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基于流化床化学气相沉积的碳化硅材料制备、性能及其在核领域的应用

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摘 要：采用流化床化学气相沉积法(FB-CVD)制备了碳化硅包覆层，利用多尺度耦合模型对包覆过程进行了模拟，以提高包覆层的均匀性。开发了可应用在核能领域的碳化硅材料，如致密碳化硅层、细晶粒碳化硅包覆层、碳化硅/碳/硅混合包覆层、多孔碳化硅包覆层、碳化硅纳米线、碳化硅纳米粒子等。为了评估碳化硅包覆层的可靠性，进行了极端条件下的模拟事故氧化考验。对于以碳化硅为主要包覆层的燃料元件，在高通量核反应堆中进行了辐照试验，结果表明：该碳化硅包覆层材料在核应用中的表现优异。

关键词：碳化硅；涂层；流化床化学气相沉积；核能应用
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Preparation, Performance and Nuclear Applications of Silicon Carbide Materials Prepared by FB-CVD Method

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Abstract: Results regarding the fabrication of SiC coating layers and other SiC-related materials for nuclear reactor applications are reported herein. A fluidized bed chemical vapor deposition (FB–CVD) method was used to prepare the SiC coatings, and the coating process was simulated using the newly proposed multiscale coupling model to enhance the coating uniformity. Different kinds of SiC series products, such as dense SiC layers, fine-grained SiC coating layers, SiC/C/Si mixed coating layers, porous SiC coating layers, SiC nanowires and SiC nanoparticles, were developed for nuclear energy applications. Postulated accident conditions, such as air ingress, wereconsideredto evaluate the reliability of the SiC coating layer. An irradiation test was also performed in ahigh-flux nuclear reactor, and the results validated the high performance of SiC coating materials in nuclear reactor applications.

Keywords: silicon carbide; coating; fluidized bed chemical vapor deposition; nuclear application

1 Introduction

Silicon carbide (SiC) has been extensively studied because of its unique physical and chemical properties, such as good mechanical strength at elevated temperatures, excellent thermal stability and chemical durability, high electrical transport, strong field emission and good oxidation resistance[1–3]. Due to these excellent properties, SiC can be widely used in the fields of structural materials, semiconductor manufacturing, and photocatalysis[4–6]. Devices made with SiC can be operated at high temperatures, high powers, high frequencies, and in harsh environments. Furthermore, due to its low thermal neutron absorption cross section and good radiation resistance, SiC is also suitable for use in nuclear reactors[7].

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Among the different applications of SiC in nuclear reactors, its use as a coating material for so-called TRISO (tristructuralisotropic)-coated fuel particles has become increasingly important due to the growing interest of many countries in developing high-temperature gas-cooled nuclear reactors (HTGRs)[8–10]. In TRISO particles, SiC is the critical layer for hindering the release of fission products and provides crucial safety measures in the HTGRs[11–12]. TRISO-type fuel particles also show great potential applications in other nuclear systems, such as molten salt reactors (MSRs)[13], gas cooled fast reactors (GFRs)[14], and fixed bed nuclear reactors (FBRs)[15]. Recently, triplex SiC fuel cladding has been widely studied to replace the dominant zirconium cladding used in light water reactors (LWRs)[16–18]. In addition to applications involving nuclear fuels, SiC- and SiC-based composite materials also display increasingly promising applications as inert matrices, structural components, flow channel inserts, and thermal insulation in many fission or fusion energy systems[19–20].

In recent years, we have focused on SiC materials preparation and performance for nuclear applications using the fluidized bed chemical vapor deposition (FB–CVD) method. SiC materials with different microstructures, such as dense SiC layers, fine-grained SiC coating layers, SiC/C/Si composite coating layers, porous SiC coating layers, SiC nanowires and SiC nanoparticles, and numerical simulations of the FB–CVD particle coating process are studied. The present paper is organized as follows. The FB–CVD method is generally described in Part 2, and the simulation study of the FB–CVD particle coating process is briefly summarized in Part 3. In Part 4, different kinds of SiC materials produced for this publication are introduced. Finally, conclusions are drawn, and the possibility of SiC materials for nuclear applications is proposed.

2 FB–CVD method for the preparation of SiC materials

The FB–CVD method is a suitable technique for preparing various kinds of coating films and layers on the surface of substrate materials with spherical or irregular shapes by initiating chemical reactions in the gas phase[21]. FB–CVD has the advantage of a large reactor volume that has sufficient space for uniform mass and heat transfer conditions. This technique can be used for many purposes, and one of the well-known applications is for synthesizing carbon nanotubes[22–23].

A schematic diagram of the FB–CVD experimental apparatus is shown in Fig. 1. This experimental apparatus can be divided into two main parts: the left part is the gas distribution system, and the right part is the fluidized bed coating system. For the SiC coating process described herein, the gas distribution system contains argon, hydrocarbon gas (ethyne and propene), hydrogen, a liquid precursor (methyl trichlorosilane (MTS) and hexamethyldisilane (HMDS)) a vapor supply device, a flow control system and a gas distribution device. The fluidized bed coating system contains a spouted bed, cooling water system, heating system, thermal insulation system, gas airtight device and temperature control device.

