially through the preform, to the annular void space around the preform and then escape through the vented retaining ring. As matrix deposition occurs, the thermal conductivity of the preform increases and thus the portion of the preform that is hot enough for appreciable deposition to occur progressively increases.

Recently, we have slightly improved the design over that shown in Fig. 1. This change consists of cementing the fibrous preform inside a graphite ring. The ring serves two purposes. First, it seals the periphery of the lower portion of the preform so that the reactant gas is forced to flow further up into the preform. Gas exits through radial holes drilled through the ring near its top. The graphite ring extends part way over the bottom of the preform and therefore also permits a better seal against the water-cooled metal gas distributor.

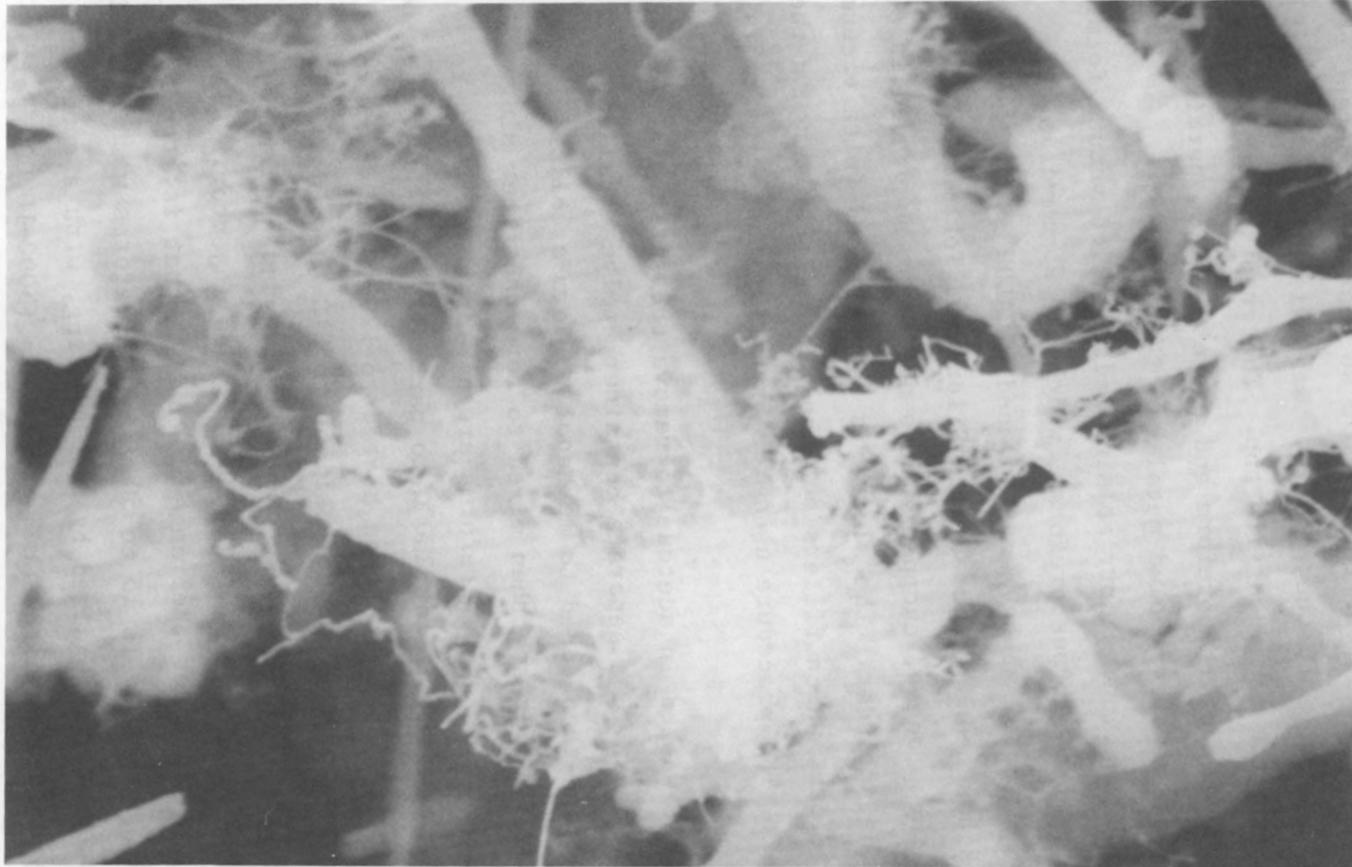
Before the infiltration, slurry molding (also called vacuum forming) is used to fabricate the fibrous preforms, which are 25-mm-diam by 6-mm-thick disks. To date, we have mainly used NICALON* SiC fibers, although some work has been performed with Silar† and Tokamax‡ SiC fibers. In the slurry molding process, chopped fibers are first suspended in a liquid that contains a binder. The slurry is then drained (with vacuum assist) through a cloth, which filters out the fibers, resulting in the formation of a disk. The procedure used for preparing the preforms is as follows:

