

## REVIEW OF SiC<sub>f</sub>/SiC COMPOSITES FOR FUSION REACTORS

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### ABSTRACT

SiC<sub>f</sub>/SiC composites are being considered as a candidate structural material for fusion reactors due to their low induced radioactivity by 14 MeV neutron irradiation, high temperature strength and low activation properties. Use of these materials need a strong research and development effort in order to solve some critical issues such as thermal conductivity, radiation stability, hermeticity, chemical compatibility with the fusion environment. The purpose of this paper is to present an overview of current research in material development, properties and irradiation response of new SiC<sub>f</sub>/SiC composites.

**Key words:** SiC<sub>f</sub>/SiC composites, fusion, thermal conductivity, radiation stability.

### FÜZYON REAKTÖRLERİ İÇİN SiC<sub>f</sub>/SiC KOMPOZİTLERİNİN İNCELENMESİ

#### ÖZET

SiC<sub>f</sub>/SiC kompozitleri 14 MeV'lik nötron ışıması altında oluşan düşük radyoaktivitelerinden, yüksek sıcaklık dayanımlarından ve düşük aktivasyon özelliklerinden dolayı füzyon reaktörleri için aday yapı malzemesi olarak düşünülmektedir. Bu malzemelerin kullanımı ısı iletkenlik, ışıma kararlılığı, sızdırmazlık, füzyon ortamı ile kimyasal uyumluluk gibi bazı önemli problemleri çözmek için yoğun bir araştırma ve geliştirmeye ihtiyaç duymaktadır. Bu makalenin amacı yeni SiC<sub>f</sub>/SiC kompozitlerinin özellikleri, ışıma davranımı, ve malzeme geliştirilmesindeki mevcut araştırmaların genel bir incelemesini sunmaktır.

**Anahtar kelimeler:** SiC<sub>f</sub>/SiC kompozitleri, füzyon, ısı iletkenlik, ışıma kararlılığı.

#### 1. INTRODUCTION

SiC fiber-reinforced SiC matrix composites (SiC<sub>f</sub> / SiC) are being considered as a candidate structural material for fusion reactors because of their low-induced reactivity by 14-MeV neutron irradiation, their excellent high-temperature strength, and corrosion resistance(1-7). The high thermal efficiency is one of the advantages of an SiC<sub>f</sub>/ SiC material system. 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 (2). There are three general fusion reactor design concepts using SiC<sub>f</sub> / SiC composites. These are ARIES-I, ARIES IV and ARIES-AT (8) designed by USA, DREAM (9-13) designed in Japan and TAURO(14) in the EU. SiC<sub>f</sub> / SiC composites are also considered as first wall structure in PROMETHEUS IFE reactor (15).

Low activation is one of the key issues in developing fusion reactor materials. In general, the three low activation material systems can be listed as increasingly favorable in the following order: SiC<sub>f</sub>/SiC composites – Vanadium alloy – F/M steels(16-18).

Fibers are the backbone of the continuous fiber ceramic matrix (SiC<sub>f</sub>/SiC) composites. Even though composite properties are controlled by several factors besides fiber strength, such as volume fraction of fibers, fiber/matrix interface structure, fiber weave architecture and matrix properties, there are key fiber properties that control radiation resistance, high temperature strength and stability and weaveability. While the fiber diameter and elastic modulus determine the weaveability of the fiber, the creep strength of SiC fibers is directly related to the the density, degree of crystallinity and stoichiometry. Based on the significant degradation of SiC composites, a search for more radiation resistant fibers has been undertaken. Table 1 gives specifics of six candidate fibers including Nicalon for reference. Currently, the most interesting fiber from a radiation damage standpoint is the Hi-Nicalon material, which is processed from the same

polymer precursor as standard Nicalon, but has been cured with electrons rather than oxygen. Another popular commercial SiC fiber is processed by chemical vapor deposition of SiC on to a pitch carbon core. This is the process which is

(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

Table 1. Characteristic of candidate fiber reinforcements(19)

