



Critical issues and current status of SiC/SiC composites for fusion

A. Hasegawa^{a,*}, A. Kohyama^b, R.H. Jones^c, L.L. Snead^d, B. Riccardi^e,
P. Fenici^f

^a Department of Quantum Science and Energy Engineering, Tohoku University, Aramaki-aza-Aoba 01, Aoba-ku, Sendai 980-8579, Japan

^b Kyoto University, Japan

^c Pacific Northwest National Laboratory, USA

^d Oak Ridge National Laboratory, USA

^e ENEA, Italy

^f JRC Ispra, Italy

Abstract

SiC fiber-reinforced SiC matrix composites (SiC/SiC) are being considered as a candidate structural material for fusion reactors because of their low induced radioactivity by 14 MeV neutron irradiation and high-temperature strength. Material development of SiC/SiC composites, including SiC fiber, matrix and interphase processing for fusion reactor applications, has been under investigation for several years. The purpose of this paper is to present an overview of current research in material development, properties and irradiation response of new SiC/SiC composites. Several technologically critical issues to be solved for fusion applications, such as irradiation resistance, thermal properties, environmental effects, hermeticity, joining technique and protective coating are also reviewed. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

SiC fiber-reinforced SiC matrix composites (SiC/SiC) are being considered as a candidate structural material for fusion reactors because of their low-induced radioactivity by 14 MeV neutron irradiation, their excellent high-temperature strength, and corrosion resistance [1–3]. Also, β -SiC exhibits very low swelling (<0.2%) after neutron irradiation over the temperature range of 800–1000°C [4], which corresponds to the temperature region for high thermal efficiency when using a gas turbine system. The high thermal efficiency is one of the advantages of an SiC/SiC material system [5].

While these composites are relatively new materials with a limited database, there is sufficient understanding of their performance to identify key issues in their application. These issues include mechanical properties,

radiation stability, nuclear transmutation, thermal conductivity, mechanical and thermal fatigue, thermal shock, hermeticity, compatibility with coolant, joining and design methodology. Material requirements for fusion reactor applications have been reviewed in previous reports [1–3]. This paper reviews the current status of SiC/SiC composites for fusion reactor applications. It also discusses the environmental effects on fibers and composites, including the effects of oxidation and compatibility with breeding materials and neutron multipliers. Critical issues related to nuclear environment are still present. These issues are mainly related to fiber and matrix stabilities under neutron irradiation and to the relatively poor thermal conductivity of SiC/SiC. Other issues including creep and technological issues such as joining and coating will also be reviewed.

2. Material requirements for fusion reactor applications

There are three general fusion reactor design concepts using SiC/SiC composites. These are ARIES-I [6]

* Corresponding author. Tel.: +81-22 217 7923; fax: +81-22 217 7925.

E-mail address: akira.hasegawa@qse.tohoku.ac.jp (A. Hasegawa).

designed by USA, DREAM [7] designed in Japan and TAURO [8] in the EU. High-pressure helium-gas cooling systems with a solid breeding and moderator blanket system were considered in the ARIES-I and DREAM designs. Material issues for the gas cooling blanket systems were reviewed by Jones et al. [1]. Liquid lead(Pb)–lithium(Li) was proposed as a combined coolant, neutron multiplier, tritium breeder and carrier in the TAURO design. Due to safety considerations, relatively low-pressure Pb–Li was utilized. A previous paper reviewed the material performances for each reactor design [2]. Strength of about 300 MPa and a thermal conductivity of 15 W/mK were used in this reactor design studies. Based on recent material development, better material performance can be expected in the near future.

In the construction process of a fusion reactor, portions of the blanket component made of SiC/SiC composites will be fabricated from simple shape pieces and attached to the blanket module. Joints having enough strength and leak tightness will be required in a high-pressure gas cooling system. Joining and sealing techniques, using low activation materials will be required to retain the low activation advantage of SiC/SiC composites.

3. Current status of material developments

3.1. SiC fiber

The fiber is the primary load-bearing element in continuous fiber ceramic matrix composites and fiber texture is also an important factor to design composite parts for particular loading conditions. Polymer derived SiC fibers such as Nicalon (Nippon carbon), Tyranno (Ube Heavy Industries) and Sylramic (Dow Chemical) have enough strength and flexibility to weave into complicated shape fabrics. The microstructure of first generation Nicalon-CG and Tyranno Lox-M fibers consisted of small (2 nm) β -SiC crystallites surrounded by a large amount of amorphous phase of SiC_xO_y . After irradiation these fibers shrunk due to crystallization of the amorphous phase. Residual oxygen and carbon accelerated shrinkage of SiC fibers [1]. The fiber shrinkage caused debonding between fiber and matrix, which resulted in a decrease of the strength of SiC/SiC composites [9]. Dimensional stability of fiber under irradiation is one of the important issues for development of irradiation resistant composites. Fig. 1 shows the density changes of several types of fibers after neutron irradiation [10]. Dow Sylramic, which is an oxygen free and stoichiometric SiC fiber, showed a smaller amount of volume change after irradiation while Nicalon-CG and Tyranno shrunk after irradiation. These results suggested that low oxygen free and stoichiometric fibers may lead to improve resistance to irradiation-induced volume changes. Recrystallization

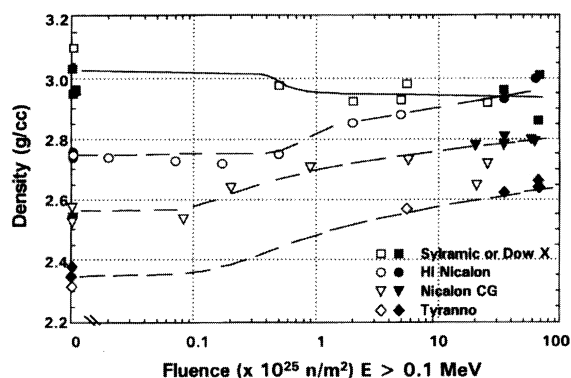


Fig. 1. Density as a function of fast neutron fluence for four SiC-based fibers.

of the amorphous part of SiC fiber also decreases fiber strength at high temperature. Therefore, for fusion and high-temperature applications, oxygen-free, stoichiometric SiC fibers are utilized now.