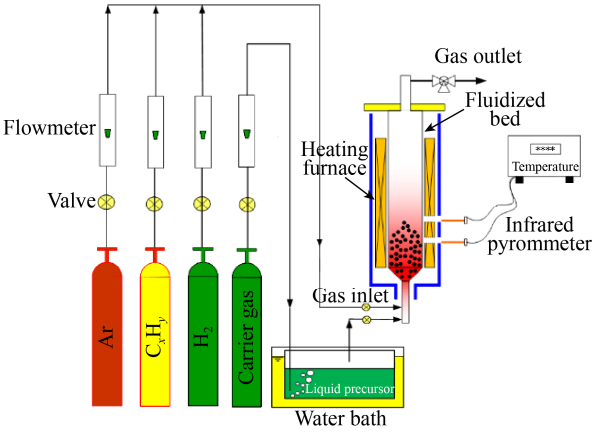


Fig. 1 Schematic diagram of the FB–CVD system

3 Simulation of the FB–CVD particle coating process

The simulation of the FB–CVD process focused on how to improve the gas-solid contact efficiency and coating layer uniformity. The particle movement behaviors, including the space-time distribution of particles and spout-fluid bed dynamics that were based on an Euler-Euler simulation, were studied first[24–25]. It was found that the specially designed multinozzle gas inlet was better than the single-nozzle gas inlet for obtaining an improved uniform fluidization that can disperse the gas to increase the gas-particle contact efficiency. We also established the DEM–CFD simulation platform[26]. The spout process was simulated based on this coupling method, and the particle spouting process in the different regions of the fluidized bed were obtained and analyzed.

Moreover, based on the experimental observation of the coating process, the population balance model (PBM) was used to describe the size distribution of nanoparticles in the FB–CVD process, and a nucleation-coalescence-breakup model was established based on the literature. Then, a deposition rate model that was related to the nanoparticle size distribution was developed based on the homogeneous and heterogeneous deposition coexistence mechanism. Furthermore, at the macroscopic scale, the computational fluid dynamics (CFD) and discrete element method (DEM) were

coupled with the deposition rate model, and finally, a multiscale simulation platform was established for simulating the FB–CVD particle coating process, as shown in Fig. 2.

For the FB–CVD particle coating process, the gas-to-particle process and nanoparticle evolution process were studied using the CFD–DEM method (Fig. 3). The nanoparticle cluster formation process and stability condition simulation were simulated under different conditions, such as chemical reaction-limited and diffusion-limited conditions. Based on the energy balance of nanoparticle clusters, a mesoscale model for describing the nanoparticle evolution process is proposed.

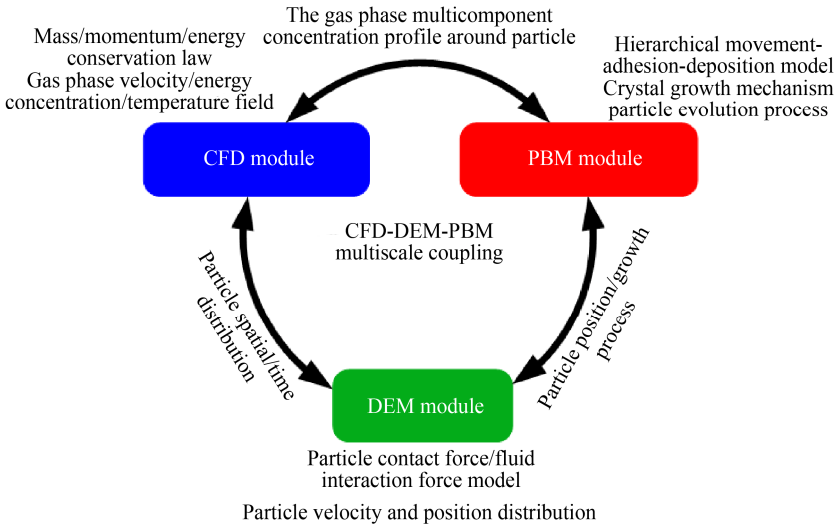


Fig. 2　The CFD–DEM–PBM model for simulating the particle coating process using the FB–CVD method[26]

