Morphological Control of Silicon Carbide Deposited on Hi-Nicalon Type S Fiber Using Atmospheric Pressure Chemical Vapor Infiltration

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ABSTRACT: Silicon carbide coated onto Hi-Nicalon Type S fiber is of great interest to the aerospace industry. This work focuses on tuning the reaction parameters of atmospheric pressure SiC CVI using CH₃SiCl₃ to control the morphology of the coatings produced. Depth of CH₃SiCl₃ from 1 to 14 cm, temperature from 1000 to 1100 °C, and flow rate of H₂ carrier gas from 5 to 30 SCCM were examined. Coating morphologies ranged from smooth to very nodular, where spherical growths were present along the entire deposition zone. The parameters that yielded a smooth deposition throughout the 20 cm deposition zone were 4–6 cm of CH₃SiCl₃(l) depth, 1100 °C, and 10 SCCM of H₂ as a carrier gas. Tensile testing using acoustic emission sensors was performed on SiCf/BN/CVI-SiC minicomposites with different coating morphologies. The tensile tests revealed that smooth coatings have better mechanical performance than the nodular coatings; nodular coatings promote premature ultimate brittle failure, while smooth coatings exhibit toughening mechanisms. Smooth coatings had higher average matrix cracking strength (248 MPa) and ultimate tensile strength (541 MPa) than average nodular coating matrix cracking strength (147 MPa) and ultimate strength (226 MPa).

INTRODUCTION

High operating temperature, low density, high hardness, chemical inertness, and retaining these properties at high temperatures are what make ceramic matrix composites (CMC) attractive for extreme environments such as hot sections of gas turbines, nuclear reactors fuel cladding, and space transportation.¹⁻⁴ The network of covalent or ionic bonds responsible for the positive properties of ceramic materials also leads to undesirable brittleness and low toughness of monolithic ceramics. A CMC is designed as a three-part system to enhance the toughness, durability, and safety as well as retaining all of the favorable properties of monolithic ceramics. The continuous phase of the composite is the matrix. Embedded in the matrix are high strength ceramic fibers that act as reinforcement for the matrix. If the matrix and fibers are too strongly bonded, the composite will behave as a brittle monolithic ceramic.⁵ The addition of the third component, the interphase, weakly bonds the fiber to the matrix. This allows toughening mechanisms such as fiber debonding and pullout, crack deflection, and crack splitting to take place.⁶ A variety of materials are available for fiber, interphase, and matrix composition.⁷⁻¹⁰ Silicon carbide (SiC) is an attractive matrix material due to high mechanical strength, creep resistance, melting point, hardness, and chemical inertness.¹¹

A single fiber-tow SiCf/SiC minicomposite can be considered the basic architectural feature of woven and laminate ceramic matrix composites (CMCs). It consists of a single SiC fiber tow coated with a thin layer (typically <1 μm) of boron nitride (BN) interphase and an overlay coating of silicon carbide matrix. They are easier to produce than a full composite, and the properties have been shown to be representative.¹² In a previous study, SiC/SiC minicomposites were produced at atmospheric pressure with a porosity below 10% and good coating uniformity within a 20 cm deposition zone using chemical vapor infiltration (CVI).¹³ However, further examination of the surface of the fibers showed variability in the morphology of the SiC coatings (matrix), ranging from smooth to very nodular. Several experimental studies have been conducted on the structure and morphology of CVI/CVD (chemical vapor deposition) SiC, but they used high deposition temperatures (>1100 °C), low pressures (<500 Torr), and nonfiber substrates.¹⁴⁻¹⁶ The deposition kinetics of the CH₃SiCl₃ (methyltrichlorosilane)/H₂ (hydrogen) system are temperature sensitive and switch between surface reactions and mass transport limited regimes.¹⁷⁻¹⁹