Using electron curing and pyrolysis of polycarbosilaine fiber, a low oxygen and stoichiometric SiC fiber (Hi-Nicalon type S) has been developed [11]. Tyranno SA by higher temperature heat treatment is also an oxygen free and stoichiometric fiber [12]. Table 1 shows the physical properties of these SiC fibers [13,14]. Hi-Nicalon type S and Tyranno-SA have relatively lower tensile strength but higher tensile modulus (420 GPa) and higher thermal conductivity compared to other fibers.

Fig. 2 shows the strength of these SiC fibers after heat treatment in argon [15]. These results show that Tyranno-SA has the best heat resistance of these fibers. It reveals that this type of fiber extends the temperature region in which composites perform without fiber damage. In 1999, these fibers became commercially available, and composite studies using these new fibers will appear in the near future. Kohyama et al. [5] presented a comparison of fabrication cost and future prospects of these fibers in this conference. Sylramic (Dow Chemical) also has good high-temperature properties, but it contains 2 wt% boron and 0.4% nitrogen [16]. Boron will transform and generate He in neutron environments and nitrogen has a long-lived isotope, therefore, reduction of these elements will be required for fusion applications.

3.2. Interphase

The fiber/matrix interfacial layer or the interphase plays a critical role in the performance of ceramic matrix composites. The interphase must retain favorable high-temperature properties in an irradiation, oxidizing or corrosive environment. Optimization of the interphase is an important factor to improve the mechanical properties of SiC/SiC composites. If the interfacial bond be-

Table 1
Nominal properties of polymer-derived SiC fibers

	Nicalon			Tyranno			Dow	
	NL-200	Hi-Nicalon	Nicalon-S	Lox M	ZMI	ZE	SA	Sylramic
Atomic composition	SiC _{1.34} O _{0.36}	SiC _{1.39} O _{0.01}	SiC _{1.05}	SiTi _{0.02} C _{1.37} O _{0.32}	SiZr _{-0.01} C _{1.44} O _{0.24}	SiZr _{-0.01} C _{1.52} O _{0.05}	SiC O ₂ Al ₂ -0.008	SiTi _{0.02} B _{0.09} O _{0.02}
Tensile strength (GPa)	3.0	2.8	2.6	3.3	3.4	3.5	2.8	3.2
Tensile modulus (GPa)	220	270	420	187	200	233	420	400
Elongation (%)	1.4	1.0	0.6	1.8	1.7	1.5	0.7	0.7
Density (g/cm ³)	2.55	2.74	3.10	2.48	2.48	2.55	3.02	>3.1
Diameter (μm)	14	14	12	11	11	11	10	10
Specific resistivity (Ω cm)	10 ³ –10 ⁴	1.4	0.1	30	2.0	0.3	–	–
Thermal expansion coefficient (10 ⁻⁶ /K)	3.2 (25–500°C)	3.5 (25–500°C)	–	–	–	–	–	5.4 (20–1320°C)
Thermal conductivity (W/mK)	2.97 (25°C) 2.20 (500°C)	7.77 (25°C) 10.1 (500°C)	18.4 (25°C) 16.3 (500°C)	1.35	2.52	3.78	64.6	40–45

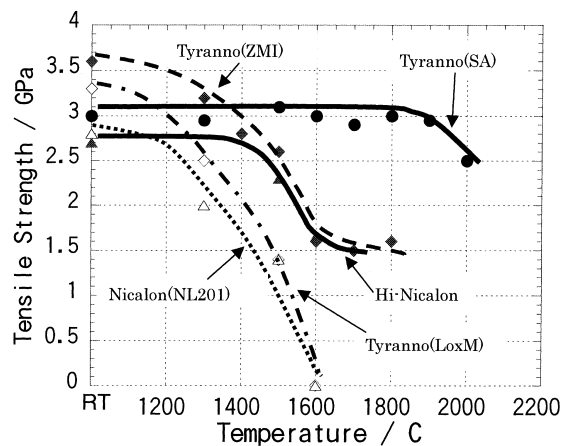


Fig. 2. Tensile strength of SiC-based fibers after heat treatment in Ar for 1 h [15].

tween SiC fibers and SiC matrices is too strong it will not impede on-coming cracks and the composites will fail in a brittle manner. For a weak interface, a crack in the matrix can lead to debonding or crack deflection at the interface, crack bridging, fiber fracture, and finally fiber pull out. All these additional energy absorbing phenomena lead to an enhanced fracture toughness and a non-catastrophic failure mode. Some key issues for the interphase are material, thickness and coating methods. Carbon single layer, porous SiC and multilayer SiC interphases have been studied. By examining dependence of bending strength of composites on carbon coating thickness, the optimum carbon coating thickness was found to lie in the range of 0.17–1 μm [18,19,66] (see Fig. 3). Droillard et al. [67] and Rebillat et al. [68] studied fracture toughness of SiC/SiC composites using various multilayered interphases to control interface strength and suggested that higher toughness was obtained with those composites possessing strong interphases. Bertrand et al. [69] and Snead et al. [17] reported crack deflection behavior of SiC–C multilayered composites. Multilayered interphases can replace the single pyrolytic carbon interphase [69]. Degradation of the ultimate bend stress was observed for all materials studied, while the matrix microcracking stress was unchanged, and the multilayer SiC interphase material exhibited the least degradation in ultimate bend stress, while the porous SiC exhibited the greatest degradation [17]. Irradiation-induced microstructural evolution of the interphase was not performed. Phase stability of the interface is also an important parameter for fusion applications.