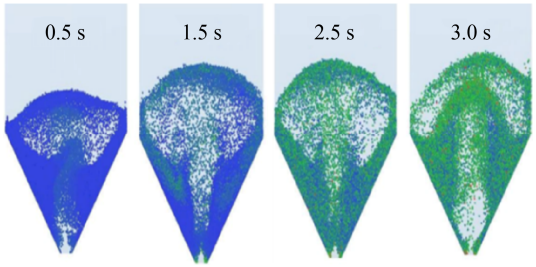


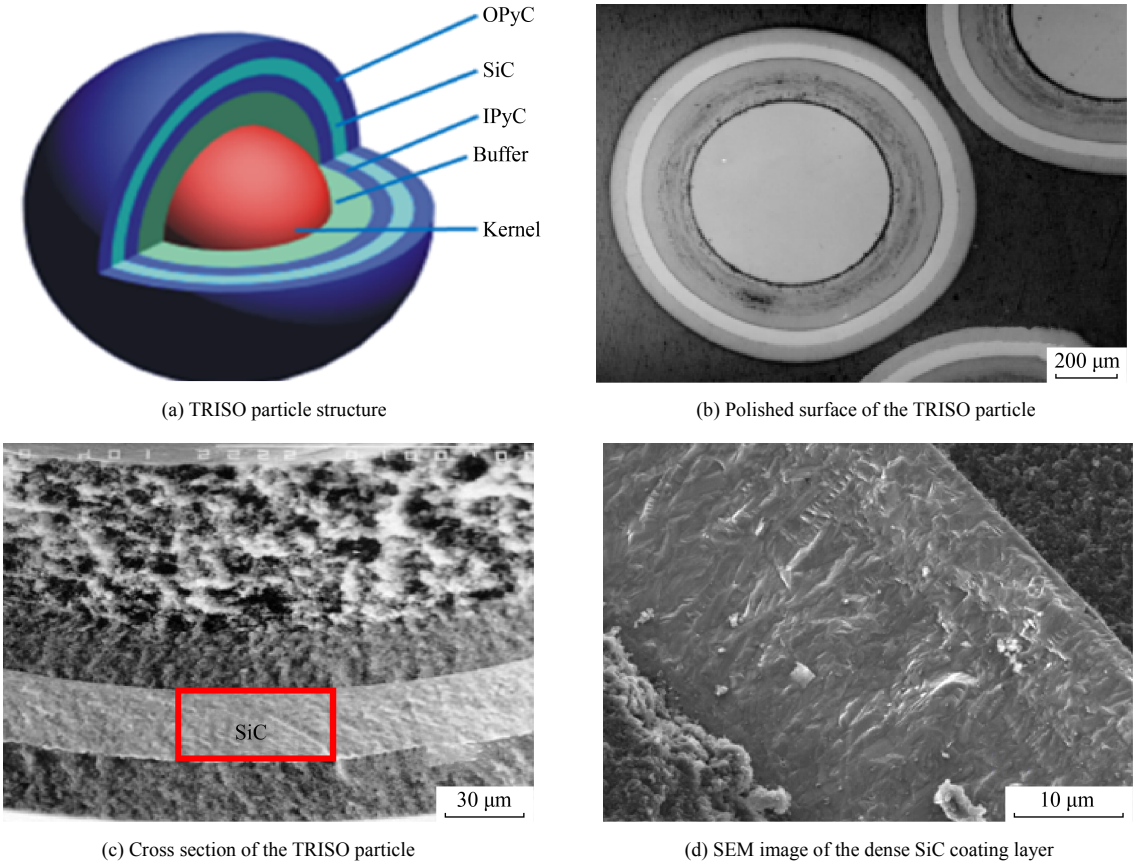
Fig. 3　Particle coating process simulation results using the CFD–DEM–PBM model (Blue indicates before coating and green indicates after coating)

4　Series of SiC materials prepared by the FB–CVD method used for nuclear fuel

4.1　Dense SiC coating layer on TRISO-coated fuel particles

The dense SiC coating layer on TRISO coated fuel particles is the key material in the fuel element of a high-temperature gas-cooled reactor (HTGR). For TRISO-coated fuel particles, the nuclear fuel kernels were coated by four functional layers, including three layers of pyrolytic carbon and one layer of SiC. Among the four layers, the SiC coating is considered the most important since it not only provides the TRISO particle with structural integrity but also is the primary layer for containing radioactive fission products at elevated temperatures. The PyC and SiC coatings were deposited on the kernels continuously by the FB–CVD process. A SiC layer with a thickness in the range of 25–50 μm was deposited by the slow decomposition of methyl trichlorosilane (MTS) using hydrogen as the fluidizing gas at 1 600 ℃[27]. Fig. 4 shows a schematic of the coated fuel particle and the microstructure of the SiC coating obtained in this study.

After deposition, SiC coatings with a density close to the SiC theoretical density (3.21 g/cm^3) were obtained. In real fission reactors, many kinds of gas and solid fission products are produced in the nuclear fuel kernels, and almost all of the fission products can be blocked by this SiC layer. Irradiation tests were also performed for the coated fuel particles. A total of 60 000 particles were irradiated in a high-flux reactor with the irradiation parameter much more severe than that of real rector conditions. The results showed that under a neutron flux of 4.95×10^{25} n/m^2 with a temperature of 1 150 ℃, none of the particles failed after 355 d of irradiation, which confirmed the excellent hindering properties of the SiC layer. This technology has been scaled up to industrial production, and a production line with a capacity of 12 600 000 particles per day was established[28]. The coated particles with high-density SiC by the FB–CVD method are planned to be used in a demonstrative high-temperature gas-cooled reactor in China in the near future.

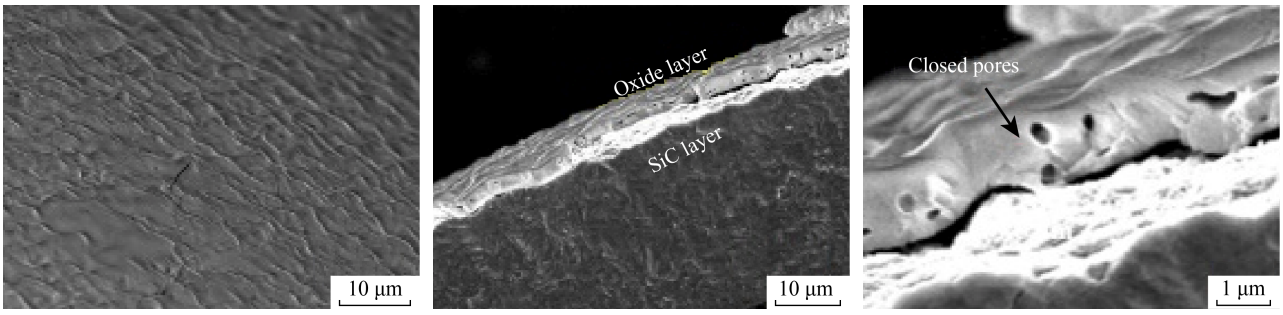


(a) TRISO particle structure (b) Polished surface of the TRISO particle

(c) Cross section of the TRISO particle (d) SEM image of the dense SiC coating layer

