

Multiscale Modeling and Process Design of Reaction-Diffusion in CVI for CMC Manufacturing

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Ceramic matrix composites (CMCs), which are expected to be used in high-temperature components of aircraft engines, consist of millimeter-scale preforms woven from yarns of hundreds of SiC fibers each with a diameter of about 10 μm . In the CMCs manufacturing process, chemical vapor infiltration (CVI) is performed in meter-scale reactors to form SiC matrix inside a preform. This CVI process requires technically precise control of reactions and diffusion in both the microstructure of preforms and yarns and the microscopic and macroscopic scales of the entire reactor to achieve uniform deposition. In this study, we analyzed micro-scale phenomena using reaction and diffusion equations and macro-scale phenomena using computational fluid dynamics (CFD) with the aim of efficiently designing reactors that are adaptable to a wide range of operating conditions.

1. Introduction

According to a report by the Japan Aircraft Development Corporation, the number of commercial jet airliners in operation worldwide is expected to grow at an annual rate of 2.4% from 2024 to 2044, raising concerns about the increased carbon dioxide (CO_2) emissions resulting from the growing number of passenger aircraft accompanying market growth⁽¹⁾. In addition to reducing CO_2 emissions, research and development of aircraft engines is focused on reducing fuel consumption to meet the demand for lower operating costs.

Among the measures for reducing the fuel consumption of aircraft, increasing the pressure and temperature of the engine cycle is considered an effective approach for enhancing thermal efficiency⁽²⁾. To accommodate higher temperatures, cooling technologies are introduced in addition to the use of heat-resistant materials and coatings in turbine sections. It has been reported that every 40°C increase in the temperature capability of turbine blades improves the fuel efficiency of jet airliners by 1%⁽³⁾. However, the temperature capability of conventional materials, such as rare-earth-added nickel-based single-crystal alloys, has already reached 1,150°C, and further improvement is limited^{(2), (3)}.

Accordingly, SiC_f/SiC CMCs, which use SiC fibers (SiC_f) as the reinforcing material and SiC ceramics as the matrix,

have attracted attention as one of the next-generation materials, and have been under development since the 1960s mainly in Western countries, particularly Germany, France, and the United States. The temperature capability of SiC_f/SiC CMCs reaches as high as 1,400°C, which significantly exceeds that of nickel-based single-crystal alloys. In addition, in terms of material density, SiC_f/SiC CMCs have a density of approximately 2.0 to $3.0 \times 10^3 \text{ kg/m}^3$, which is about one-third that of nickel-based single-crystal alloys at approximately $9.0 \times 10^3 \text{ kg/m}^3$. Therefore, SiC_f/SiC CMCs can also contribute to improved fuel efficiency from the standpoint of weight reduction. Thus, SiC_f/SiC CMCs are materials capable of achieving both the enhanced heat resistance and weight reduction required for reducing fuel consumption.

The fabrication process of CMCs consists of multiple steps and can be broadly classified into (1) fabrication of preforms, (2) fabrication of composite materials, and (3) secondary processing of the composite materials. The preforms in (1) are textile preforms fabricated by three-dimensionally weaving yarns consisting of bundles of several hundred SiC fibers, each with a diameter of approximately 10 μm . **Figure 1** shows the schematic diagrams of the yarn and the preform. The yarns are densely bundled with pores of approximately 1 μm between individual fibers. In the preform fabricated by weaving these yarns, the gaps outside

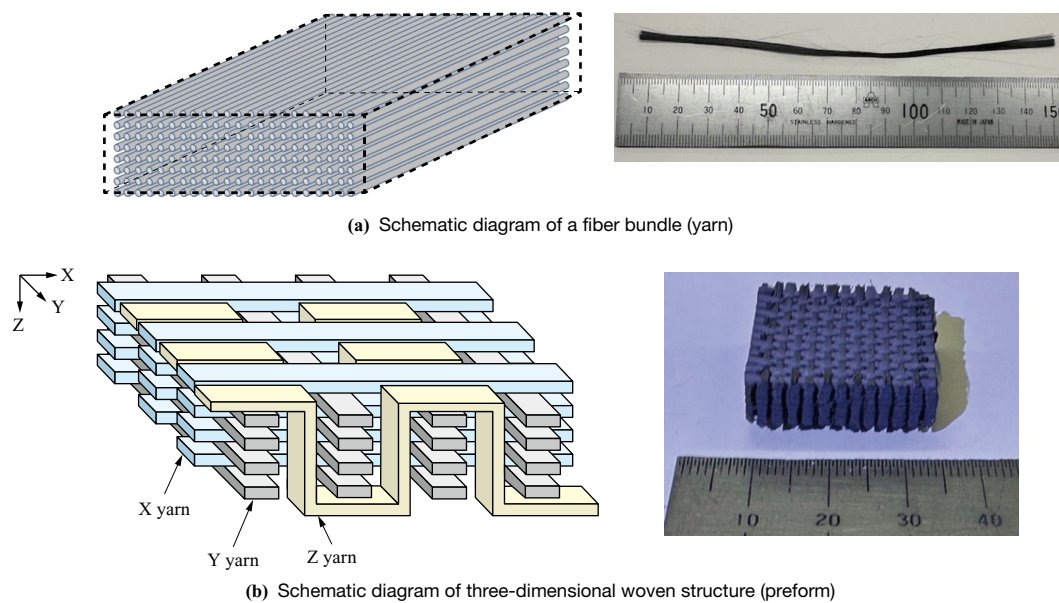


Fig. 1 Schematic diagrams of yarn and preform

the yarns are approximately several hundred micrometers. As a result, the preform has a complex multilayered structure with pore sizes ranging from 1 to 1,000 μm . The composite material fabrication process in (2) is extremely challenging because a uniform, highly crystalline SiC matrix must be formed within the preform, which has a complex three-dimensional structure ranging from the microscopic to the macroscopic scales. One of the methods commonly used to address this challenge is the chemical vapor infiltration (CVI) method. This paper introduces the current status of research and development on the CVI method.