The goal of the current work is to establish a relationship between the reaction parameters of temperature, flow rate, and bubbler depth to the morphology of the SiC coatings. This is
to be accomplished by varying all of the above parameters and then examining the resulting coating (SiC matrix) using scanning electron microscopy (SEM). Experiments will be run at atmospheric pressure, and Hi-Nicalon Type S SiC fiber will be the substrate for all depositions. Temperature is varied between 1000 and 1100 °C, flow rates of H2 carrier gas are varied between 5 standard cubic centimeters per minute (SCCM) and 30 SCCM, and the depth of CH3SiCl3(l) bubbled through ranged from 14 to 1 cm. SiC/BN/CVI-SiC minicomposites are produced with 25 vol % fiber content, 200 nm BN interphase, and 65 vol % CVI-SiC matrix. The different morphologies are tested in monotonic tension to failure with the acoustic emission damage monitoring technique. The mechanical and acoustic emission results and fracture surfaces are analyzed to determine the effects of coating morphology on the initiation and progression of damage as well as the ultimate failure of the fabricated minicomposites.

**RESULTS**

**Depth Study.** The depth of CH3SiCl3(l) is defined in these experiments as the distance from the bottom of the stem to the meniscus of CH3SiCl3(l) when the stem contains no CH3SiCl3(l). All of the depth experiments had a flow of 10 SCCM H2(g) and a temperature of 1050 °C. Experiments began at a depth of 14 cm of CH3SiCl3(l) filling the bubbler to just below the elastomer ring seal. Depth experiments were conducted until the CH3SiCl3(l) meniscus was even with the bottom of the stem. Each reaction was run until 1 cm of CH3SiCl3(l) depth inside the bubbler was consumed. Each of the micrographs shown in Figure 1 is from the 10th cm of a 20 cm deposition zone for a different CH3SiCl3(l) depth, but the full 20 cm were imaged for each depth. The chosen micrographs from the center of the deposition zone are representative of the majority of the coating for each respective depth. Coatings were deposited directly onto Hi-Nicalon Type S SiC fiber. A micrograph summary of the effect of depth on coating morphology can be found in Figure 1. The first few cm of the full bubbler produced nodular coatings throughout the entire deposition zone. This shifted once the depth reached 11 cm, where the coatings went from nodular, having large protruding spheres, to bumpy: where the spherical growth is still present but not as extreme. This bumpiness continued to decrease as the depth of CH3SiCl3(l) continued to decrease. The coatings were almost entirely smooth throughout the entire deposition zone when the CH3SiCl3(l) depth got to the 6 cm mark, and the coatings stayed smooth as the bubbler continued to empty. At a depth of 3 cm of CH3SiCl3(l), the coatings did not return to being nodular, but did become noticeably rougher. The slight roughness stayed relatively the same as the bubbler emptied to the stem.

**Temperature Study.** The full 20 cm deposition zone is pictured for all of the temperatures tested (Figure 2). Each micrograph is spaced approximately 2.54 cm from the previous micrograph. All micrographs for groups A, B, and C are from the same reaction. The scale bar applies to all micrographs in the figure.
smooth near the inlet but gradually became rougher further into the deposition zone, eventually becoming nodular in the last two micrographs pictured. Increasing the flow to 20 SCCM provided a deposition zone trend similar to the 5 SCCM deposition; the first few micrographs show smooth coatings, but they became rougher further from the gas inlet. The major difference between those runs is that the coatings in the rear of the deposition zone were significantly more nodular than the 5 SCCM run. Further increasing the flow to 30 SCCM had a similar trend as the previous two flow rate experiments: the first few micrographs indicate smooth coatings that became rougher further from the gas inlet. Severe nodular growth much sooner in the 30 SCCM experiment, with prominent spherical growths as soon as the fifth micrograph.