Fig. 4 Sketch map of the TRISO particles and microstructure of the SiC coating with a high density

Bide irradiation tests and many postulated accident conditions were also performed to evaluate the SiC performance, such as the anti-oxidation properties of the SiC layer[29–30]. The oxidation test showed that no obvious oxidation occurred below 1 000 ℃, and the thickness of the oxide layer increased slowly below 1 200 ℃. Under a very high oxidation temperature of 1 600 ℃, an oxide layer with a dense surface and a wrinkled crack-free morphology was formed, and closed pores were observed in the oxide layer (Fig. 5). The maximum oxide layer thickness obtained was much less than the thickness of the whole SiC layer, which indicated the robustness of SiC coatings under such postulated accident conditions.



(a) Surface (b) Fracture surface (c) Oxide layer with closed pores

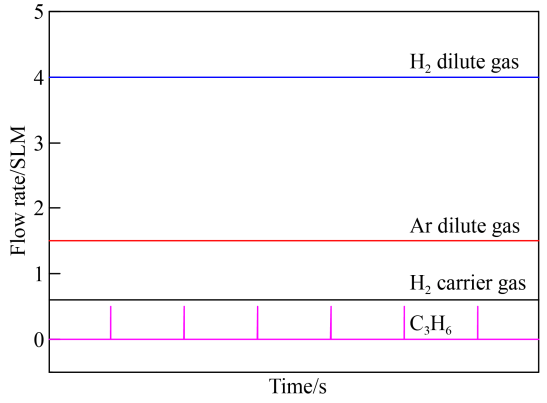
Fig. 5 SEM images of the SiC coating layer oxidized at 1 600 ℃ for 4 h[29]

4.2 Fine-grained SiC coating layers

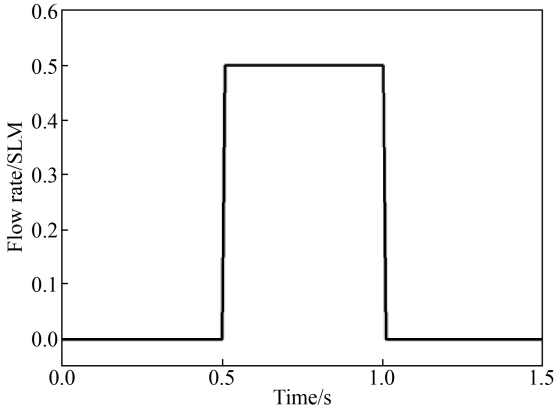
As discussed above, the preparation temperature of the high-density SiC layer was approximately 1 600 ℃. The SiC layer obtained at such high temperatures is usually composed of columnar grains with an average grain size larger than tens of micrometers. The growth direction of the columnar grains is usually parallel to the normal of the coating layer, with some extremely large grains penetrating the whole layer. Irradiation results indicated that most radioactive fission products could be hindered by this SiC layer except for silver because of the formation of large columnar grains. From previous studies, large columnar grains released 90% of the silver produced, whereas small grains

released only 30% of the silver[31].

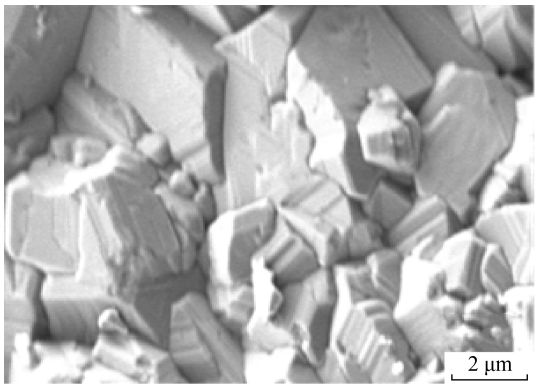
To prepare SiC with fine grains, we propose a novel method called the pulsed propylene method[32]. In this method, dilute propylene was injected into the furnace in a very short pulse at appropriate time intervals during the SiC deposition process (Fig. 6(a)). By adjusting the propylene concentration and pulse interval, a SiC layer with an average grain size of less than 300 nm was obtained, which was much smaller than the grain size obtained by the conventional method (Figs. 6(b)–6(c)). The key principle of the pulsed propylene method was the control of the nucleus formation during the SiC deposition process. The formation of fine grains was attributed to the promotion of the nucleation and interruption of the crystal growth of the SiC. In a designed sandwiched SiC layer (columnar grain/fine grain/columnar grain), the hardness of the fine-grained SiC was 10% higher than that of the SiC layer with columnar grains (Fig. 6(d)). The fine-grained SiC layer also showed much higher oxidation resistance than that of the large columnar-grained SiC layer. At the oxidation temperature of 1 600 ℃, the oxidation layer thickness was more than 2 μm for the conventional large columnar-grained SiC, but it was less than 1 μm for the SiC layer with an average grain size of 300 nm. This elevated oxidation resistance was attributed to the increased oxygen diffusion path present in the disordered grain boundaries of the fine grains.