2. CVI method

The CVI method has been developed with the aim of applying the well-known chemical vapor deposition (CVD) method to infiltration and deposition in porous or complex microstructures. There are few differences in practical operation between the CVD and CVI methods. However, in the CVI method, a hot-wall-type reactor with overall heating is commonly used, and thermal decomposition of the precursor as well as gas-phase reactions proceed in the heating region, making the reaction mechanism more complex than that of the CVD method. In addition, the CVI method requires gases to diffuse into the preform with its three-dimensional structure to deposit the matrix, and therefore enhancing gas diffusivity by reducing the total pressure inside the reactor is an important consideration.

In general, infiltration and deposition of the SiC matrix by the CVI method commonly use a mixed gas of methyltrichlorosilane (CH_3SiCl_3 : MTS) and hydrogen (H_2) as the source gas, and this study also employs the MTS/ H_2 gas system. MTS is characterized by its ability to relatively easily form stoichiometric 3C-SiC (cubic silicon carbide, also referred to as β -SiC) because each molecule contains one silicon (Si) atom and one carbon (C) atom. In addition, it is inexpensive and has low toxicity.

In the hot-wall-type CVI method investigated in this study, the supply rate of the source gases is controlled by a mass flow controller by generating a pressure difference between the gas supply side and the reactor. The gases are introduced under laminar flow conditions, and the reaction is carried out while maintaining the reactor at constant pressure and high temperature. Because the source gases are exhausted before reaching equilibrium, the reaction is governed by kinetics^{(4), (5)}. The source gases undergo continuous thermal decomposition in the gas phase and reach the interior of the preform through gas-phase diffusion. The diffusion mode inside the reactor and the preform is governed by either molecular diffusion, in which molecules move due to concentration gradients caused by intermolecular collisions, or Knudsen diffusion, in which molecules diffuse through collisions with the walls. An overview of these reaction mechanisms is schematically shown in Fig. 2. Diffusion and reaction proceed sequentially, and in this reaction system, the surface reaction is the rate-determining step under many reaction conditions⁽⁴⁾. However, reactors used in the CVI method are often large, ranging from sub-meter to meter scale. Therefore, when considering the reactor as a whole, the absolute thickness of the boundary layer δ becomes large, and from the viewpoint of the preform size, the process tends to become diffusion-controlled near the reactor walls.

3. Optimal design of CVI method

Deposition within the preform is described below. The rate constants, physical property values, and related equations relevant to the reaction mechanisms were taken from previous research reports^{(4), (5)}. Here, to facilitate understanding of the outline of the reaction mechanisms, we assumed that the film-forming species (precursor) directly converted into the SiC film is MTS.

3.1 Diffusion within yarns

Figure 3 shows the calculated mean free path and diffusion

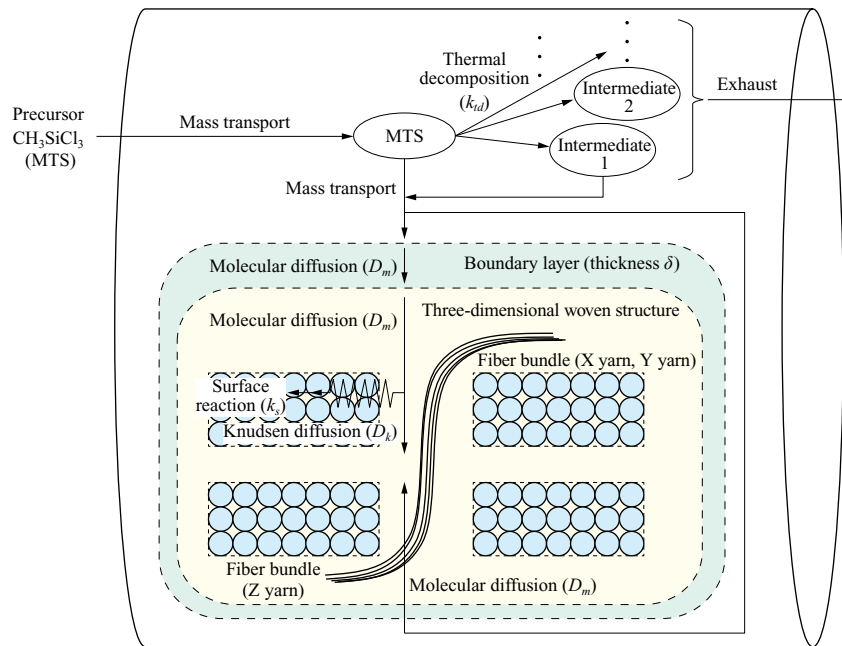


Fig. 2 Reaction mechanism for MTS/H₂ gas

coefficient of MTS molecules. Because the gaps between fibers within the yarns are approximately 1 μm , it is necessary to consider the Knudsen number, $K_n = \lambda/L$ (where λ is the mean free path (m) and L is the characteristic length, i.e., the gap between fibers (m)), to determine whether the diffusion is molecular diffusion or Knudsen diffusion. When

the Knudsen number is generally less than 1, the system is in the continuum regime, and molecular diffusion dominated by intermolecular collisions occurs. When the Knudsen number is generally greater than 1, the system enters the molecular flow regime, and Knudsen diffusion dominated by collisions with the walls rather than intermolecular collisions