Fiber	Make-up	Structure	Tensile strength $\sigma$ (GPa)	Modulus of Elasticity E (GPa)	Density $\rho$ (g/cc)	Comment
Nicalon	65%SiC, 23%SiO <sub>2</sub> ,11%C	3 nm $\beta$ -SiC	3	220	2.55	Available
Hi-Nicalon	75%SiC, 21%C ~other	~6 nm $\beta$ -SiC	2.8	270	2.74	Limited availability
Textron SCS-6	SiC on C core	faulted $\beta$ -SiC	3.45	370	3.15	Not weavable
Dow Corning SiC	>95%SiC, free C	0.5 $\mu$ m $\beta$ -SiC	2.6	420	~3.1	Not available
MER converted	SiC and free C	~0.1 $\mu$ m $\beta$ -SiC	2.0	-	<3.1	Developmental
Amoco k1100	Graphite	Highly aligned	2.76	900	2.15	Limited availability

used to produce the Textron SCS-6 fiber. Also of interest is the MER converted SiC fiber. The process for conversion follows the general reaction:



There are lower and higher operating temperatures for structural materials in fusion environment. The lower operating temperature limit for SiC<sub>f</sub>/SiC may be due to the ~11% volumetric swelling associated with radiation-induced amorphization or to thermal conductivity degradation issues. Amorphization occurs in SiC at temperatures below ~120 °C and doses above ~1 dpa for fusion reactor-relevant damage rates. The maximum operating temperature for SiC<sub>f</sub>/SiC due to void swelling concerns is taken to be 990 ± 40 °C(20).

## 2. MANUFACTURING

Several fabrication processes for SiC<sub>f</sub>/SiC composites have been under investigation for more than 10 years to optimize their properties. The chemical vapor infiltration (CVI) process is currently viewed as the industrial leader, but the CVI process is slow with many inherent difficulties such as cost and substantial residual porosity. The polymer impregnation pyrolysis

part. Some results of PIP-processed composite using advanced fibers have been presented. A hybrid process combining CVI and PIP is under investigation 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. 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 through the thickness of composites. Hot pressed (HP) composites have also been studied in an effort to obtain higher density.

Table 2 lists some typical material properties of currently available SiC<sub>f</sub>/SiC composites. Bend strength of Tyrannohex UD composites achieved up to 600 MPa. 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  $3.0 \text{ g/cm}^3$ ) and thermal conductivity (up to  $50 \text{ W/mK}$ ). 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  $\text{SiC}_f/\text{SiC}$  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. Polymer-infiltrated and pyrolyzed SiC coatings also have been studied as a gas permeation barrier of CVI-processed composites. Further studies are required to obtain a hermetic coating for a high-pressure gas system(2). The new  $\text{SiC}_f/\text{SiC}$  composites made by the new PIP processes shows excellent improvements in mechanical properties and attractive potential for large size.

component fabrication. In order to improve the efficiency of the PIP process, a new precursor polymer, poly-vinylsilane (PVS) with SiC filler, was adopted as a matrix precursor and process optimization was performed. Consequently, high-density  $\text{SiC}_f/\text{SiC}$  composite was efficiently produced, which exhibited excellent strength improvement.

Near-stoichiometric SiC was developed by blending PCS and PMS. The Tyranno-Lox M/SiC composites with near-stoichiometric matrix showed excellent improvement in tensile properties and fatigue characteristics at  $1573 \text{ K}$ , which was attributed to the use of PMS and PCS polymers with inorganic powder fillers, BMAS and  $\text{ZrSiO}_4$ , as the matrix precursor (21).

### 3. JOINING AND COATINGS

The  $\text{SiC}_f/\text{SiC}$  component of a fusion reactor cannot be realised directly in the finished form but they have to be realised by assembling small components or half finished products. In particular the TAURO blanket assembly require the joining of small and simple parts (flat and curved panels, T and L sections, etc.). Nevertheless limitations occur in assembling complex geometry  $\text{SiC}_f/\text{SiC}$  elements because welding is not possible before reaching the melting temperature of the SiC fibers, with a partial loss of their mechanical properties, and because diffusion bonding does not seem suitable since the inter diffusion of SiC is very low even at high temperature. For these reasons the availability of a reliable joining technique is fundamental to realise fusion reactors structures. In general the joints shall utilise low activation elements, should operate at high temperature ( $800\text{--}1000^\circ\text{C}$ ), should have radiation stability even

Table 2 Typical properties of various types of unirradiated  $\text{SiC}_f/\text{SiC}$  composites(2)