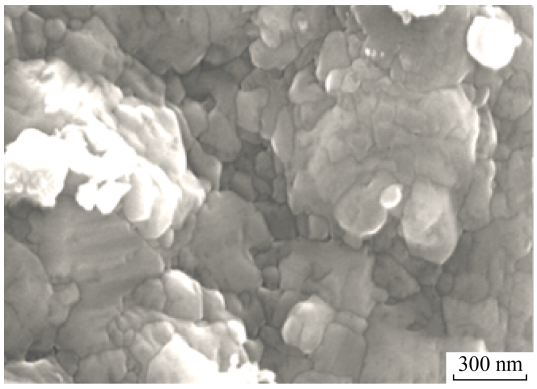
(a) Illustration of the pulsed propylene method



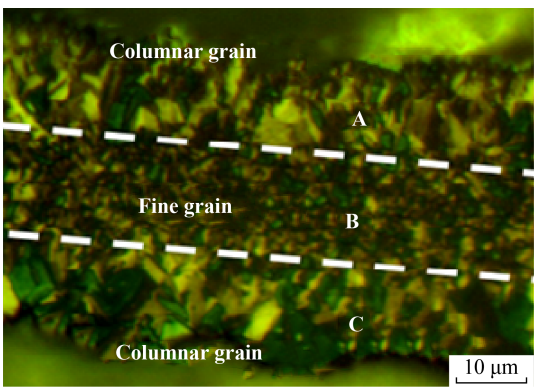
(b) A typical propylene pulse

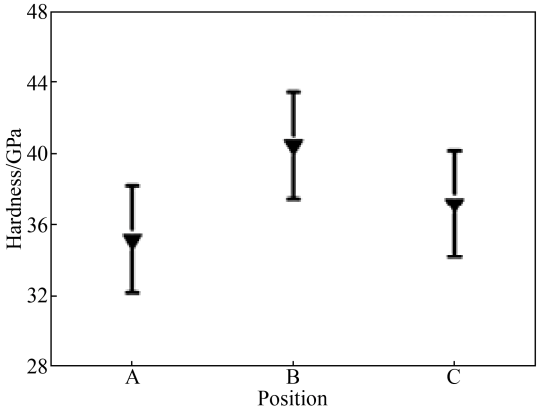


(c) SiC coatings by conventional method



(d) SiC coatings by the pulsed propylene method



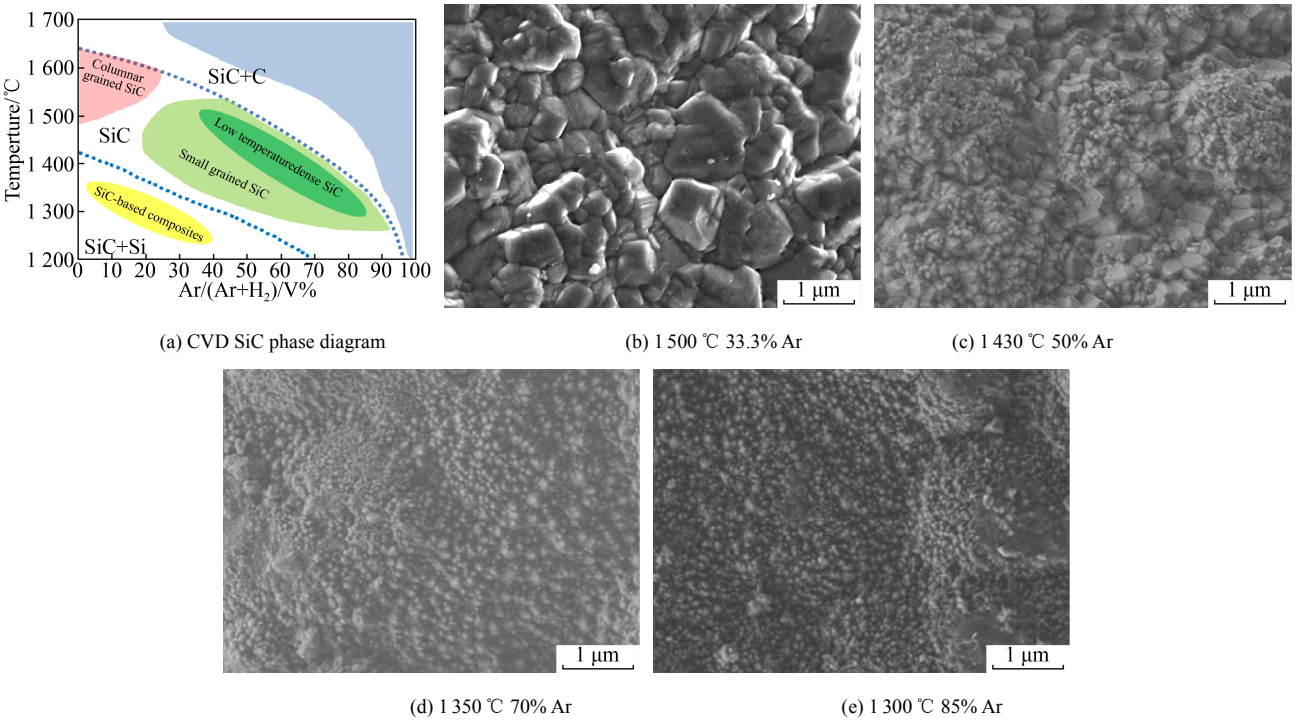


(e) Corresponding hardness at different positions for the sample prepared with sandwiched SiC layer