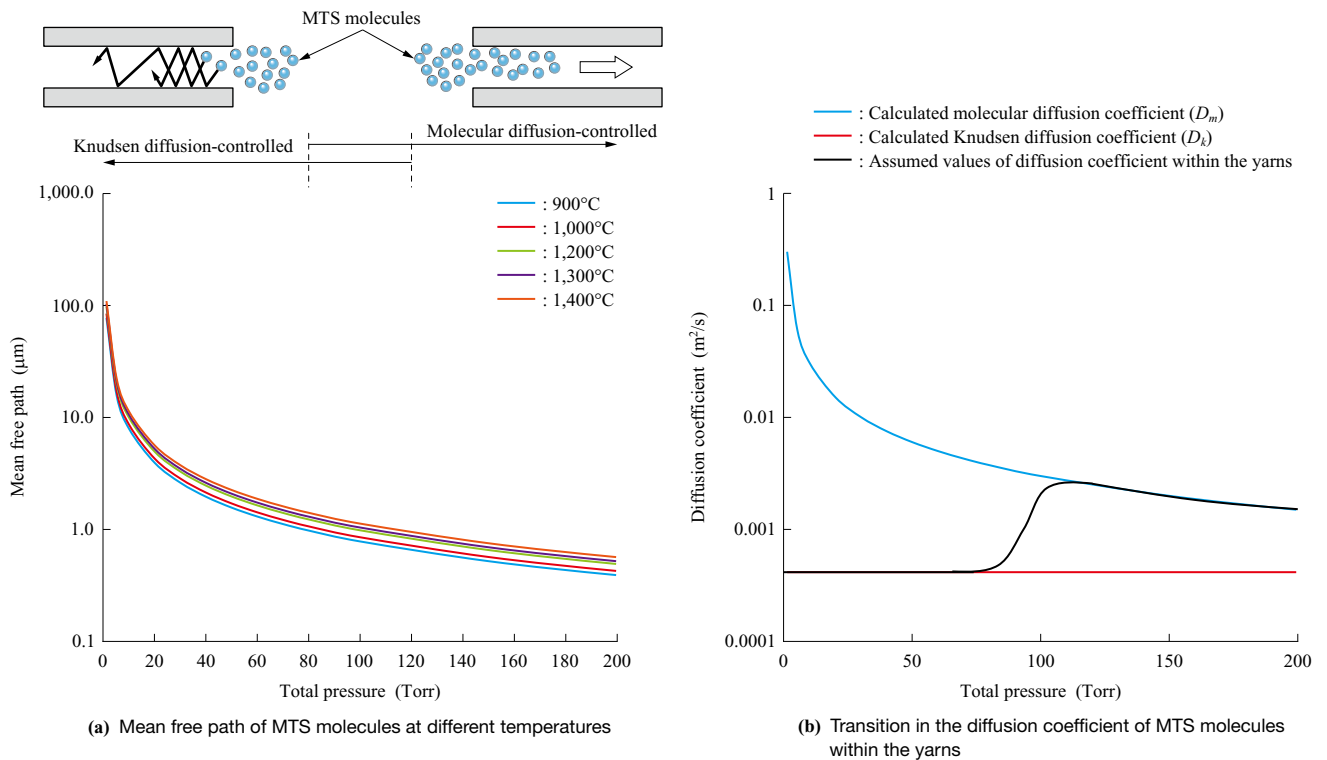


Fig. 3 Mean free path and diffusion coefficient of MTS molecules

occurs. Because the characteristic length L is the gap between fibers (approximately 1 μm), the diffusion mode transitions depending on whether the mean free path λ exceeds 1 μm .

Thus, assuming the diameter of MTS molecules to be 0.5913 nm, the mean free path λ was calculated for temperatures ranging from 900 to 1,400°C and total pressures up to 200 Torr (26.6 kPa), as shown in **Fig. 3-(a)**. The temperature dependence is small, and the diffusion mode generally shifts from Knudsen diffusion at pressures below 100 Torr (13.3 kPa) to molecular diffusion at higher pressures. The molecular diffusion coefficient D_m (m^2/s) can be calculated using the Chapman-Enskog equation and has a pressure dependence expressed by the following proportional relation:

$$D_m \propto T^{\frac{3}{2}} P^{-1} \quad \dots\dots\dots (1)$$

where T is temperature (K) and P is pressure (Pa). In contrast, the Knudsen diffusion coefficient D_k (m^2/s) is expressed by

$$D_k = \frac{1}{3} v_T L \quad \dots\dots\dots (2)$$

$$v_T = \sqrt{\frac{8RT}{\pi M}} \quad \dots\dots\dots (3)$$

and is independent of pressure (where v_T is the mean thermal velocity (m/s), R is the gas constant (J/(K·mol)), and M is the molecular molar mass (kg/mol)). **Figure 3-(b)** shows the calculated diffusion coefficients of MTS molecules at 1,000°C. As with the mean free path, these coefficients remain in the same order of magnitude even when calculated over the temperature range of 900 to 1,400°C. The region near 100 Torr is a transition regime, and because the diffusion mode changes around this pressure, the diffusion coefficient varies significantly, as indicated by the black line in the figure.