**Mechanical Testing.** Minicomposite samples were tensile tested at NASA to understand the stress-dependent damage initiation and progression for two batches of minicomposites. Minicomposites characteristics and test results are summarized in Table 1. The evolution of normalized acoustic emission energy of each tested minicomposite from batch 1 (smooth) and batch 3 (nodular) is plotted as a function of the applied tensile stress in Figure 4. The plot reveals that the two different batches of material produced significantly different mechanical testing results, despite having similar fiber volume fractions and the same 200 nm BN interphase. Minicomposites in batch 1 had smooth coatings, while batch 3 had nodular coatings. The average SiC matrix cracking strengths of batch 1 and batch 3 were 248 and 147 MPa, respectively. The average ultimate tensile strength (UTS) of batch 1 and batch 3 were 541 and 226 MPa, respectively. Thus, it is proposed that minicomposites from batch 3 exhibit lower SiC matrix cracking strength and ultimate tensile strength due to the rough, nodular morphology.

**Fracture Surface Differences.** Examining the fracture surfaces of the batch 3 and batch 1 minicomposites with nodular and smooth coatings, respectively, after tensile testing revealed different coating microstructures (Figure 5). Smooth coating specimens exhibit columnar growth throughout the coating. Nodular coating specimens exhibit some degree of columnar growth, but the nodules themselves have a disordered, clustered grain morphology. The fracture surfaces also revealed differences in composite failure behavior. Nodular coating samples showed little fiber pullout, while the smooth coating samples did exhibit fiber pullout (Figure 6) and significantly higher UTS.

**DISCUSSION**

In this work, the effects of CH$_3$SiCl$_3$ depth in a bubbler, reactor temperature, and flow rate of H$_2$ through CH$_3$SiCl$_3$ on the effect of SiC morphology when deposited via CVI onto Hi-Nicalon Type S SiC fiber were studied. The mechanical strengths and microstructural differences between minicompo-

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![Figure 3: SEM micrographs of SiC deposition at (A) 5 SCCM H$_2(g)$, (B) 20 SCCM H$_2(g)$, and (C) 30 SCCM H$_2(g)$. The temperature was 1100 °C and CH$_3$SiCl$_3(l)$ depth was 6 cm for all flow rates. The numbers in the upper-right corner indicate the order of micrographs from the gas inlet, where 1 is closest to the gas inlet. Each micrograph is 2.54 cm from the previous micrograph. All micrographs for groups A, B, and C are from the same reaction. The scale bar applies to all micrographs in the figure.](https://dx.doi.org/10.1021/acsomega.0c03493)
sites with nodular versus smooth SiC coatings were also examined.

The reactive gas concentration in the furnace was affected by the depth of CH$_3$SiCl$_3$(l): deeper CH$_3$SiCl$_3$(l) provided a longer path for the H$_2$ carrier gas to travel through. This longer path provided more time for H$_2$(g) to become saturated in CH$_3$SiCl$_3$(g) to be carried into the reactor, effectively increasing the reactive gas concentration in the reactor. A similar effect would occur from a higher flow rate of H$_2$(g) through CH$_3$SiCl$_3$(l): more CH$_3$SiCl$_3$(g) would enter the reactor per unit time than with a lower flow rate, which would provide a greater reactive gas concentration. At a high enough reactive gas concentration, vapor phase precipitation may occur. The vapor phase precipitant will then impact other gas molecules when traveling toward the substrate, resulting in growth. Eventually, the enlarged particle will land on the surface of the substrate, creating a rough morphology. This region will continue to retain a rough appearance, even if a smooth coating forms over it. Also, other vapor phase precipitants will continue to form, deposit, and perpetuate this cycle.

The gas concentration argument is centered on molecules reacting in the gas phase, so as the temperature increases you could expect more molecules to react in the gas phase to precipitate, leading to a nodular coating. However, the opposite of this was observed, where higher temperatures produced a smoother coating over a longer distance in the reactor deposition zone. This is due to the heightened temperature increasing the rate of gas molecules reacting with the surface of the substrate and forming nucleation sites where more deposition can easily occur. At low temperatures, not every molecule that interacts with the surface will react to form SiC. Higher temperatures increase the number of molecules that react when they are bound to the surface of the substrate. This leaves more nucleation points closer together on the substrate surface. SiC deposition is an autocatalytic process, where the material initially nucleates on a surface and then grows faster off of the nucleation sites than off the substrate. Nucleation sites spaced further apart would promote increasingly prominent spherical growth due to the autocatalytic nature of the growth. Thus, this occurs more readily at lower temperatures.