Fibers are first washed in acetone to remove the sizing, air dried, and then added to water. In a separate container, polycarbosilane is dissolved in hexane and then added to methanol to form a colloidal suspension. The two liquids are then mixed to form the fiber slurry. After slurry molding, the fibrous preforms are air dried at 150°C to remove the liquids and air cured at 250°C to partially set the binder to permit handling. The preforms are then heated to 1200°C in argon to complete the conversion of the polycarbosilane to SiC (see Fig. 2) to bond the fibers sufficiently to allow some machining and handling associated with inserting the preform in the CVD gas injector. Preform densities have been varied over the range 15 to 50%; that is, the fiber content of the final composite is 15 to 50 vol %. Preforms with densities in the 40 to 50% ranges were obtained by wet pressing, with a punch and die, the wet slurry-molded preforms at 34 to 68 MPa (5000–10,000 psi).

*Nippon Carbon Company, Japan.

†Arco Company, Greer, S.C.

‡Tokai Carbon Company, Japan.



10 μ m

Fig. 2. Scanning electron micrograph of preform before CVD infiltration; Tokamax SiC fibers bonded with SiC derived from polycarbosilane.

Two chemical systems have been investigated: (1) SiC fibers with a CVD SiC matrix and (2) SiC fibers with a CVD Si₃N₄ matrix. A total of 24 infiltration runs have been made, 12 with each matrix. To deposit SiC, only one gas inlet tube is needed, but for Si₃N₄ two tubes (see Fig. 1) are required because the NH₃ and SiCl₄ reactant gases must be kept separated until they enter the deposition region; otherwise premature deposition plugs the gas inlet line. The chemical reactions involved are



Process parameters for the CVD infiltration have been varied to investigate their influence on the extent of infiltration. Temperatures have been in the range 1100 to 1500°C for SiC and 1200 to 1300°C for Si₃N₄ deposition. Furnace pressures have been varied from 3.3 to 100 kPa (25 to 760 torr). The concentrations of the reactants, including hydrogen, and the total gas flow rate have been varied. Infiltration times of 2 to 12 h have been investigated. Following a series of test runs, nominal conditions for deposition of SiC were 1200°C, a methyltrichlorosilane gas flow rate of 50 mm³/min, a hydrogen-to-methyltrichlorosilane ratio of 10, and a pressure of 100 kPa (1 atm).

RESULTS AND DISCUSSION

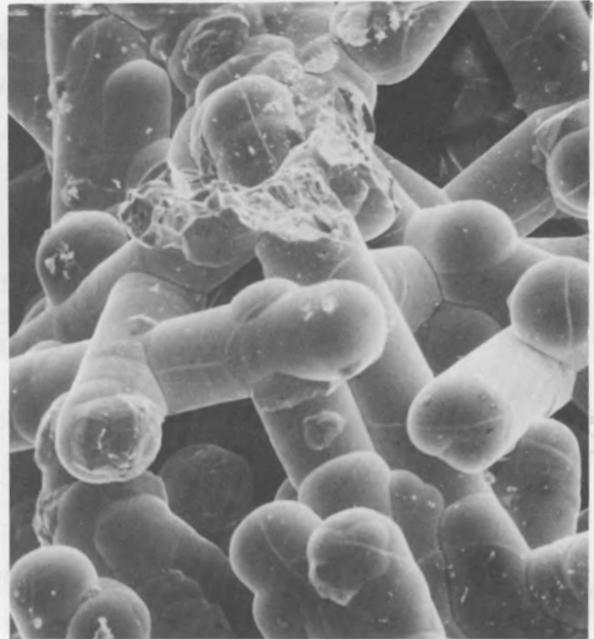
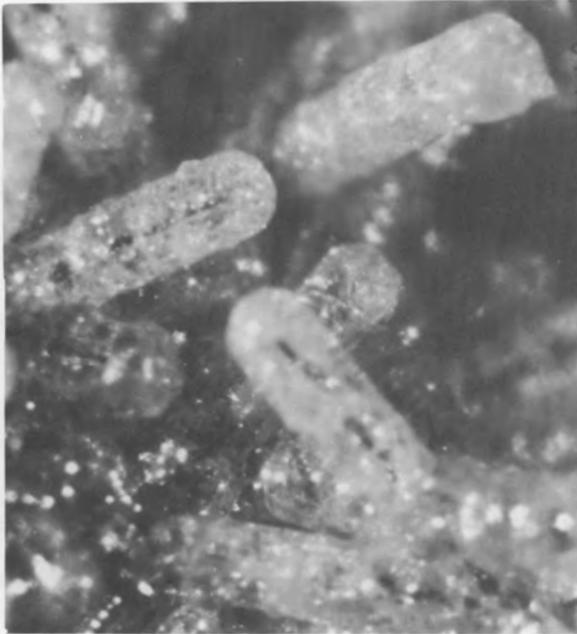
SIC FIBERS, Si₃N₄ MATRIX