When considering only the interior of the yarns, the system shifts to the continuum regime at higher pressures, resulting in an increase in the diffusion coefficient. This indicates that infiltration within the yarns improves at higher pressures, which is opposite to the generally accepted tendency for the CVI process as a whole. Although the details are omitted here, deposition experiments conducted under conditions where Knudsen diffusion was dominant, using a substrate that simulates the fine pores within the yarns, showed that infiltration improved as the total pressure increased in the pressure range around 100 Torr.

In contrast, infiltration decreases at higher pressures for diffusion between yarns within the preform. Unlike diffusion within the yarn, which transitions from Knudsen diffusion to molecular diffusion around 100 Torr, diffusion between yarns remains molecular diffusion in this pressure range. For the preform as a whole, as shown in **Fig. 2**, the source gas diffuses from the gas phase into the preform, then further diffuses within the yarns and reacts on the fiber surfaces, thereby advancing SiC deposition. These processes occur sequentially (in series), and the extent of infiltration into the deepest region is determined by whether diffusion or reaction becomes the rate-limiting step. Therefore, in many cases,

diffusion between the yarns becomes the rate-limiting step at high pressures, resulting in reduced infiltration regardless of how fast diffusion within the yarns is.

In the following discussion, calculated deposition distribution within the yarns is presented; for simplicity, the diffusion mode is assumed to switch at 100 Torr.

The distribution equations for the precursor concentration C (mol/m^3) within a pore are given by:

$$\frac{C}{C_0} = \frac{\cosh\left(\phi \frac{x}{L}\right)}{\cosh\phi} \quad \dots\dots\dots (4)$$

$$\phi = L \sqrt{\frac{k_s S}{D V}} \quad \dots\dots\dots (5)$$

$$k_s = \frac{1}{4} v_T \eta \quad \dots\dots\dots (6)$$

where C_0 is the initial precursor concentration and ϕ is a coefficient having the same form as the well-known Thiele modulus, a dimensionless number representing the relation between diffusion and reaction rate within a pore. In addition, x is the position within the pore (m), S is the reaction area in the unit cell (m^2), V is the pore volume in the unit cell (m^3), k_s is the surface reaction rate constant (m/s), η is the sticking probability (ranging from 0 to 1), and D is the diffusion coefficient (m^2/s)⁽⁴⁾. D is either D_m or D_k . S/V in the equation corresponds, in the case of yarns, to the ratio of the fiber surface area to the pore volume between the fibers. The values for k_s and η can be obtained from deposition experiments⁽⁴⁾. The coefficient ϕ , in the form of the Thiele modulus in Equation (5), is determined by the relation between the surface reaction rate constant (k_s) and the diffusion coefficient (D); a smaller value of this coefficient indicates better infiltration. For example, even under conditions where the reaction temperature is high and k_s increases, infiltration can be improved if D increases due to the diffusion mode, or if the S/V ratio decreases by enlarging the space within the preform, since these effects decrease ϕ .

Figure 4 shows the calculated precursor-concentration distribution within the yarns using Equation (4) under the conditions of a yarn thickness of 0.12 mm, a deposition temperature of 900 to 1,400°C, and a total pressure range of 0 to 200 Torr. For the diffusion coefficient D , the values shown in **Fig. 3-(b)** were used, and D was switched between D_k and D_m around 100 Torr.

Figure 4-(a) shows the precursor-concentration distribution at total pressures below 100 Torr. Because the diffusion mode below 100 Torr is Knudsen diffusion, the infiltration is independent of pressure. It was found that at total pressures below 100 Torr, where the diffusion coefficient becomes small, a certain amount of MTS molecules reached the deepest region of the yarn ($x = 0.06$ mm) even at 1,400°C. **Figures 4-(b)** and **-(c)** show the precursor-concentration distribution at total pressures above 100 Torr. Because the diffusion mode becomes molecular diffusion, the infiltration decreases as the total pressure increases in accordance with

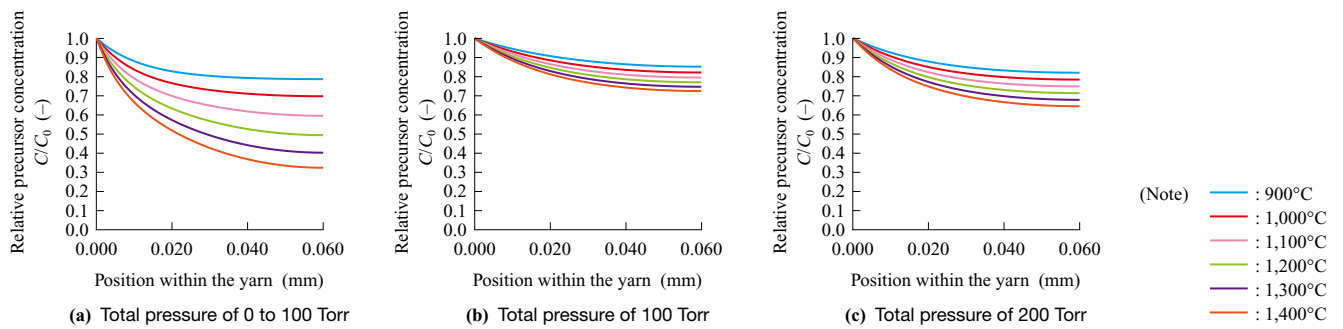


Fig. 4 Normalized precursor concentration distribution in yarn

Equation (1). However, the results showed that the decrease in infiltration is small at all temperatures.