3.3. Matrix and composite processing

Several fabrication processes for SiC/SiC composites have been under investigation for more than 10 years.

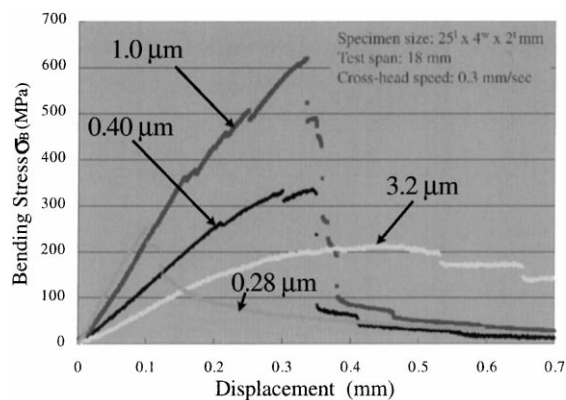


Fig. 3. Effect of carbon coating thickness on three-point bending strength [19].

The chemical vapor infiltration (CVI) process is currently viewed as the industrial leader [1,20], but the CVI process is slow with many inherent difficulties such as cost and substantial residual porosity. The properties of

CVI-processed composites using Hi-Nicalon S fibers were presented in this conference [21]. The polymer impregnation pyrolysis (PIP) process is simple and relatively lower cost process compared to the CVI process. Also, the PIP process allows the manufacturing of larger and more complex shapes. However, multiple processing cycles are required to produce a dense part. Some results of PIP-processed composite using advanced fibers have been presented [22–26]. A hybrid process combining CVI and PIP is under investigation [27] because it may provide better control of matrix densification and shape of the part.

The reaction sintering (RS) process realizes full density, near net shape, complex shape capability, and high thermal conductivity [28]. A high-temperature (1447°C) heat treatment is required in the RS-process to melt silicon (Si) into fabrics. Reduction of the residual Si-phase in the matrix, interphase and fiber optimization are the remaining issues of RS-processed composites for fusion applications. The chemical vapor reaction (CVR) process, a potentially less expensive process method is being developed to obtain high thermal conductivity

Table 2
Typical properties of various types of unirradiated SiC/SiC composites

Method	Fiber	Vf (%)	Bulk density (g/cm ³)	Heat resistant temperature (°C)	Tensile strength (MPa)	Work of fracture (kJ/m ²)	K _{IC} (MPa m ^{1/2})	Thermal conductivity (W/mK)
CVI (DuPont)	Hi-Nicalon 2D	40	2.7		217/336 ^a	–	–	–
	Nicalon 2D	40	2.5		200		30–	3 (inplane, RT) 2.5 (through thickness, RT)
CVI [33]	Nicalon 2D	36	2.3	1200	215/280	–	27–29	~7 (RT –1000°C)
CVI (SEP)	Nicalon 2D	40	2.5		285		30	3 (inplane, RT) 2.5 (through thickness, RT)
PIP	Tyranno 3D [36]	35–39	2.4	1200	400 ^a	4.3–8	11.5–16	–
	Nicalon 2D [37]	35	2.3	1420	240/460 ^a	–	–	–
PIP DOW/Kaiser	Nicalon 2D	40	2.1		207/366 ^a		–	43 (inplane, RT)
RS [32]	Hi-Nicalon UD	30	3.0	1359	500	6.4	–	50 (RT) 30 (1000°C)
Fiber-bonded ceramics (Tyranno-hex)	Tyranno UD, 2D [34]	90	2.5	1500	450–550, UD 190–210, 2D	8.9	46.2	4.5 (RT)
	Tyranno-SA UD [35]	>90	–	1500	600 ^a	1.2	–	50 (RT) 25 (1400°C)

^a Bend strength.

through the thickness of composites [29,30]. Hot pressed (HP) composites have also been studied in an effort to obtain higher density [31].

Table 2 lists some typical material properties of currently available SiC/SiC composites [1,32–37]. Bend strength of Tyrannohex UD composites achieved up to 600 MPa [19]. Fiber selection and composite processing are the key parameters to achieve high strength with high fracture energy. Density is also process dependent. Generally, RS-processed composites have the highest density, (up to 3.0 g/cm³) and thermal conductivity (up to 50 W/mK). The newly developed fiber Tyranno-SA, which exhibits high temperature resistance, can be utilized where higher processing temperatures are required, such as RS.

Hermeticity of composites is an important issue for fusion applications, because the blanket structure is a pressure boundary for high-pressure helium cooling gas in the SiC/SiC blanket system. To improve hermetic properties, open pore and long cracks must be closed. A leak-tight CVD-SiC coating has been considered as a hermetic barrier [17]. Polymer-infiltrated and pyrolyzed SiC coatings also have been studied as a gas permeation barrier of CVI-processed composites [38]. Further studies are required to obtain a hermetic coating for a high-pressure gas system.

3.4. Thermal conductivity

As is shown in Table 1, stoichiometric SiC fibers have high thermal conductivity. Thermal conductivity of composites processed by CVR [29,39] or by RS [28] showed relatively higher thermal conductivity compared to that of PIP and CVI-processed composites because of their low porosity. Appropriate weave texture can improve anisotropy of thermal conductivity of composites. Densification of the matrix is one of the key parameters to improve the thermal conductivity. Yamada [39] showed the density dependence on thermal conductivity of SiC/SiC composites fabricated by CVI process. Thermal conductivity was shown to increase with sample density and it was suggested that localized large cavities had a worse effect on the thermal diffusivity than smaller size pores distributed in the whole volume [39]. To improve thermal conductivity, matrix processing for porosity control and densification are required for fusion applications.