The early work was with SiC fibrous preforms having initial densities of 15 to 25% and densities after infiltration with Si₃N₄ in the range of 1.3 to 2.0 Mg/m³. The matrix was generally α-Si₃N₄ but depending on deposition conditions could be amorphous or β-Si₃N₄.

Optical and scanning electron micrographs of both the hot and cold faces of typical specimens are shown in Fig. 3. The relatively small size of the SiC fiber and the appreciable thickness of the CVD Si₃N₄ deposit at the hot face is shown in Fig. 3(a). It is apparent from Fig. 3(c,d) that

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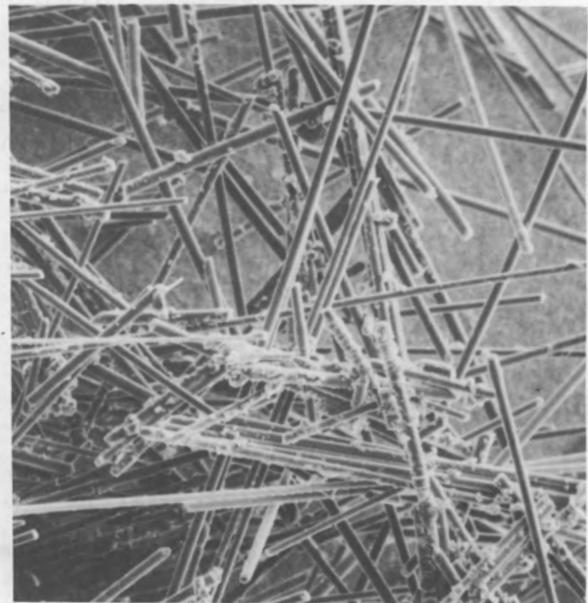
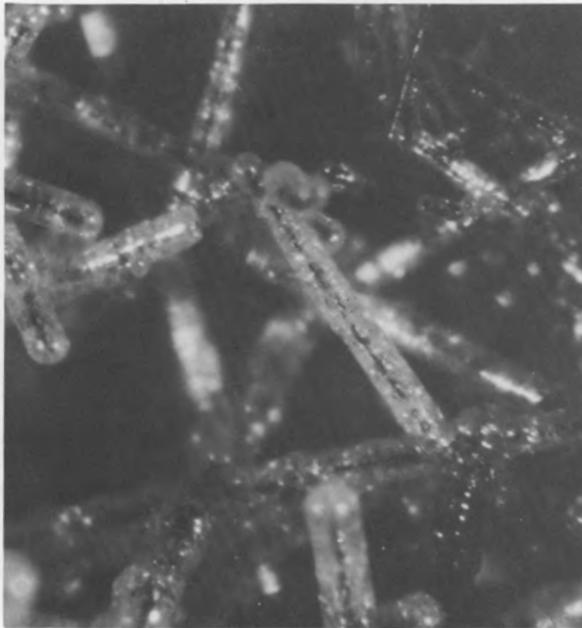


(a)

(b)

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M-18810



(c)

(d)

400 μm

Fig. 3. Micrographs of SiC fiber-Si₃N₄ matrix composite disk showing considerable deposition of Si₃N₄ at the hot face (a,b) and much less deposition, as desired, near the cold face (c,d).