Fig. 6　Microstructure and corresponding hardness of the SiC layer by the pulsed propylene method[32]

4.3 SiC/C/Si mixed coating layers based on the phase diagram of SiC

A systematic study of the SiC coating layer preparation at different temperatures and gas ratios in the CH_3SiCl_3–Ar–H_2 system by FB–CVD was established (Fig. 7)[33]. From the as-obtained phase diagram, different kinds of SiC/C/Si mixed coating layers were prepared using the FB–CVD method. We found that in a wide region of low temperatures and high Ar concentrations, especially in the preparation parameters along the SiC/SiC+C boundary in the phase diagram, the stoichiometric SiC layer showed very fine grains. As shown in Fig. 7 for conventional columnar-grained SiC, the grain size was usually larger than 5 μm; however, the observed grain size on the surface of the SiC layer was 1–2 μm and 300–800 nm at 1 500 ℃ with 33.3% Ar and 1 430 ℃ with 50% Ar, respectively. SiC with a grain size in the range of 100 to 200 nm was obtained by further decreasing the temperature and increasing the Ar ratio. In addition to the enhanced hindering of silver, this SiC layer with fine grains is expected to exhibit other improved properties, such as improved oxidation resistance, mechanical properties, and heavy metal fission product blocking ability.



(a) CVD SiC phase diagram (b) 1 500 ℃ 33.3% Ar (c) 1 430 ℃ 50% Ar

(d) 1 350 ℃ 70% Ar (e) 1 300 ℃ 85% Ar

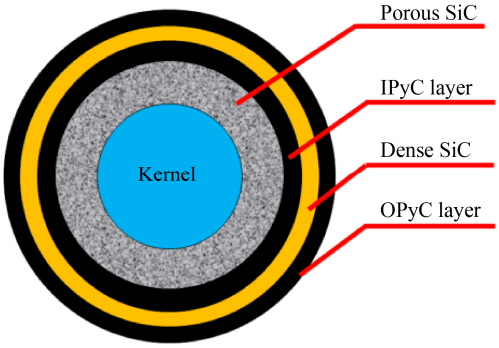
Fig. 7 SiC-based series products based on the CVD SiC phase diagram and SiC layer with fine grains obtained along the SiC/SiC+C boundary in the phase diagram[33]

4.4 Porous SiC coating preparation

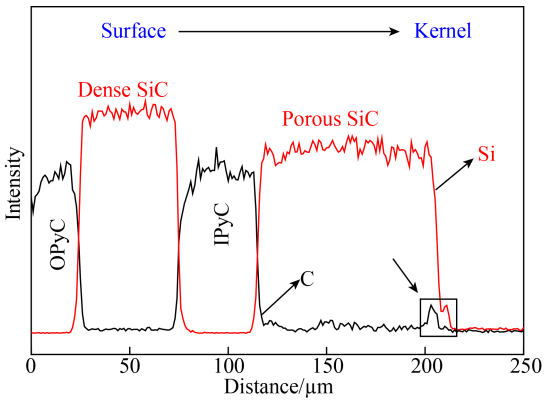
To improve the properties of TRISO particles, a porous SiC coating was designed and prepared for this study. The PyC buffer layer in the TRISO particles can attenuate fission recoils and provide void volume for gaseous fission products as well as carbon monoxide (CO). Although it showed great success during the irradiation test and in real internal reactor operation, this layer may demonstrate problems when the fuel element is operated in the environment at high temperatures with deep burn-ups. One main problem is the CO gas, which is produced through free oxygen from the fission of the nuclear kernel reacting with this carbon layer. CO and other gas products may induce an overpressure on the SiC layer. Another problem is the so-called amoeba effect, which is defined as kernel migration resulting from carbon transport under a temperature gradient. This kernel migration moves toward the SiC layer, which can chemically attack and fracture the SiC layer. Based on the above reasons, we proposed an improved design for the TRISO particles that comprised porous SiC layers to replace porous carbon layers (Fig. 8)[34–35]. An inner porous SiC layer with a thickness of 80–110 μm and a density of 30%–90% of the theoretical density of SiC was designed. This design possesses advantages, including significantly reducing the total internal pressure of the particles, reducing the risks of the amoeba effect and providing a preliminary sacrificial layer to resist chemical attack of the solid fission products.

For the preparation of the porous SiC layer, three methods are proposed. The first method, which is called the high MTS concentration method, is similar to the deposition of the dense SiC layer, but the MTS concentration is increased many (5–10) times. The second method is called the high Ar concentration

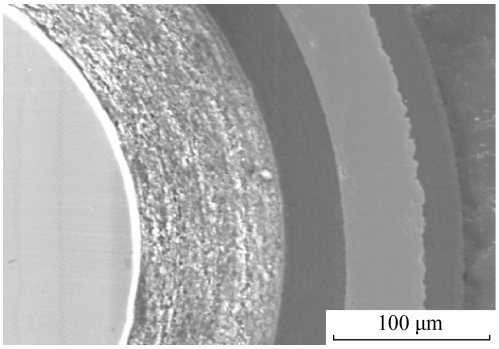
method, which is a two-step method based on the SiC phase diagram discussed above. The SiC mixed with the carbon layer is obtained under high Ar concentration conditions first, and porous SiC is then obtained by eliminating the excess carbon. The third method is the HMDS (hexamethyldisilane $(CH_3)_6Si_2$)) method, in which the porous SiC layer can be prepared using HMDS as the precursor. The density of the porous SiC layer could be tuned well by adjusting the experimental parameters.