4. Irradiation effects

4.1. Monolithic SiC

The temperature dependence of irradiation-induced swelling of monolithic SiC is summarized in Refs. [1–4,70,71]. At temperatures below about 100°C, SiC

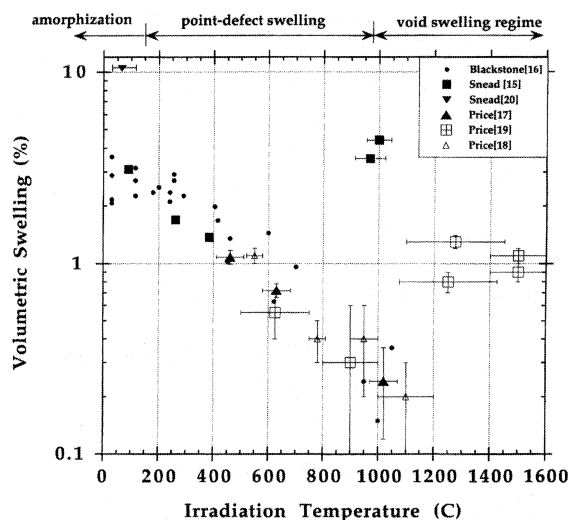


Fig. 4. Volume swelling of SiC as a function of irradiation temperature.

readily becomes amorphous under irradiation. The critical temperature above which amorphization does not occur is about 100°C for the electron irradiation and 300°C for ion irradiation [1,40]. In the case of neutron irradiation, Snead [41] reported that the critical temperature was estimated to be greater than 125°C. The temperature dependence of volume swelling is summarized in Fig. 4 [4]. Swelling, due to accumulation of point defects, decreases with increasing irradiation temperature. A minimum of swelling about 0.2% was observed at irradiation temperatures between 800°C and 1000°C. Above 1000°C, void swelling due to vacancy migration and coalescence of vacancy cluster appears in SiC. The irradiation response in this temperature range is very important for fusion applications because it corresponds to the operating temperature. Unfortunately, there is only a small amount of data for this temperature range. Transmuted helium may also enhance void formation in SiC. Therefore, understanding of the swelling behavior at high temperature with transmuted helium is required.

4.2. SiC fiber

The radiation response of SiC-based fiber was reported by Youngblood et al. [10] and Osborne et al. [72]. Advanced fibers, such as Nicalon-S and Tyranno-SA, have stoichiometric compositions and high crystallinity. Therefore, these fibers are expected to be behaving similarly to monolithic SiC during irradiation. As was mentioned earlier, changes that can account for fiber density increases include: (1) relaxation of amorphous structure, (2) crystallization, or (3) loss of oxygen. These fibers are currently under irradiation in HFIR, under the Japan-US collaboration program (JUPITER), with the

expected dose to be 5 dpa at 300°C, 500°C and 800°C. It will be possible to investigate the effects of radiation in the near future.

Irradiation creep behavior of SiC fiber (SCS-6) under the 14 MeV deuteron irradiation has been studied by Scholz [42] using a torsional creep method. SCS-6 is a SiC fiber produced by depositing SiC on a carbon fiber substrate by CVD technique. The grain structure and orientation of the resulting β -SiC sheath are similar to that of the matrix of SiC/SiC composites produced by CVI. Therefore, the results can be considered to have similarities to the creep behavior of matrix SiC. Significant irradiation-induced creep enhancement in the temperature range 450–1000°C was reported. The irradiation creep rate was constant in the temperature range from 900°C to 1000°C almost from the beginning of the irradiation. A transient creep rate region was observed below 800°C. Irradiation creep of advanced SiC fibers was reported by Scholz and Youngblood using the BSR method with combined 9 MeV protons at temperatures of 600°C and 800°C [43]. The bend stress relaxation (BSR) test of SiC fibers is also under irradiation at 800°C up to 5 dpa in HFIR.

4.3. SiC/SiC composites

Neutron irradiation effect studies on SiC/SiC composites have been mainly limited to SiC-fiber-reinforced composites containing earlier types of oxygen containing, polymer-derived SiC fibers. Irradiation effect data for newly developed composites, made with advanced fibers are not yet available at this moment. In the case of the conventional composites Nicalon/C/CVI-SiC, Youngblood et al. reported that the fracture strength decreased rapidly with increasing fluence plateau at about 5 dpa with little further decrease up to 80 dpa [44].

Snead and Kohyama compared bending strength of three composites containing Nicalon, Hi-Nicalon and Nicalon-S fiber, respectively after neutron irradiation up to 1 dpa in HFBR [45]. A slight increase of the bend strength for Nicalon-S composites was observed, while 30–35% and 5–10% reduction of strength was observed in the Nicalon and the Hi-Nicalon composites, respectively [45]. In an effort to obtain better radiation resistant, SiC composites, fabricated using various processing methods and advanced SiC fibers are being developed. Irradiation experiments are in progress.

Previous works revealed a decrease in thermal conductivity of SiC after irradiation which was caused by small defect accumulation [2,9]. However, the magnitude of the reduction of thermal conductivity decreased with increasing irradiation temperature. Only a small amount of data on the thermal conductivity change at operating temperature currently exists [73]. A tentative result of thermal conductivity change for CVD-SiC during neutron irradiation was reported by Yamada and

Snead [46] using TRIST-TC1 experiment in HFIR, which was conducted by the Japan–US collaboration program. The thermal conductivity of monolithic CVD β -SiC decreased in the early stage of irradiation and saturated. The saturated thermal conductivity was about 35% of the initial value of SiC for 700°C irradiation.

4.4. Transmutation gases

Transmuted He gas effects have been simulated using He implantation or simultaneous He-ion and heavy-ion irradiations. Calculation of the total amount of transmuted He based on fusion reactor material design was carried out by Noda [47]. The Helium/dpa ratio is predicted to be about 100 appm/dpa in first wall region of the solid breeding blanket system when using SiC/SiC composite as the structural material. Helium is an insoluble element in almost all the solid materials, and it stabilizes vacancy type clusters. It may enhance void swelling of SiC under fusion reactor conditions.