the extent of matrix deposition near the cold face of the preform was much less, as desired, than at the hot face. The interlocking of the fibers with the CVD Si_3N_4 matrix as the coating progressed is quite evident in the scanning micrograph of the hot face [Fig. 3(b)]. A cross section of an SiC fiber- Si_3N_4 matrix sample is shown in Fig. 4. The low fiber concentration (15 vol %) of this specimen is evident, as is the appreciable amount of deposited Si_3N_4 and remaining porosity. The larger voids correspond to areas in the preform with fibers absent. Recent results with higher fiber contents show that matrix infiltration is more complete (higher overall density), probably as a result of smaller fiber-to-fiber distances.

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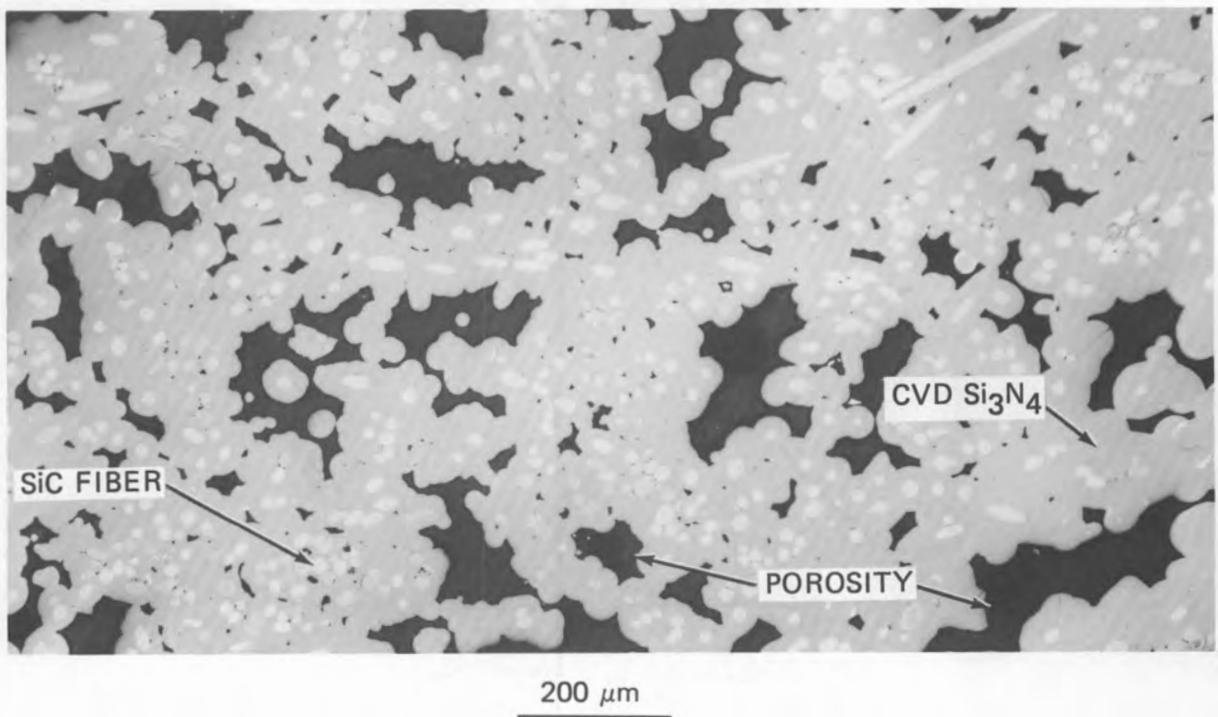


Fig. 4. Cross section of SiC fiber- Si_3N_4 matrix composite fabricated by chemical vapor infiltration.

Although the molding and the infiltration process were not fully developed at this stage, an initial set of samples was prepared for flexure testing. Three samples with 18 vol % fiber concentration and CVD infiltrated densities of 1.8 to 2.0 Mg/m³ had strengths ranging from 43 to 57 MPa (6300 to 8300 psi) in a four-point flexural test. Although the strengths are lower than desired, two of the three specimens exhibited the desired ability to carry a load after crack initiation. This is shown in the load versus ram displacement curve for one of the specimens in Fig. 5. Higher strengths are expected from increased fiber concentrations.