Temperature and gas concentration influenced morphological changes along the entire length of the deposition zone. In some cases, the deposition zone started nodular and became smooth, or started smoothly and steadily became more nodular. A smooth to nodular coating transition was present in all of the experiments determining what effect flow rate of H$_2$(g) through CH$_3$SiCl$_3$(l) had on the deposition morphology. This is due to the development of an increasingly thick boundary layer in combination with the autocatalytic growth of CVI-SiC. The kinetics of atmospheric pressure CVI reactions are controlled by diffusion of the gas through a boundary layer of laminar flowing gases above the substrate. The gas diffuses through the boundary layer, adsorbs to the substrate, chemically reacts to form products and byproducts, then the byproducts desorb from the substrate and diffuse back through the boundary layer and into the main flow of gas. Further from the gas inlet, the byproducts increasingly contribute to the thickness of the boundary layer until the rate of diffusion and formation of byproduct establishes an equilibrium that makes the boundary layer thickness constant. This promotes nodular growth because there are fewer reactive molecules diffusing through the boundary layer to nucleate on the surface. This will have two effects that both promote nodular growth. The first is that the thicker boundary layer will restrict molecules from reaching the substrate and forming a nucleation site, leaving them more spaced out which promotes nodules. The
second effect combines the autocatalytic nature of this process with the thicker boundary layer. After a nucleation site forms, not only does this provide a thinner boundary layer necessary for the molecule to diffuse through when compared to the substrate, but also a more favorable place for the molecule to react due to the autocatalytic nature of this reaction.

The opposite trend occurred during the 1000 °C temperature study, where the first two micrographs are extremely nodular but further from the gas inlet; the coating becomes smoother. This is due to the CH3SiCl3(g) cracking, or thermally decomposing into its reactive gas form, later in the furnace at the lower temperature, creating a momentarily high concentration of reactive gas at the cracking point that leads to nodule formation. A thermal profile of the tube furnace reveals an isothermal zone in the center of the furnace and a sharply dropping temperature gradient closer to the ends of the furnace. The only change present for different temperatures is that the entire thermal profile would shift. This will effectively change what depth in the furnace CH3SiCl3(g) reaches the cracking point. First, the CH3SiCl3(g) cracks, producing a high concentration of reactive gas molecules. These molecules will react with each other in the gas phase and with the surface of the substrate, quickly forming a nodular SiC coating. This decreases the reactive gas concentration as the remaining molecules continue further into the reactor. Each molecule that reacts is then going to produce three molecules of HCl(g) byproduct that will then enter the gas stream, diluting the reactive gasses and inhibiting the formation of soot, promoting smooth coatings further from the gas inlet.

Stress-dependent damage initiation and progression and ultimate failure of minicomposites from batch 1 is comparable to that of CVI-SiC/BN/Hi-Nicalon Type S minicomposites with similar fiber volume fraction that were processed by Rolls-Royce and studied previously. The tensile fast fracture behavior of minicomposites from batch 3 is very different from that of batch 1 minicomposites. Minicomposites from batch 3 started cracking at an average of 100 MPa lower tensile stress than batch 1 minicomposites. This is due to the existence of surface nodules on minicomposites from batch 3, which acted as surface defects and local stress raisers. Thus, stress concentration at the nodules is expected to be higher in comparison to the smooth surface of minicomposites from batch 1. In addition, there is likely a difference in the density between the CVI-SiC with nodules (batch 3) and without nodule (batch 1). If the density of CVI-SiC in batch 1 is greater than that of CVI-SiC in batch 3, one should expect the batch 3 CVI-SiC matrix to have lower strength. Furthermore, the ultimate tensile strength of the batch 3 minicomposites was significantly lower (by an average of 215 MPa) than that of batch 1. Strong adhesion between the rough and nodular CVI-SiC matrix and the BN interphase reduced or prevented crack deflection at the CVI-SiC and BN interphase, and the batch 3 minicomposites showed reduced toughness with premature brittle ultimate failure. The BN interphase was somewhat effective as multiple CVI-SiC cracks formed before the ultimate minicomposite failure.