As the threshold energy of He production by neutron induced transmutation is several MeV, a high concentration of He in SiC has not been obtained by fission reactor irradiation. Therefore, accelerator irradiation experiments have been used to study helium effects. Single-, dual-, or triple-beam experiments have been conducted to investigate helium effects on microstructural evolution. The microstructural development of a composite containing advanced fiber fabrics below 800°C was not clearly observed, probably because the irradiation temperature was too low for helium to move and to cluster [48,49], but small cluster defects were observed. Helium diffusion behavior and microstructural observation of SiC/SiC composites, SiC fiber and monolithic SiC were reported by Hasegawa [50]. Helium in carbon phases becomes mobile between 500°C and 800°C and, in the SiC phase, becomes mobile above 900°C or 1000°C. In the higher temperature region, the He desorption rate of SiC fiber becomes much larger than monolithic SiC. This result is consistent with a previous diffusion study by Jung [51]. These results suggested that helium might be mobile in C and the amorphous like Si–C phase in composite in the range of the expected reactor operating temperature. Further systematic irradiation experiments are required at temperatures above 1000°C.

Mechanical property changes were investigated using high energy He implantation by cyclotrons [52,53]. Bending tests of SiC/SiC composites after high-temperature implantation (950°C) up to a helium concentration of 2500 appm showed a mean 38% decrease of strength. The load–displacement curve that exhibited significant decrease of the values of the maximum deflection was also seen [52]. Swelling of the SiC matrix and shrinkage of Nicalon fiber were reported by Scholz [54]. Decrease in strength was observed at about 150 appm implanta-

tion at temperatures ranging from 400°C to 800°C [53]. These results indicate that the typical post matrix-cracking toughening mechanisms of interphase sliding during fiber pull-out process may be degraded by helium implantation. Microstructural observations of He-implanted SiC/SiC composite were reported by Hasegawa, as shown in Fig. 5 [55]. Helium bubbles were neither observed at the interface between matrix/interphase (C) and between fiber/interphase (C) nor in the SiC fiber after about 10 000 appm helium implantation at room temperature and annealing at 1400°C. Bubbles were observed only at grain boundaries in SiC. Helium de-

sorption results can explain this microstructure. More research is required to clarify the mechanical property degradation of SiC fibers and composites by helium.

5. Environmental effects

5.1. Oxidation

Several mechanisms to explain environmentally induced crack growth of SiC/SiC at elevated temperatures, with varying amounts of oxygen have been proposed [56]. One mechanism is defined as the oxidation embrittlement mechanism (OEM), and the other is the interphase removal mechanism (IRM). For OEM, oxidation results from the formation of SiO₂ that either grows on the surface of exposed fibers and weakens them, or bond the fiber to the matrix, leading to premature failure of bridging fibers. For IRM, crack growth occurs by the active oxidation and less of the carbon interphase. The IRM mechanism occurs at low concentrations of O₂ where the SiO₂ formation rate is too low to seal cracks. It is expected that fusion will operate within the IRM regime.

Interphase oxidation in SiC/SiC composites at varying partial pressures of oxygen was reported by Giannuzzi [57]. A microstructural study on crept SiC fiber and SiC/SiC composites in argon gas containing low partial pressure of oxygen using high-resolution electron microscope was reported by Shibayama [58]. They reported SiC debonded by self-decomposition of SiC fiber at a quite low oxygen concentration. A previous model based predictions of the environment-defined life-time of SiC/SiC composites assumed that IRM was operative. However, it will be necessary to verify the specific crack growth mechanism for SiC/SiC under fusion relevant conditions.

5.2. Compatibility

The compatibility of SiC/SiC commercial composites with solid breeder beds in fusion relevant conditions was investigated [59,60]. Solid Li₄SiO₄ and Li₂TiO₃ were used for the exposure experiments. The composite surface was coated uniformly with a CVD-SiC coating with thickness about 0.1 μm. The cells were operated with Li₄SiO₄ and Li₂TiO₃ up to 10 000 h at 800°C in flowing He containing 1000 ppm H₂ gas. After 10 000-h exposure to Li₄SiO₄, lithium metasilicate was found on the samples. Fig. 6 shows a cross-sectional observation of the exposed SiC/SiC composite. The original SiC coating was present after exposure but it transformed slightly. Slight changes of the mechanical properties were also reported at different temperatures. In the case of exposure to Li₂TiO₃, the protective coating was partially eroded after 10 000 h of exposure. The SiC layer was full