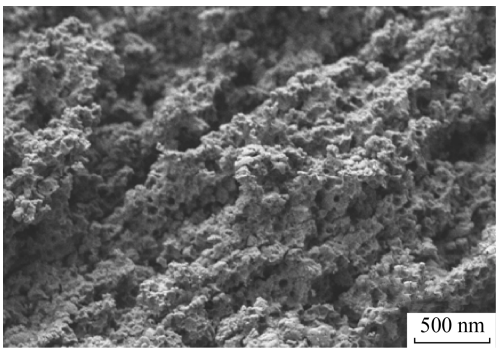
(a) New particle design



(b) Element line scan



(c) Polished surface of the coated particle



(d) Microstructure of the porous SiC inner layer

Fig. 8　Novel coated-particle design using porous SiC as the inner layer and a dense SiC outer layer[34]

4.5　SiC nanowires for accident tolerance fuel (ATF) cladding

In addition to the studies of the SiC coatings on nuclear kernels, we also researched SiC-related nanomaterials by the FB–CVD method. For the new designs of advanced nuclear systems, nanomaterials are increasingly being applied due to their unique microstructure and high surface area. For example, one-dimensional SiC, such as fibers and wires, can be used as new fuel cladding in ATF cladding[36]. SiC nanomaterials with novel morphologies were also obtained by the FBCVD method for this study.

By using the FBCVD system, we successfully prepared SiC nanowires[37]. Fig. 9 illustrates the fluidized bed synthesis system and deposition process of the SiC nanowires. The perpendicular uneven temperature distribution was designed especially for the fluidized bed chemical vapor deposition process.

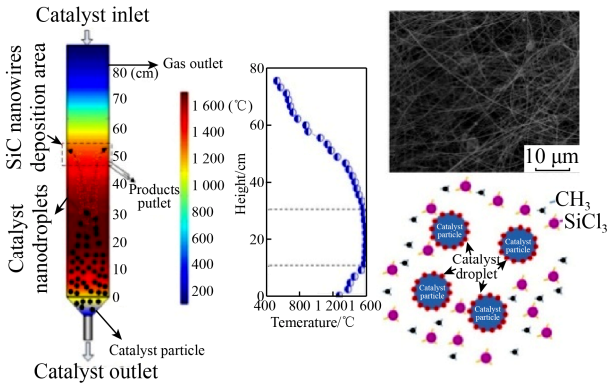
Highly flexible SiC nanowires with diameters in the range of 10 to 50 nm and lengths of several hundreds of micrometers to several millimeters were synthesized by a novel catalyst-assisted FB–CVD method. The fluidized bed was specially designed to form a perpendicular uneven temperature distribution. Suspended catalyst liquid droplets were produced in the middle high-temperature zone, and SiC nanowires were formed *in-situ* on the catalyst droplets and then accumulated on the upper part of the fluidized bed. The nanowires showed single-crystal features with stacking faults. From the HRTEM investigation, a nanoscale smooth plane between the nanowire and the catalyst tip was observed, and the two phases showed highly epitaxial growth. Mass production can be realized by integrating a powder collection system at the end of a furnace.

4.6　SiC nanoparticles for fully ceramic encapsulated (FCM) fuels

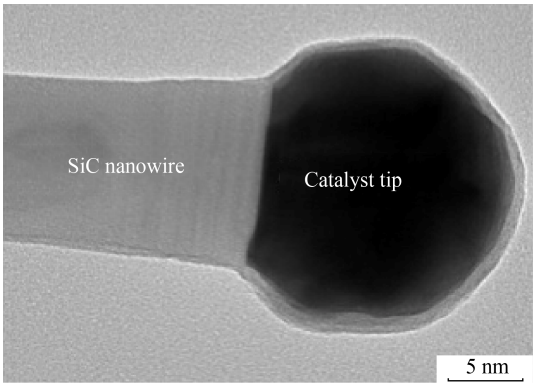
Fully ceramic encapsulated (FCM) fuels represent another new design to enhance the safety of nuclear fuels. In this fuel design, the TRISO-coated fuel particles were dispersed in the matrix to construct a fully ceramic fuel[38], as shown in Fig.10. In this study, SiC nanoparticles were used as the raw powder, which can significantly decrease the sintering temperature of the composites.

By using HMDS as the precursor, we successfully synthesized monodispersed SiC nanospheres with average particle sizes from 10 to 200 nm by the FB–CVD method[39]. The diameter and component of the nanospheres can be adjusted by changing the