3.2 Diffusion within the preform

As explained in Fig. 1, the preform has a complex multilayered structure. In this section, the discussion is developed using appropriate approximations and simplifications to obtain an overview of the phenomena.

As described in Section 3.1, the diffusion within the yarn changes around 100 Torr; however, for the sake of simplicity, this section considers only pressures below 100 Torr. Diffusion to the deepest region within the preform is not considered at pressures above 100 Torr because diffusion to that region becomes difficult.

At pressures below 100 Torr, the diffusion mode within the yarn is the Knudsen diffusion, and the diffusion coefficient is on the order of 10^{-4} m²/s. In contrast, the space outside the yarn consists of gaps of several hundred micrometers, which are sufficiently larger than the mean free path shown in Fig. 3-(a); therefore, the diffusion mode is molecular diffusion. Accordingly, as indicated by the blue line in Fig. 3-(b), the diffusion coefficient outside the yarn is of the order of 10^{-1} to 10^{-3} m²/s, which is more than an order of magnitude larger than that within the yarn. Based on this, in the sequential diffusion of MTS molecules through the preform and into the yarn, the Knudsen diffusion within the yarn becomes the rate-limiting step. Therefore, when considering deposition within the preform, diffusion within the yarn can be neglected, and only the precursor concentration that reaches the deepest region of the preform by molecular diffusion needs to be considered.

As in the calculation of precursor diffusion within the yarn,

the precursor diffusion distribution within the preform was calculated using Equation (4). Figure 5 shows the results for a preform thickness of 10 mm, a deposition temperature range of 900 to 1,400°C, and a total pressure range of 10 to 100 Torr. Here, the S/V ratio was calculated by regarding the yarn as a single rectangular solid and taking the ratio of the surface area S of the rectangular solid to the pore volume V outside the yarn. In Fig. 5-(a), at a total pressure of 10 Torr (1.33 kPa), the precursor significantly reaches the deepest region ($x = 5$ mm) even at 1,400°C. However, as the total pressure increases to 50 Torr (6.65 kPa) in Fig. 5-(b) and 100 Torr in -(c), the infiltration markedly decreases at reaction temperatures of 1,100°C or higher. That is, under high-temperature and high-pressure conditions such as those shown in Fig. 5-(c), the precursor cannot reach the deepest region within the preform, and therefore matrix deposition cannot occur.

3.3 Deposition within the preform

Up to this point, the discussion has focused on the concentration distribution of MTS molecules, and Fig. 5 shows that diffusion of the precursor to the deepest region within the preform decreases as the total pressure increases. However, the distribution is not necessarily the same when the deposition amount is considered.

In general, the reaction rate (deposition rate) r (mol/(s·m²)) is expressed by

$$r = kC^m \quad (7)$$

where C is the concentration and k is the reaction rate constant. Here, k denotes the surface reaction rate constant k_s . When Equation (7) is considered at the deepest region within the preform, the reaction rate constant k_s increases as

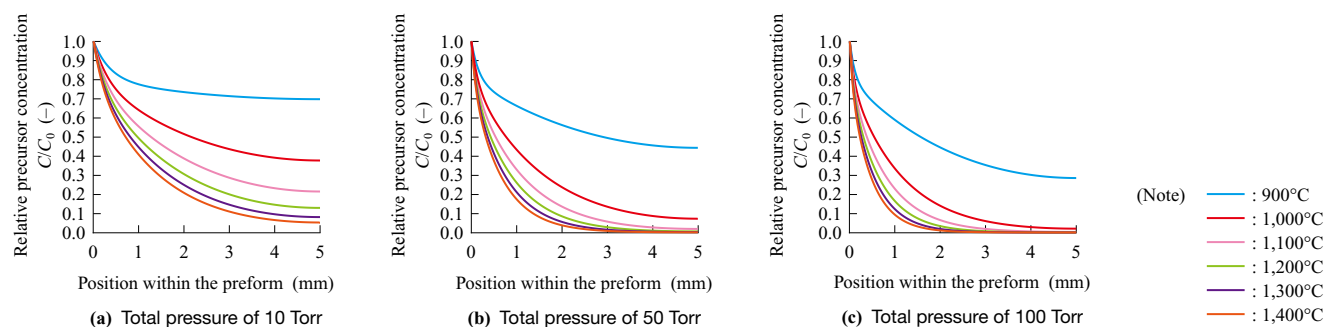


Fig. 5 Normalized precursor concentration distribution in preform

the reaction temperature increases, whereas the concentration C decreases, as shown in **Fig. 5**. Thus, when the reaction temperature changes, the deposition amount increases or decreases because of this trade-off relationship. In the equation, m is the reaction order and it can take positive, negative, or zero values. In this study, a first-order reaction with $m = 1$ was assumed to simplify understanding of the phenomenon. Since the reaction mechanism is of the Langmuir-Hinshelwood type, the actual value of m can range from 0 to 1; however, in most cases, assuming $m = 1$ provides an appropriate understanding.