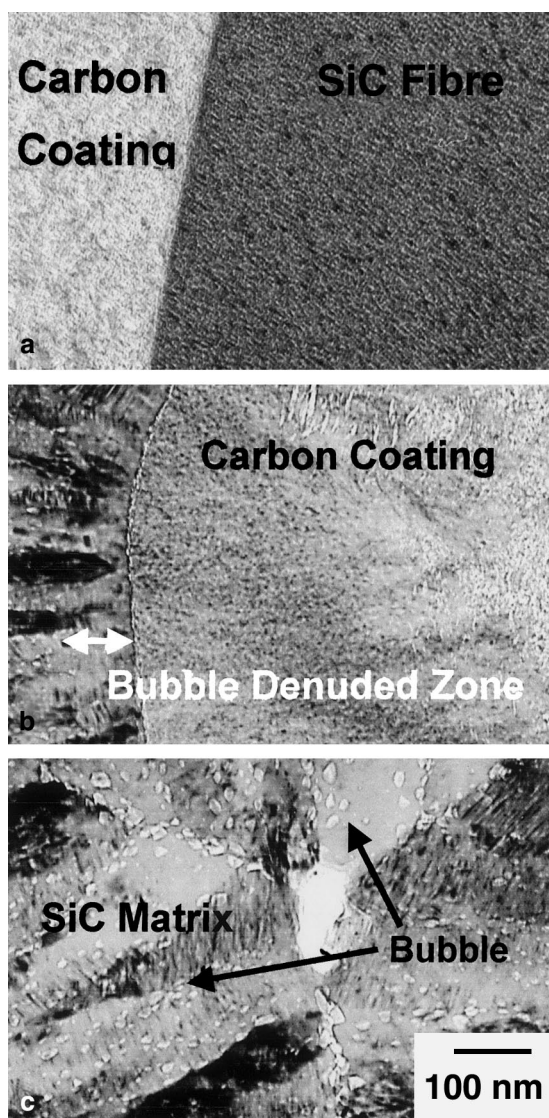


Fig. 5. TEM micrographs of SiC/SiC composites after helium implantation and annealing at 1400°C for 1 h: (a) SiC fiber/carbon coating; (b) carbon coating/SiC matrix; (c) SiC matrix.

of voids and cracks across its entire thickness. In spite of these changes, the mechanical properties of the composites did not change significantly, indicating the bulk was not damaged after 10 000-h exposure.

The chemical reactivity of SiC/SiC composites with Be and Li oxide breeder materials was studied by Kleykamp [61]. α -SiC/Be/ α -SiC pellets were annealed in a closed cylindrical refractory metal capsule for 10 days at 700°C and 900°C. No indication of an incompatibility between Be and SiC was reported after the 700°C annealing, whereas, at 900°C, a two-phase Be₂C–Si was observed on the SiC surface of the pellet, because Be₂C and Si are in thermodynamic equilibrium at 1000°C. Powder reaction experiments were also made between α -SiC and Li₄SiO₄, LiZrO₃ and LiTiO₃ pellets in a quartz tube furnace at 700°C for two weeks under static argon gas. No reaction between α -SiC and the Li ceramics was observed. The development of a protective coating on SiC/SiC to prevent Be diffusion into the SiC at reactor operating temperature will be required in the breeding blanket. Only limited data are available on the compatibility of SiC/SiC with respect to Pb–17Li. The data are from a test performed at 800°C in stagnant liquid Pb–17Li using 2D SiC/SiC composites.

One important technical issue for reactor engineering is joining, as required for fabrication of large and complexly shaped blanket parts. Candidate joining materials for fusion systems are as follows: (1) melt infiltrated and reaction-formed SiC, (2) preceramic-polymer derived SiC, (3) low activation, high-temperature glasses, (4) in situ reinforced silicides and (5) high-temperature braze. Singh [62] has reported on reaction-forming methods of SiC/SiC composites. The flexural strength of the joints were about 150 MPa at RT and about 217 MPa at 1350°C. For calcia–alumina glass–ceramics

joints, which have been studied for joining and coating materials [63,64], the strength of the joint was 28 MPa at room temperature. The use of a high-temperature adhesive based on Ceraset preceramic polymer was reported by Fareed [65]. Bend strength of joints using Ceraset was about 40–60 MPa at 1100°C and at room temperature, respectively. The strength of these joints were weak compared to the strength of composites. Improvement of joint strength is required for application to gas cooled reactor systems. Irradiation behavior of these joints will also require studying.

A coating for hermeticity is also an important issue for a gas cooled reactor system. In the ARIES-I design, CVD-SiC coatings were considered. Glass–ceramics can be versatile joining or coating materials with tailorable thermal and mechanical properties; in addition, they are not affected by oxidation and can be self-sealant at temperatures above the glass softening point. A eutectic composition in the binary phase CaO–Al₂O₃ glass has been studied for joining and coating materials because it has a high characteristic temperature with B and Li. The coated composites did not seem to be affected after 140 h at 800°C in contact with a ceramic breeder [63].

6. Summary

The current status and critical issues of SiC/SiC composites for fusion applications have been reviewed.

1. *Material developments.* Advanced SiC fibers, which are expected to have high radiation resistance, have been developed commercially, and the optimization of composite forming process using these advanced fibers is on-going.
2. *Irradiation effects.* Irradiation response up to 1 dpa of new advanced composites have been obtained. Irradiation experiments to doses above 10 dpa on these newly developed composites are on-going.
3. *Environmental effects.* Compatibility of the composites with Li ceramic breeding materials was studied. A SiC layer used as a protective coating was effective under specific conditions, but there is no data, which include irradiation conditions. SiC is not compatible with Be at temperature above 900°C. A protective coating in reducing atmospheres will be required.
4. *Technological issues of reactor engineering.* Development of joining and hermetic coating techniques are in progress in collaborations with non-fusion application programs.

References

- [1] R.H. Jones, D. Steiner, H.L. Heinisch, G.A. Newsome, H.M. Kerch, J. Nucl. Mater. 245 (1997) 87.

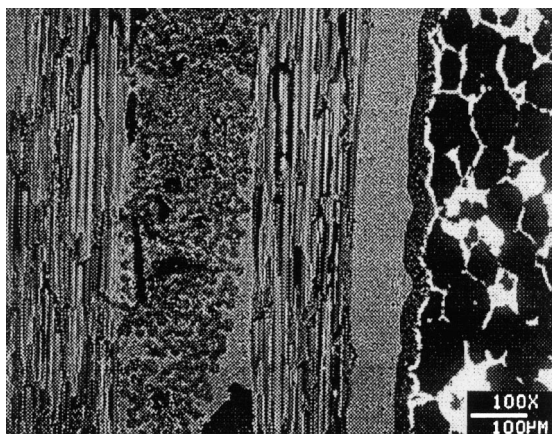


Fig. 6. SEM cross-sectional view of SiC/SiC boundary between Li₄SiO₄ after 10 000-h exposure to flowing He + 1000 ppm H₂ gas at 800°C.