Method	Fiber	Heat resistant temperature ( $^\circ\text{C}$ )	Tensile strength (MPa)	Work of fracture ( $\text{kJ/m}^2$ )	$K_{IC}$ ( $\text{Mpa}^{1/2}$ )	Thermal conductivity ( $\text{W/mK}$ )
CVI (DuPont)	Hi-Nicalon 2D		217/336 <sup>a</sup>	-	-	-
CVI	Nicalon 2D	1200	215/280	-	27-29	~7 (RT – $1000^\circ\text{C}$ )
CVI (SEP)	Nicalon 2D		285		30	3 (inplane, RT) 2.5 (through thickness, RT)
PIP	Tyranno 3D	1200	400 <sup>a</sup>	4.3-8	11.5-16	-
	Nicalon 2D	1420	240/460 <sup>a</sup>	-	-	-
PIP DOW/Kaiser	Nicalon 2D		207/366 <sup>a</sup>		-	43 (inplane, RT)
RS	Hi-Nicalon UD	1359	500	6.4	-	50 (RT) 30 ( $1000^\circ\text{C}$ )
Fiber-bonded ceramics (Tyranno-hex)	Tyranno UD, 2D	1500	450-550, UD 190-210, 2D	8.9	46.2	4.5 (RT)
	Tyranno-SA UD	1500	600 <sup>a</sup>	1.2	-	50 (RT) 25 ( $1400^\circ\text{C}$ )

<sup>a</sup>Bend strength.

at high temperature and should be chemically compatible with the coolant and breeder. Several joining techniques are under development. They include: assembling by sewing at the textile stage, metallic brazing, homogeneous joining by preceramic polymers, joining by glass and glass ceramic. A brazing alloy series for joining 2- and 3-D SiC<sub>f</sub>/SiC composites has been developed at CEA Grenoble and named Brasic. This brazing compound is composed of low activation elements and was conceived to work at elevated temperatures and to be compatible with SiC. In particular the brazing system contain a sufficient amount of silicon to prevent reaction with the SiC substrate, promote good wetting and to induce some infiltration in the composites. Silicon is associated with reactive elements to improve the joining strength. By using different alloys and compositions (Brasic H2 and V2 or V3) and brazing in vacuum or in an inert atmosphere it was possible to control the infiltration of the alloy. Using Brasic V3 and carrying out the joining at 1300°C in a neutral atmosphere a sound joint was obtained with a perfect filling of the joint gap but no infiltration of the composite. For this joint a shear strength of 174 MPa was obtained at room temperature and about 100 MPa at 800°C. The main limitation of the method rely on the free Si content and the open high porosity of the composite. This low activation brazing alloy can in principle be used for coating SiC<sub>f</sub>/SiC composites.

An homogeneous joining technique has been developed by ENEA and Padua University. This technique is based on the application of a preceramic polymer which pyrolysed at high temperature to provide an adhesive bonding layer consisting of a silicon oxi-carbide phase. Relevant shear strength, measured by means of an almost pure shear test were obtained for a joining sintered  $\alpha$ -SiC (40 MPa). Concerning

SiC<sub>f</sub> /SiC composites (SEP CERASEP N3-1) the best results were obtained using SR 350 silicone resin with Al/Si powders used as additives (31.6 MPa maximum) with pyrolysis carried out at 1200°C.

Ferraris and Salvo from Politecnico of Turin developed a joining technology based on the use of pure silicon giving a room temperature shear strength of 22 MPa. Encouraging results were also obtained using a glass ceramic phase to join SiC

CMCs. In this case a 33 MPa shear strength were reached at room temperature. Glass and glass ceramic compound were also used for coating SiC<sub>f</sub>/SiC CMCs. The formulation was recently optimised in order to give a coating with reduced neutron activation. A double layer coating was set up: the first consists of a glass ceramic phase stable at 800°C and the second consists of a glassy phase which is able to self heal after crack appearance when heated to about 1100°C. (4)

Glass-ceramics can be a versatile joining or coating material with tailorable thermal and mechanical properties; they are not affected by oxidation and can be self-sealant at temperatures above the glass softening point. Calcia-alumina glass-ceramic, referred to as CA is particularly appealing for fusion reactor applications, since it is a Low Activation Material (LAM), has high characteristic temperatures and does not contain boron, lithium and silicon oxides: the boron is incompatible with high neutron flux environment, the lithium is transformed to tritium by neutron irradiation and the silica reacts with the ceramic breeders(22).