■ CONCLUSIONS

The effects of CH3SiCl3(g) depth, reactor temperature, and H2 carrier gas flow rate on the morphology of SiC coating/matrix deposition have been examined. As CH3SiCl3(g) depth decreased, the coatings became smoother. Increasing the temperature from 1000 to 1100 °C makes the overall coatings within the deposition zone become smoother. Increasing the flow rate from 10 to 30 SCCM produced nodular coatings in the rear of the deposition zone. The onset of nodular growth occurred sooner in experiments where the flow rate was higher. Decreasing the flow to 5 SCCM produced a slightly rough coating in the rear of the deposition zone, but the nodular growth was not as severe as the 20 SCCM experiment. Nodular growth reduced the stress at the onset of CVI-SiC matrix cracking in SiC/BN/SiC minicomposites due to the increase of surface defects that increased the local stress concentration in the vicinity of the surface nodules. In addition, the rough surface of CVI-SiC reduced the ultimate tensile strength of SiC/BN/SiC minicomposites due to the strong bonding between the CVI-SiC matrix and the BN interphase; that strong bond reduced overall minicomposite toughness and caused the premature brittle failure. Specimens with smooth coatings exhibit a columnar grain fracture surface while nodules do not. Conditions that yielded a smooth coating throughout a 20 cm deposition zone were 4–6 cm of CH3SiCl3(g) depth, 1100 °C, and a flow rate of 10 SCCM H2 carrier gas. The mechanical behavior of minicomposites that have smooth CVI-SiC coating was comparable to the results reported previously with an average matrix cracking stress of 248 MPa and ultimate tensile strength of 540 MPa.

■ EXPERIMENTAL SECTION

Materials. Methyltrichlorosilane (CH3SiCl3, 98%) (Gelest, Inc.), ultrahigh purity nitrogen and ultrahigh purity hydrogen (Airgas, Inc.), boron trichloride (>99%), and anhydrous ammonia (>99%) (Praxair Technology Inc.) were used without further purification. The Hi-Nicalon Type S SiC fiber tow (NGS Advanced Fibers Co., Ltd.) has a 500 filament count, and each filament is approximately 12 μm in diameter. The fibers have a 0.5–2.0 weight percent poly(vinyl alcohol) sizing. The carbon fiber used was THORNE T-300 (Amoco/ Cytce Engineered Materials) with a filament count of 3000 and an average filament diameter of 7 μm. It was supplied with 1% UC309 epoxy compatible sizing. The fibers were not modified before being placed in the reactor.

Reactor Setup and Sample Mounting. The same reactor setup and sample mounting were used for all experiments (Figure 7). First, a piece of graphite foil is cut to fit the length of the 47 mm inner diameter, 50 mm outer diameter fused silica tube. Five graphite mounts are then attached to the foil using double-sided carbon tape, as in Figure 7B. The positions of the graphite mounts are determined by conducting a deposition with only the graphite foil in the furnace to determine the deposition zone. The
outermost pieces of tooling are placed on the ends of the tube to ensure that the fiber tows will remain taught throughout the experiment. The next two innermost pieces of tooling are placed on either end of the deposition zone; the carbon tape on these pieces is used solely to secure the tows during mounting. The final piece of tooling is placed in the center of the deposition zone to prevent the sample from sagging. Next, two lengths of SiC fiber tow are cut to the length of the fused silica tube. Both of the fiber tows are then taped to the first piece of tooling with carbon tape. The tows are then pulled tight over the remainder of the pieces of tooling and secured with carbon tape. The graphite foil is then rolled and carefully placed into the fused silica tube, ensuring that all of the mounts are properly aligned.