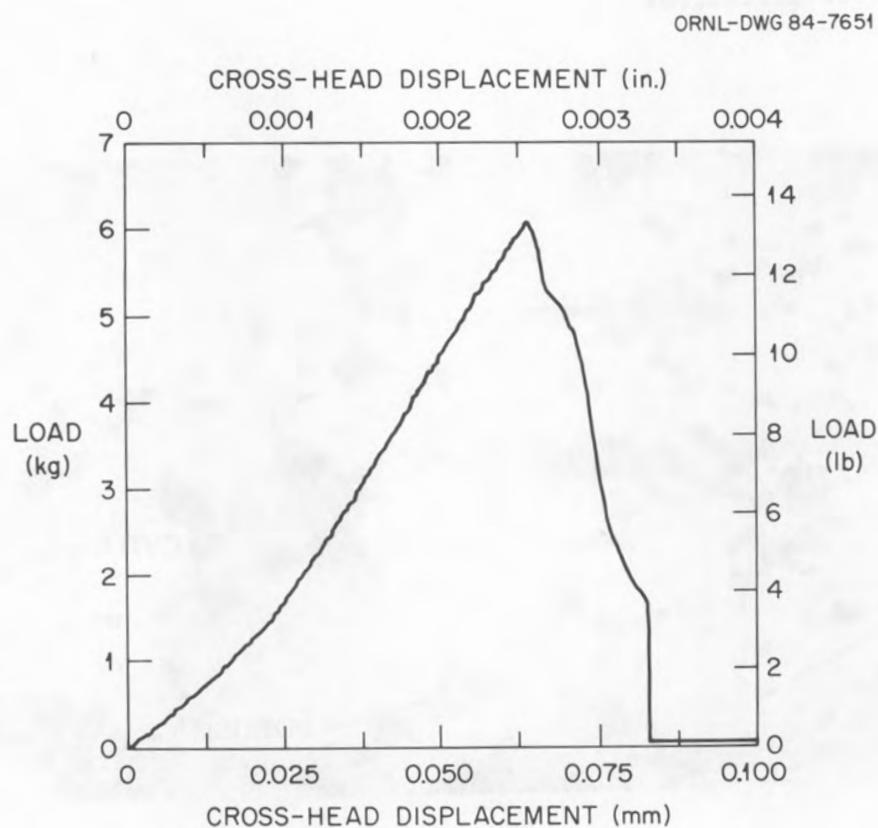


Fig. 5. Load versus ram displacement for an SiC fiber-Si₃N₄ matrix composite in a four-point bend test.

SiC FIBERS, SiC MATRIX

Although deposition conditions have not yet been optimized, SiC-SiC composites of reasonably high densities (2.2 Mg/m^3) have been prepared with infiltration times of about 6 h for fiber volume contents in the range 20 to 50%. The SiC matrix was predominantly β -SiC, but traces of silicon have been observed by X-ray diffraction for some samples. In addition, the presence of α -SiC cannot be totally discounted because its major X-ray diffraction peaks coincide with those of β -SiC. However, the absence of minor peaks for α -SiC shows that any α -SiC is present in quantities less than 10%.

The highest matrix densities were obtained by a two-step infiltration process. In the initial stage, all the gas flowed straight through the preform; in the second stage, the gas flowed into the preform, then radially through the cooler edges of the preform (Fig. 1).

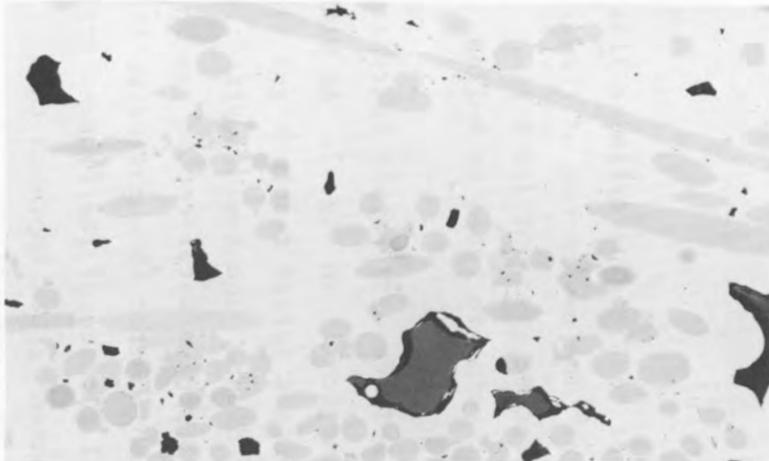
Micrographs showing as-infiltrated composites with densities of 1.8 and 2.0 Mg/m^3 are shown in Fig. 6(a) and 6(b), respectively, while the respective preforms with initial densities of 25 and 50% are shown in Fig. 6(c) and 6(d).