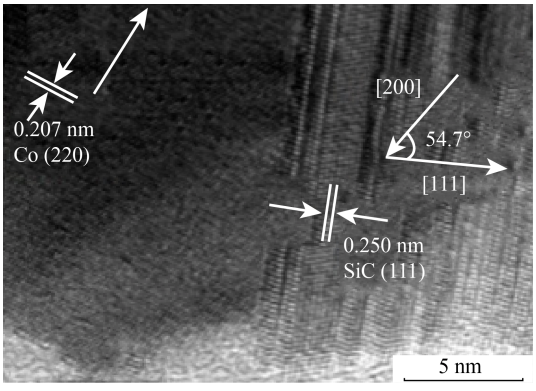
temperature and gas ratios (Fig. 11). The BET surface area of the nanoparticles can be modulated by changing the particle sizes or by additional heat treatment. SiC nanospheres with composite compositions of SiC–Si and SiC–C were also obtained by controlling the reaction atmospheres. As for the SiC coating layers, we established an experimental CVD phase diagram of SiC nanoparticles in the HMDS–Ar–H_2 system. Stoichiometric SiC was obtained in a wide range of H_2/Ar ratios. Free Si was formed at low temperatures with high H_2, while free carbon was formed at high temperatures with low H_2. A novel hierarchical structure with 5–8 nm SiC nanoparticles



(a) Prepared process and formation mechanism of the SiC nanowires



(b) TEM images of the SiC nanowire



(c) HRTEM of the SiC nanowire and the catalyst tip

Fig. 9 SiC nanowires obtained by a catalyst-assisted FB–CVD method[37]

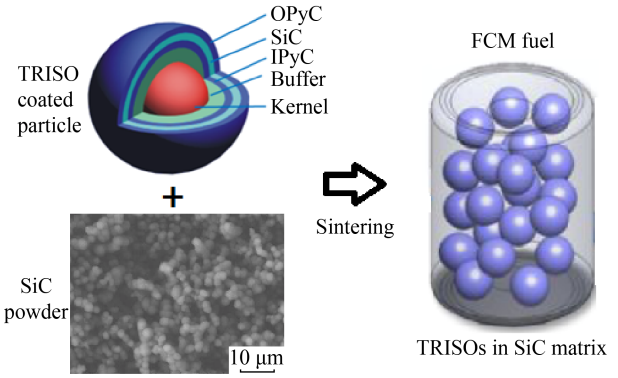
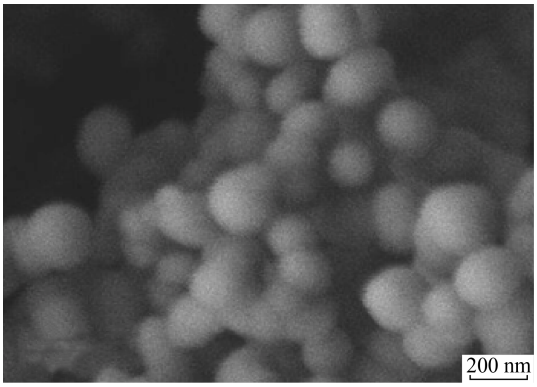
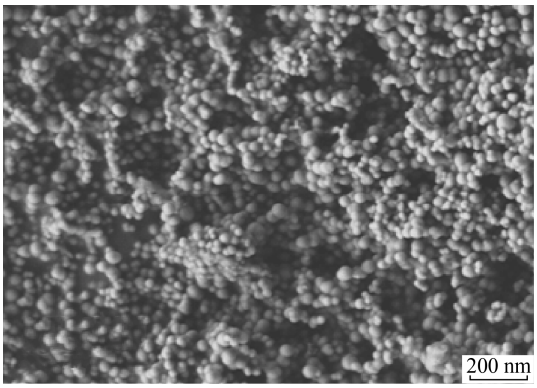


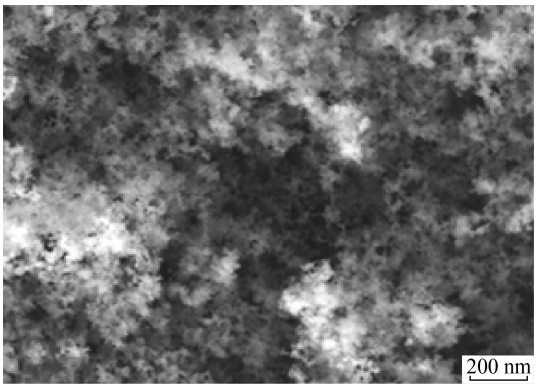
Fig. 10 FCM fuel made of SiC powder (nanoparticles) and TRISO-coated particles in recent ATF design for next-generation nuclear fuel



(a) 800 ℃ 60% Ar



(b) 1 000 ℃ 30% Ar



(c) 1 200 ℃ 10% Ar

Fig. 11 SiC nanoparticles with adjusted particle size under different FB–CVD conditions[39]

embedded in the graphite matrix was obtained after heat treatment of the SiC–C nanospheres. For SiC sintering technology optimization using FCM, nanoparticle design should be focused on in the future. SiC–Si, SiC–C hierarchical nanoparticles and SiC@Al_2O_3 core-shell composite nanoparticles were prepared, and related research on these different kinds of nanoparticles is underway.

5 Conclusions

The FB–CVD method is a flexible method, which can be used to prepared different kinds of SiC coating layer materials and nanomaterials. The CFD–DEM–PBM model was proposed to simulate the FB–CVD particle coating process, aimed at analyzing and improving the coating uniformity. Different kinds of SiC series products, such as dense SiC layers, fine-grained SiC coating layers, SiC/C/Si mixing coating layers, porous SiC coating layers, SiC nanowires and SiC nanoparticles aimed at nuclear applications are developed in our lab. In the future, more attentions will be given to the design and preparation of SiC nanomaterials, which can be used in the recent emerging accident tolerant fuel and fully ceramic encapsulated fuel concepts in diverse nuclear systems.

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