The reaction rate r in Equation (7) can be expressed by Equations (4) to (6). However, since Equation (4) represents a normalized concentration distribution, it is necessary to multiply it by the actual initial precursor concentration C_0 to obtain the actual concentration. The initial precursor concentration C_0 of MTS can be calculated from the partial pressure of MTS using the ideal gas equation of state, and is given by

$$C_0 = \frac{n}{V} = \frac{P}{RT} \quad \dots\dots\dots (8)$$

(n : amount of precursor substance in mol). The resulting expression is related to the Hertz-Knudsen equation.

Figure 6 shows the calculated reaction rate r at the deepest region ($x = 5$ mm) for a preform thickness of 10 mm, a temperature range of 900 to 1,400°C, and a total pressure range of 1 to 100 Torr (the reaction rate is normalized by the maximum value). In general, many studies have reported that deposition in the MTS/H₂ gas-based SiC-CVI method is conducted at total pressures of 1 to 10 Torr to improve infiltration⁽⁴⁾. However, depending on the deposition temperature, low pressure is not always optimal, and the optimum total pressure for improving infiltration was found to vary.

4. Verification using the preform

This chapter presents the results of verifying the findings of the simplified analyses described up to **Chapter 3** through deposition experiments using an actual preform. For the deposition experiments, a fully heated hot-wall tubular reactor was used. The preform was placed at the position corresponding to the center of the electric furnace for deposition. A preform thinner than that assumed in the calculations was used.

Figure 7 shows the deposition amounts (film thickness on the fibers) and SEM images of the deepest region for SiC deposition within the preform by CVI at deposition temperatures of 1,000 and 1,200°C and total pressures of 5, 20, and 50 Torr. When comparing 1,000°C and 1,200°C at 50 Torr, the reaction rate at 1,200°C is higher; however, this trend is opposite to that shown in **Fig. 6**. This can be attributed to the fact that the simplified calculations did not take into account thermal decomposition of the MTS precursor or its consumption during deposition. According to the gas analysis results obtained during the experiments, several tens of percent of the MTS precursor remained even at the outlet of the reaction tube at 1,000°C, whereas the precursor was completely consumed at 1,200°C. Therefore, when precursor consumption is taken into account, the actual precursor concentration distribution inside the reactor is considered to differ significantly between these two conditions. When comparing the deposition amounts at 5, 20, and 50 Torr at 1,200°C, the deposition amount at 20 Torr (2.66 kPa) was the largest. Although the absolute values differ from those in **Fig. 6**, the tendency that an optimum total pressure condition exists is consistent.