FABRICATION OF TUBES

Many applications of materials, such as heat engine cylinder liners and heat exchangers, require tubular shapes. The new method of chemical vapor infiltration, which combines a temperature gradient and a gas pressure gradient, should be capable of extension to tubes or hollow cylinders.

The conceptualized apparatus that we believe will permit fabrication of tubes or hollow cylinders is shown schematically in Fig. 7. As with disks, infiltration is assisted by simultaneous forced reactant gas flow and the presence of a steep temperature gradient. The inside cylindrical surface of the fibrous preform will be cooled by a water-cooled gas injector. Reactant gas will flow from this injector through the wall of the fibrous preform. The outer cylindrical surface of the fibrous preform will be heated by a furnace. When the reactant gases approach this surface they will react to deposit the matrix on the fibers. Product gases and any unreacted gases will flow axially through the fibrous form and exit at either end of the tube.

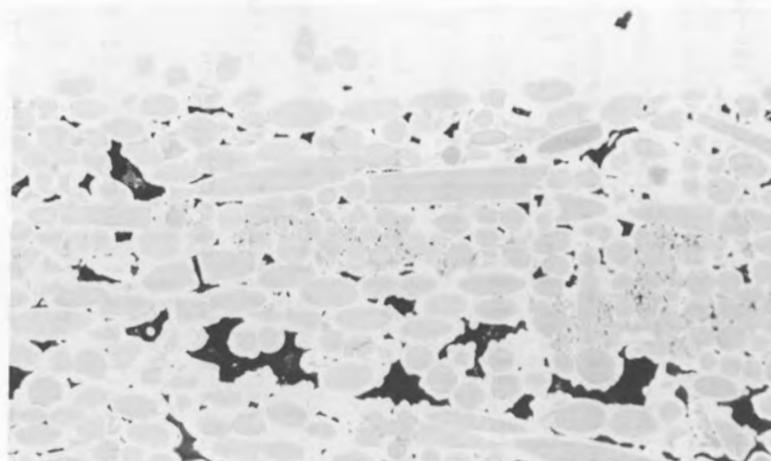
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(a)

100 μm

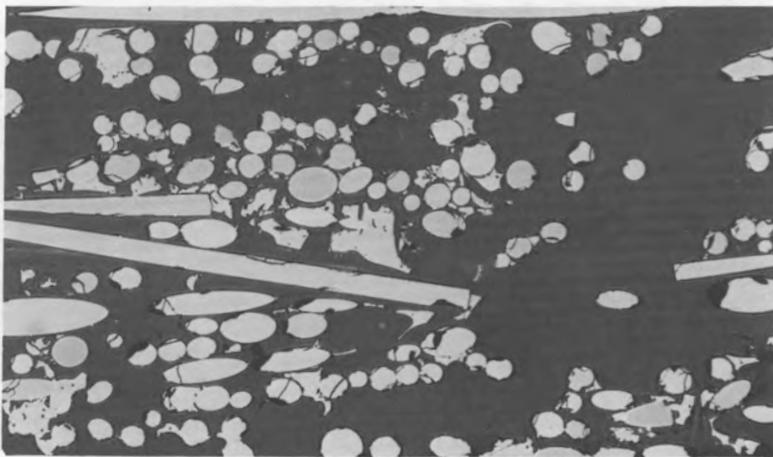
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(b)

100 μm

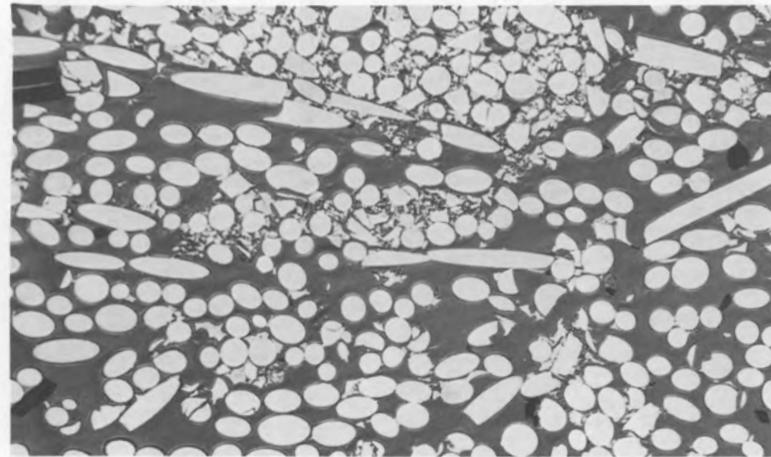
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(c)