**BN Interphase Coating.** Boron nitride was deposited onto as-received Hi-Nicalon Type S SiC fiber tow using low-pressure CVI. The BN deposition reaction was 125 SCCM BCl₃(g) reacting with 400 SCCM NH₃(g) at 800 °C for 24 min at 8 Torr diluted in 400 SCCM N₂(g). Fiber tows were tied onto a graphite mandrel using T-300 carbon fiber. The fused silica tube for this reaction had an inner diameter of 20 cm and was 122 cm long.

**SiC Matrix Coating.** Purging with N₂(g) begins after the tube is loaded into the reactor as in Figure 7A and is done for a minimum of 12 h to remove ambient oxygen and moisture. The reactor is then heated to the target temperature at a rate of 20 °C/min, while N₂(g) continues to flow. Once the reactor reaches the temperature and thermally equilibrates, the N₂(g) flow is switched off and a metered flow of H₂(g) flowing through a bubbler containing CH₃SiCl₃(l) is switched on. The thermal decomposition of CH₃SiCl₃(l) to deposit SiC(μ) is a well-known reaction. All reactions were run until the height of CH₃SiCl₃(l) in the bubbler was lowered by 1 cm: approximately 20 h of processing time, which produced a 14 μm thick coating. The height of CH₃SiCl₃(l) is defined as the distance between the bottom of the stem where carrier gas flows to the top of the CH₃SiCl₃(l) meniscus when the stem does not contain any CH₃SiCl₃(l) (Figure 8). Finally, the reactor is switched from H₂(g)/CH₃SiCl₃(l) back to N₂(g) and allowed to cool to room temperature.

**Minicomposite Fabrication.** SiCf/BN/CVI-SiC minicomposites (20 cm in length) were fabricated with 25 vol % fiber content, 200 nm BN interphase, and 65 vol % CVI-SiC matrix. Specimen batches with two different SiC coating morphologies were produced for the determination of mechanical strength and microstructure characterization.

**Mechanical Property and Microstructural Characterizations.** Monotonic fast fracture tensile loading was performed on minicomposites using Instron Frame model 5500R-4502 with a 500 N load cell and a displacement control loading rate of 0.127 mm/min. The volume fractions of fiber, matrix, and interphase of each specimen and specimens’ cross-sections were determined as described in previous work. The minicomposite stress was determined by dividing the applied tensile load by the calculated minicomposite cross-sectional area. Acoustic emission (AE) waveforms were recorded during specimen tensile loading using a Digital Wave Corporation four-channel fracture wave detector acquisition system and two B1025 piezoelectric AE transducers with 300–3000 kHz sensitivity. AE waveforms associated with events that were detected in the specimen gage section were filtered and analyzed using a software from the same company. For each AE event, there is a waveform and wave energy recorded by each sensor. AE energy from both sensors was averaged. The cumulative AE energy at each sequential event number and time was then calculated as a function of time and applied tensile stress as shown previously. The evolution of the normalized cumulative AE energy during fast fracture tensile testing is directly propositional and representative of the evolution of damage in ceramic matrix minicomposites.

Scanning electron microscopy (SEM) at UCONN was conducted using a Thermo Fisher Scientific Teneo LVSEM with an accelerating voltage of 5 kV. SEM of fractured surfaces was done at NASA with a Tescan MIRA3 with an accelerating voltage of 7 kV.

![Figure 8. Picture of bubbler filled with CH₃SiCl₃(l). The upper red bar indicates the meniscus level, while the lower red bar indicates the bottom of the stem. The distance between these two bars at any given time is defined as the depth of CH₃SiCl₃. The picture was taken by the authors and is a free domain.](https://dx.doi.org/10.1021/acsomega.0c03493)

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**Notes**

The authors declare no competing financial interest.

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