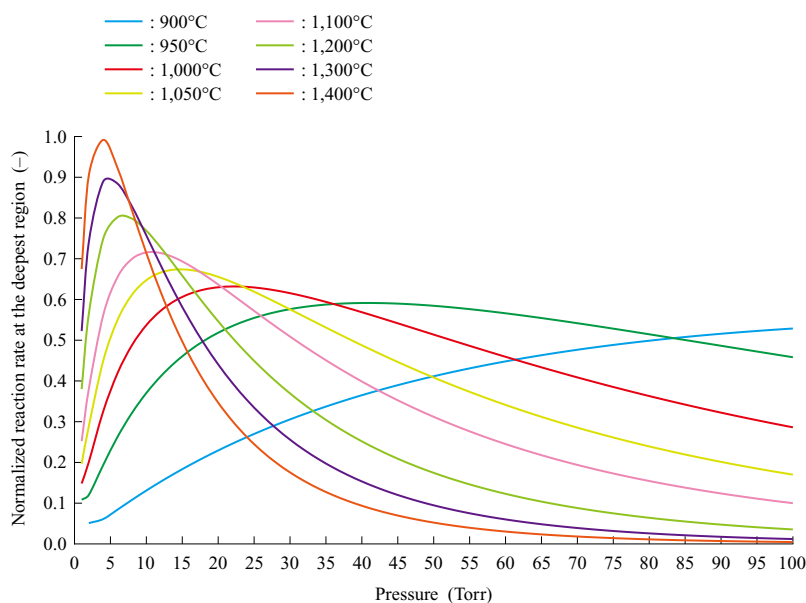
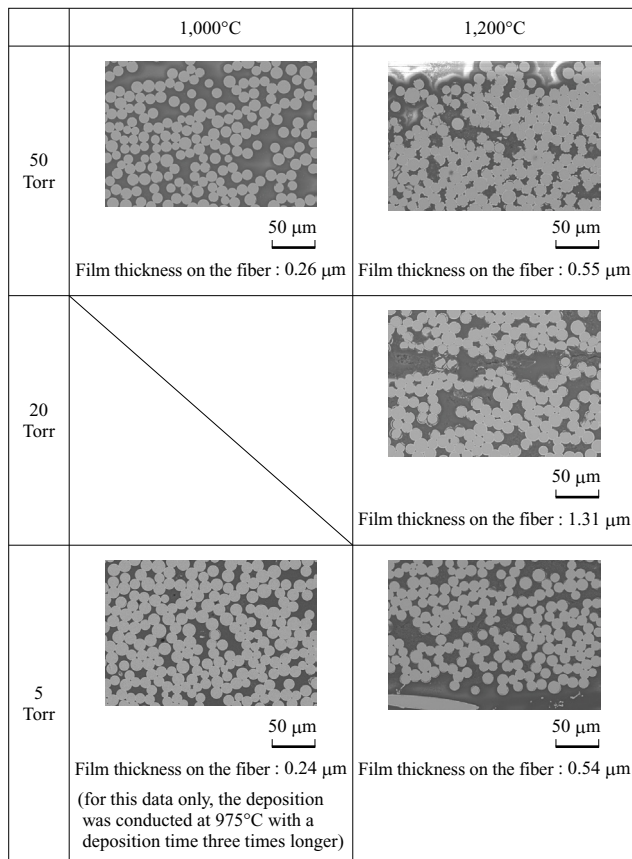
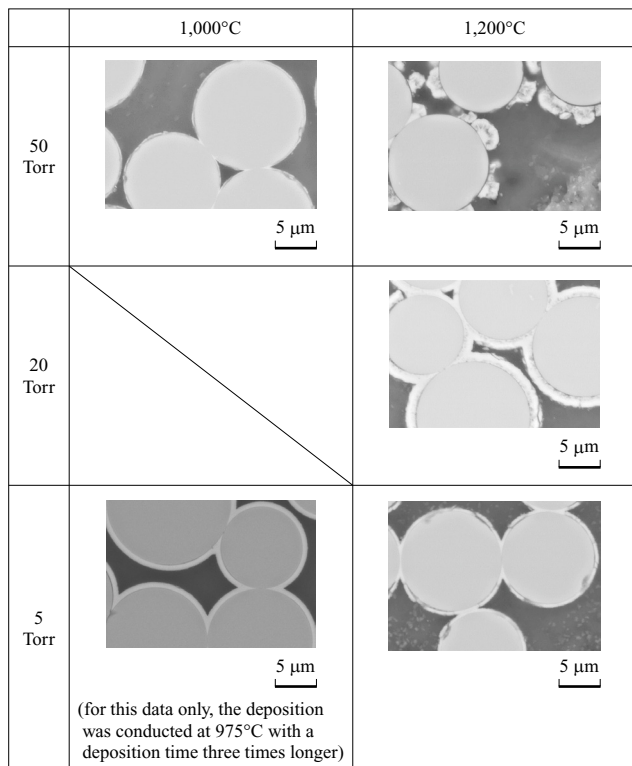


Fig. 6 Variation in normalized reaction rate at the deepest region of the preform with pressure and temperature



(a) SEM image of the preform cross section : 500x magnification



(b) SEM image of the preform cross section : 5,000x magnification

(Note) The film thicknesses on the fibers in (a) are shown as representative values for a constant deposition time.

Fig. 7 CVI growth depth in deepest cross-section of preform

5. CFD analysis of the CVI method

As described in **Chapter 4**, the phenomena in the reactor are greatly affected by precursor consumption and gas flow. Although simplified calculations are useful for obtaining an overall perspective, it is difficult to predict the deposition behavior accurately. Therefore, based on the diffusion concepts described above and the existing reaction mechanisms^{(4), (5)}, computational fluid dynamics (CFD) analysis was conducted using commercial CFD software.

5.1 CFD analysis method

The purpose of the CFD analysis is to understand the effects of gas flow on the deposition behavior. To achieve this, it is necessary to determine the macroscopic distribution of chemical species, in addition to the flow and temperature fields in the reactor.

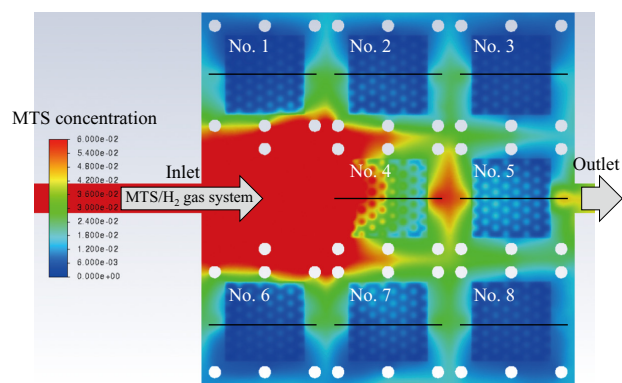
Because the preform affects the flow field inside the reactor, it is necessary to take its shape into consideration. In contrast, the analysis of the chemical reaction processes occurring inside the yarns that constitute the preform requires a fine mesh, resulting in a high computational load. Therefore, in this analysis, gas diffusion into the preform was considered, whereas the chemical reaction processes occurring inside the yarns were not explicitly modeled, on the assumption that they are governed by macroscopic conditions, thereby reducing the computational load.

In the CVI method, the gas flow velocity inside the reactor is set sufficiently low; therefore, the flow can be regarded as laminar without generating turbulence. The temperature distribution in the reactor is determined mainly by convection and radiative heat transfer. For the chemical reactions, the calculations were performed considering both the gas-phase reactions caused by the thermal decomposition of MTS and the surface reactions in which the pyrolysis products result in SiC deposition on the substrate surface. The reaction temperature and total pressure were set to 950°C and 10 Torr, respectively.