100 μm

Y-196105



(d)

100 μm

Fig. 6. Micrographs of SiC fiber-SiC CVD matrix with infiltrated densities of (a) 1.8 Mg/m^3 and (b) 2.0 Mg/m^3 , which had initial preform densities of (c) 25% and (d) 50%, respectively.

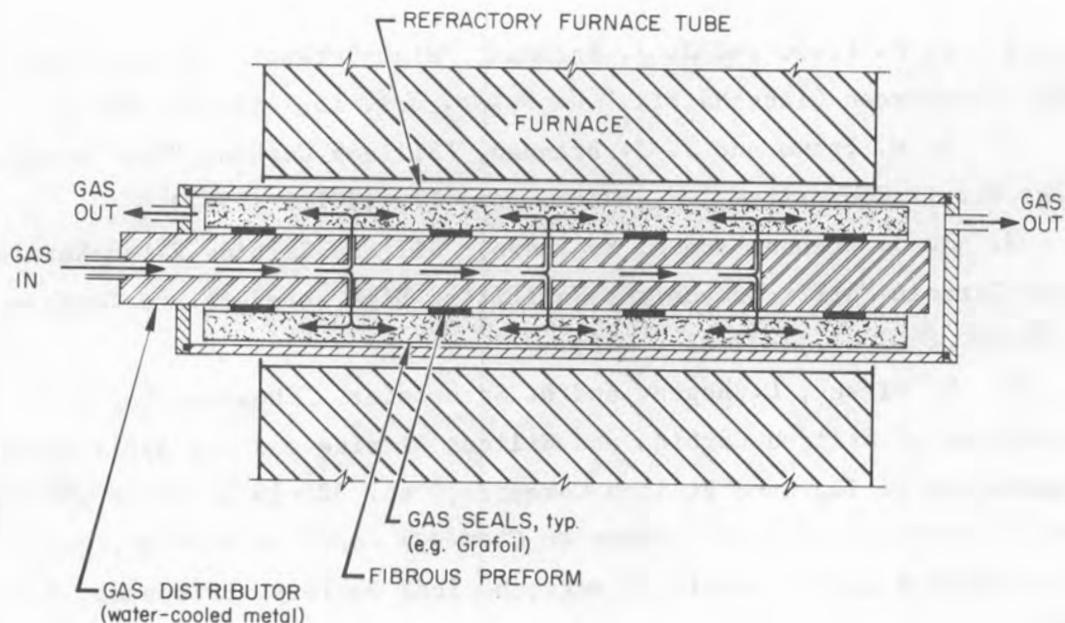


Fig. 7. Concept for the fabrication of fiber-reinforced tubes or hollow cylinders by chemical vapor deposition using the thermal gradient and gas flow-through system.

CONCLUSIONS

Ceramic fiber-ceramic matrix composites were prepared by the chemical vapor deposition process. Our major goal of significantly reducing the infiltration times was accomplished. We have conceived a process, fabricated hardware, and demonstrated on a laboratory scale (25-mm-diam \times 6-mm-thick disk) a process that permits CVD infiltration of an SiC fibrous preform with either an Si_3N_4 or an SiC matrix in 2 to 12 h. The process should be applicable to infiltration of fibrous preforms prepared by slurry molding, stacking of layers of cloth, or filament winding. Two types of composites ($\text{SiC-Si}_3\text{N}_4$ and SiC-SiC) have been produced at densities of about 2.0 Mg/m^3 , which was near the desired range. A scheme has also been conceived whereby the new infiltration process could be used for fabrication of tubes or hollow cylinders. This is an important advance because the ability to fabricate tubes or hollow cylinders would offer the hope that the process could be used for fabrication of toughened high-strength heat exchanger tubes and heat engine cylinder liners and valve guides.

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