- [2] P. Fenici, A.J. Frias Rebelo, R.H. Jones, A. Kohyama, L.L. Snead, *J. Nucl. Mater.* 258–263 (1998) 215.
- [3] L.L. Snead, R.H. Joes, A. Kohyama, P. Fenici, *J. Nucl. Mater.* 233–237 (1996) 26.
- [4] E.E. Bloom, *J. Nucl. Mater.* 258&259 (1998) 7.
- [5] A. Kohyama et al., these Proceedings, p. 20.
- [6] F. Najmabadi, UCLA-PPG.-1323, 1991.
- [7] S. Ueda, S. Nishio, Y. Seki, R. Kurihara, J. Adachi, S. Yamazaki, Dream design team, *J. Nucl. Mater.* 258–263 (1998) 1589.
- [8] A. Perez Ramirez, A. Caso, L. Giancarli, N. Le Bars, G. Le Marois, J.F. Salavy, *J. Nucl. Mater.* 233–237 (1996) 1257.
- [9] G.W. Hollenberg, C.H. Henager Jr., G.E. Youngblood, D.J. Trimble, S.A. Simonen, G.A. Newsom, E. Lewis, *J. Nucl. Mater.* 219 (1995) 70.
- [10] G.E. Youngblood, R.H. Jones, A. Kohyama, L.L. Snead, *J. Nucl. Mater.* 258–263 (1998) 1551.
- [11] H. Ichikawa, K. Okamura, T. Seguchi, in: Proceedings of the Second International Conference on High-temperature Ceramic–Matrix Composites, 1995, p. 65.
- [12] T. Nakayasu, M. Sato, T. Yamamura, K. Okamura, Y. Katoh, A. Kohyama, *Ceram. Eng. Sci. Proc.* 20 (1999) 301.
- [13] K. Okamura, *Adv. Composite Mater.* 8 (1) (1999) 107.
- [14] H.M. Yun, J.A. DiCarlo, *Ceram. Eng. Sci. Proc.* 20 (1999) 259.
- [15] M. Satou, Technical Reports of Tyranno Fiber, UBE industries, Ltd.
- [16] R. Jones, J. Rabe, D. Petrak, in: Proceedings of the Third IAE International Workshop on SiC/SiC Composites, 29–30 January, 1999, Cocoa Beach, FL, p. 23.
- [17] L.L. Snead, E. Lara-Curzio, in: Fusion Materials, Semi-annual Progress Report for Period Ending, 31 December, 1998, DOE/ER-0313/25, p. 100.
- [18] R.A. Lowden, in: *Adv. Composites Materials*, Ceramic Transactions, vol. 19, American Ceramic Society, Westerville, OH, p. 619.
- [19] A. Kohyama, H. Hinoki, H. Serizawa, S. Sato, in: Proceedings of the 11th International Conference on Composites Materials, 1997.
- [20] P. Fenici, H.W. Scholz, *J. Nucl. Mater.* 212–215 (1994) 60.
- [21] T. Noda, H. Araki, W. Yang, A. Kohyama, *Ceram. Eng. Sci. Proc.* 20 (1999) 387.
- [22] R. Yamada, T. Taguchi, N. Igawa, these Proceedings, p. 574.
- [23] R. Jones, D. Petrak, J. Rabe, A. Szweda, these Proceedings, p. 556.
- [24] M. Kotani, A. Kohyama, K. Okamura, T. Inoue, *Ceram. Eng. Sci. Proc.* 20 (1999) 309.
- [25] A. Kohyama, T. Nakayasu, M. Kotani, M. Sato, T. Yamamura, K. Okamura, Y. Katoh, these Proceedings, p. 565.
- [26] S.T. Schwab, P.F. Fleig, J.D. Katz, T.W. Hardek, L.L. Snead, N.L. Vaughn, K.W. Buesking, A. Kohyama, presented at 9th Int. Conf. on Fusion Reactor Materials, Colorado Springs, CO, USA, Oct. 1999.
- [27] A. Ortona, A. Donato, F. Filacchioni, U. deAngelis, A. La Barbera, C.A. Nannetti, B. Riccardi, J. Heatman, ISFNT-5.
- [28] A. Sayano, F. Sutoh, S. Suyama, Y. Itoh, S. Nakagawa, *J. Nucl. Mater.* 271–272 (1999) 467.
- [29] G.E. Youngblood, D.J. Senor, T.H. Jones, N.D. Witold Kowbel, presented at 9th Int. Conf. on Fusion Reactor Materials, Colorado Springs, CO, USA, Oct. 1999.
- [30] W. Kowbel, C.A. Bruce, K.L. Tsou, K. Patel, J.C. Withers, G.E. Youngblood, these Proceedings, p. 570.
- [31] K. Yoshida, T. Yano, these Proceedings, p. 560.
- [32] T. Kameda, A. Sayano, N. Amiji, H. Ichikawa, H. Hamada, A. Fujita, T. Uozumi, *Ceram. Eng. Sci. Proc.* 18 (1997) 419.
- [33] W.S. Stefier, *Ceram. Eng. Sci. Proc.* 14 (1993) 1045.
- [34] T. Ishikawa, S. Kajii, Y. Kohtoku, T. Yamamura, *Ceram. Eng. Sci. Proc.* 18 (3) (1997) 771.
- [35] T. Ishikawa, S. Kajii, T. Hisayuki, Y. Kohtoku, *Ceram. Eng. Sci. Proc.* 19 (3) (1998) 283.
- [36] T. Tanaka, N. Tamari, I. Kondoh, M. Iwase, *Ceram. Eng. Sci. Proc.* 18 (1997) 303.
- [37] M. Tanaka, Y. Imai, H. Ichikawa, Y. Kagawa, H. Iba, H. Kawisawa, *Ceram. Eng. Sci. Proc.* 18 (1997) 779.
- [38] S.D. Connery, L.L. Snead, D. Stainer, presented at 9th Int. Conf. on Fusion Reactor Materials, Colorado Springs, CO, USA, Oct. 1999.
- [39] R. Yamada, T. Taguchi, J. Nakano, N. Igawa, *Ceram. Eng. Sci. Proc.* 20 (1999) 273.
- [40] L.L. Snead, S.J. Zinkle, W.S. Eatherly, D.K. Hensley, N.L. Vaughn, J.W. Jones, presented at 9th Int. Conf. on Fusion Reactor Materials, Colorado Springs, CO, USA, Oct. 1999.
- [41] L.L. Snead, J.C. Hay, *J. Nucl. Mater.* 273 (1999) 213.
- [42] R. Scholz, *J. Nucl. Mater.* 258–263 (1998) 1533.
- [43] R. Scholz, G.E. Youngblood, these Proceedings, p. 372.
- [44] G.E. Youngblood, C.H. Henager, Jr., R.H. Jones, Fusion Materials, Semiannual Progress Report for Period Ending, 31 December, 1996, DOE/ER-0313/21 117.
- [45] L.L. Snead, Y. Katoh, A. Kohyama, J.L. Bailey, N.L. Vaughn, R.A. Lowden, these Proceedings, p. 551.
- [46] L.L. Snead, R. Yamada, K. Noda, Y. Katoh, S.J. Zinkle, W.S. Eatherly, A.L. Qualls, these Proceedings, p. 545.
- [47] T. Noda, H. Araki, S. Ito, M. Fujita, K. Maki, in: Proceedings of the Second IEA/JUPITER Joint International Workshop on SiC/SiC Ceramic Composites for Fusion Applications, October 1997, Sendai, p. 121.
- [48] Y. Katoh, T. Hinoki, A. Kohyama, T. Shibayama, H. Takahashi, L.L. Snead, S.J. Zinkle, presented at 9th Int. Conf. on Fusion Reactor Materials, Colorado Springs, CO, USA, Oct. 1999.
- [49] S. Nogami, A. Hasegawa, K. Abe, T. Taguchi, R. Yamada, these Proceedings, p. 268.
- [50] A. Hasegawa, B.M. Oliver, S. Nogami, K. Abe, R.H. Jones, these Proceedings, p. 811.
- [51] P. Jung, *J. Nucl. Mater.* 191–194 (1992) 377.
- [52] A.J. Frias Rebelo, H.W. Scholz, H. Kolbe, G.P. Tartaglia, P. Fenici, *J. Nucl. Mater.* 258–263 (1998) 1582.
- [53] A. Hasegawa, M. Saito, K. Abe, R.H. Jones, *J. Nucl. Mater.* 253 (1998) 31.
- [54] H.W. Scholz, A.J. Frias Rebelo, D.G. Rickerby, P. Krogul, W.E. Lee, J.H. Evans, J.H. Penici, *J. Nucl. Mater.* 258–263 (1998) 1572.
- [55] A. Hasegawa, M. Saito, S. Nogami, K. Abe, R.H. Jones, H. Takahashi, *J. Nucl. Mater.* 264 (1999) 355.
- [56] C.A. Lewinsohn, G.E. Youngblood, C.H. Henager Jr, E.P. Simonen, R.H. Jones, these Proceedings, p. 584.