5.2 CFD analysis results

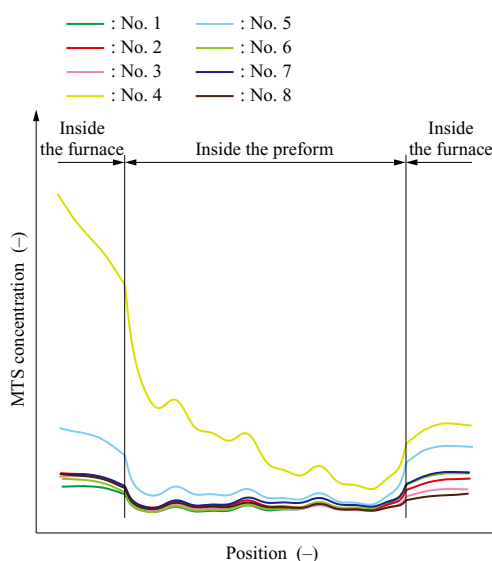
In the simplified analyses up to **Chapter 3**, the microscopic and macroscopic structures of the yarns and the preform were examined in detail. However, CFD analysis requires simultaneous calculation over a wide range of scales, from 1 μm gaps within the yarns to a 1 m-scale reactor. Therefore, even with current supercomputers, there are limitations on the mesh resolution if the calculations are to be completed within a practical computation time (for example, within one month). Thus, while taking into consideration the findings described up to **Chapter 3**, the computational load was significantly reduced by introducing the following additional approximations: the diffusion coefficient D (m²/s) of each chemical species was taken as the molecular diffusion inside the reactor, and the preform was assumed to have a structure with uniform pores without considering the yarn structure, with diffusion in the preform represented using the Knudsen diffusion coefficient.

Figure 8 shows the MTS concentration distribution and the SiC deposition rate distribution obtained from the CFD

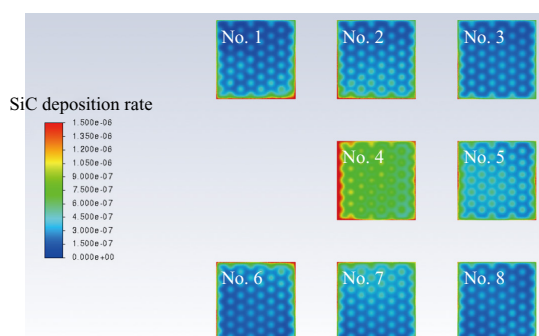


(Note) The black line in each preform indicates the cross-sectional profile shown in (b).

(a) MTS concentration distribution inside the reactor



(b) MTS concentration distribution in each preform cross-sectional profile



(c) SiC deposition rate distribution inside each preform

Fig. 8 MTS concentration distribution and SiC deposition rate distribution in preforms placed in the reactor

analysis for the case in which eight preforms were placed inside the reactor. First, the MTS concentration distribution is shown in **Fig. 8-(a)**. The cross-sectional profiles of the MTS concentration within each preform are shown in **Fig. 8-(b)**. The MTS concentration exhibited different distribution patterns among the preforms. In particular, it

was found that the concentration gradient became large in preform No. 4, which was strongly affected by the flow from the precursor inlet. This gradient is caused by the progress of the decomposition reaction as the gas flows around the preform, or by mixing with gas entering from other paths. These results suggest that the diffusion of MTS into the preforms strongly depends on the flow field. Although small peaks were observed at several locations in the concentration distribution within the preforms, these were caused by the influence of the preform-holding jigs.

Figure 8-(c) shows the CFD analysis results of the SiC deposition rate distribution within the preforms. In the calculation, it was assumed that SiC deposition proceeded with a constant surface deposition rate once MTS reached the SiC fiber surfaces. For preform No. 4, which is strongly affected by the inlet gas flow, the deposition rate distribution generally corresponds to the MTS concentration distribution. In the other preforms as well, the deposition rate varies along the flow direction of the precursor gas.

Although the details are omitted in this paper, deposition behavior was predicted by considering the effects of gas flow, which are difficult to estimate using simplified analyses alone, based on a model incorporating both microscopic and macroscopic structures. Quantitative comparison with experimental results confirmed that the predictions reproduced the experimental values within a factor of a few.

6. Conclusion

This paper introduced the basic concepts of the CVI method, which is one of the fabrication methods for CMCs. The CVI method for fabricating CMCs involves a reaction system in which microscopic and macroscopic scales interact in a complex manner, and an understanding of the phenomena from a kinetic perspective is essential.

In this paper, diffusion and reactions within the preforms were discussed through simplified analyses using approximations in order to obtain a clear understanding of the CVI method for fabricating CMCs. As a result, it was shown that this approach can explain the trends observed in the deposition behavior.

In addition, it was shown that CFD analysis can evaluate the effects of precursor consumption, gas flow, reactor geometry, temperature distribution, and other factors that cannot be fully considered through simplified analyses alone on film deposition, and that it is an effective means for assessing the validity of new process design.

CFD is expected to accelerate CVI process development as a detailed preliminary analysis tool for addressing the demands of various production sites. However, at present, the computation time remains long, and the quantitative agreement is still limited because microscopic structures cannot yet be fully taken into consideration. In the future, we aim to accelerate the development of the CVI method by improving gas-phase and surface reaction mechanisms suitable for CFD analysis and by reducing the computational load.

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