- [57] L.A. Giannuzzi, C.A. Lewinsohn, *Ceram. Eng. Sci. Proc.* 20 (1999) 115.
- [58] T. Shibayama, G. Wei He, H. Takahashi, Y. Katoh, A. Kohyama, *Ceram. Eng. Sci. Proc.* 20 (1999) 161.
- [59] A. Donato, S. Casadiom, A. La Berbera, G. Filacchioni, A. Masci, I. Moreschi, A. Nannetti, A. Orsini, F. Salvi, ENA Contribution to EURATOM Long-Term Program materials, 1995–1988.
- [60] A. LaBarvera, B. Riccardi, A. Donato, A. Orsini, I. Moreschi, F. Salvi, S. Casadio, G. Filacchioni, A. Masci, C.A. Nannetti, F. Padella, *Fusion Technology 1998 – 20th SOFT Marseille*.
- [61] H. Kleykamp, *these Proceedings*, p. 1385.
- [62] M. Singh, *Scripta Materialia* 37 (1997) 1151.
- [63] M. Ferraris, M. Salvo, C. Isora, M.A. Montorsi, A. Kohyama, *J. Nucl. Mater.* 258–263 (1998) 1546.
- [64] Y. Katoh, M. Kotani, A. Kohyama, M. Montorsi, M. Salvo, M. Ferraris, *these Proceedings*, p. 1262.
- [65] A.S. Fareed, C.C. Cropper, B.R. Rossing, *Ceram. Eng. Sci. Proc.* 20 (1999) 61.
- [66] R.A. Lowdon, *Ceram. Trans.* 19 (1991) 619.
- [67] C. Droillard, J. Lamon, *J. Am. Ceram. Soc.* 79 (1996) 849.
- [68] F. Rebillat, J. Lamon, R. Naslain, E. Lara-Curzio, M.K. Ferber, T.M. Besmann, *J. Am. Ceram. Soc.* 81 (1998) 2315.
- [69] S. Bertrand, P. Forio, R. Paillet, J. Lamon, *J. Am. Ceram. Soc.* 82 (1999) 2465.
- [70] T. Suzuki, T. Yano, T. Mori, H. Miyazaki, T. Iseki, *Fus. Technol.* 27 (1995) 314.
- [71] Z. Zhu, P. Jung, *J. Nucl. Mater.* 212–215 (1994) 1081.
- [72] M.C. Osborne, C.R. Hubbard, L.L. Snead, D. Steiner, *J. Nucl. Mater.* 253 (1998) 67.
- [73] D.J. Senior, G.E. Youngblood, C.E. Moore, D.J. Trimble, G.A. Newson, J.J. Woods, *Fus. Technol.* 30 (1996) 943.