#### 4. THERMAL CONDUCTIVITY

Thermal conductivity is important for minimizing the stress in a material exposed to a thermal gradient and for removing heat from energy conversion systems. A very basic problem of the SiC<sub>f</sub>/SiC ceramic composites is their presently insufficient heat transport properties(21). The thermal conductivity of CMCs is generally less than that of their monolithic counterparts because of fiber/matrix interfaces and high porosity. Examples of the thermal conductivity for SiC<sub>f</sub> / SiC composites are shown in Figure 1. At 1000°C, the through-thickness thermal conductivity is about 6 W/mK for SiC/SiC. Westwood and Notis reported the thermal conductivity of high-quality single crystal SiC as 490 W/mK while BeO-doped SiC had a thermal conductivity of 270 W/mK at 25 °C and single crystal SiC a conductivity of 90 W/mK at 25 – 150 °C. Thermal conductivity of polycrystalline SiC is a function of variables such as impurity content, processing method, but has been reported to be 12 to 35 W/mK. Hollenberg et al.

Studied radiation effects on the conductivity of SiC<sub>f</sub>/SiC composites for damage levels of 25

dpa at 800°C. Thermal conductivity was reduced from 7 W/mK after irradiation. Although both the unirradiated and irradiated thermal conductivities are relatively low, the key parameter is the thermal stress factor. (3)

The low thermal conductivity of SiC<sub>f</sub>/SiC composites is an important issue regarding their use in fusion systems. While some composite materials have been fabricated with room temperature thermal conductivities in the range of 100 Wm<sup>-1</sup> K<sup>-1</sup> (through the use of BeO presumably as a sintering aid during hot pressing) the conductivity of typical CVI SiC composite is unacceptably low for thermal management applications such as the first wall of a fusion reactor. It should also be noted that the thermal conductivity will decrease following neutron irradiation(19). The use of SiC matrix/graphite fiber composites are attractive for use in fusion systems for several reasons. Firstly, though development of this system has been limited, some authors have reported these materials to have similar strength to SiC<sub>f</sub>/SiC composites, and in the case of CVI infiltration of very high strength 2-d lay-ups of T-300 graphite fiber, composite bend strength as high as 800 MPa have been shown that for CVI infiltrated graphite composite, the fracture toughness is somewhat higher than for SiC<sub>f</sub>/SiC composites. The potential for this composite in fusion applications is that it offers a high thermal conductivity composite with significantly lower tritium retention than the advanced carbon/carbon composites (C/Cs) currently considered for plasma facing components.

The thermal conductivity of the composite system can be engineered to be isotropic or optimized for high conductivity in a single direction, as needed. For the case of the three dimensional composite fabricated in this study, the thermal conductivity was 215 Wm<sup>-1</sup> K<sup>-1</sup> in the direction of the high conductivity fibers. This conductivity is approximately one order of magnitude higher than that of SiC<sub>f</sub>/SiC systems.

It should be mentioned, by simple rule of mixtures, the calculated thermal conductivity for this SiC/graphite fiber composite should be greater than 400 Wm<sup>-1</sup>K<sup>-1</sup> based on manufacturer's quoted fiber conductivity. Due to a processing problem with the early batch from which this fiber was purchased the thermal conductivity of the

composite was significantly reduced from this maximum value. It is reasonable to assume that if the composite were fabricated with a newer batch of fiber, the composite conductivity would be on the order of 400 m<sup>-1</sup>K<sup>-1</sup>. (19)

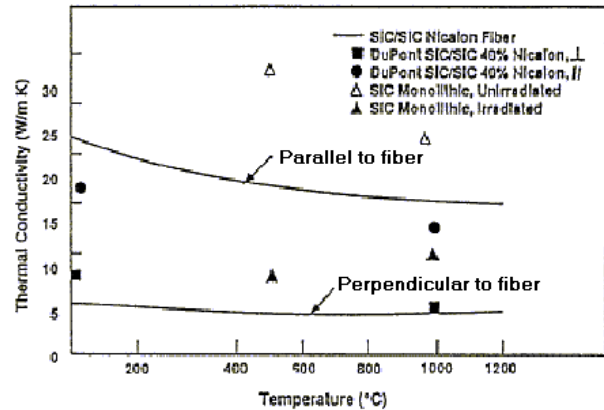


Fig. 1. Thermal conductivity of SiC<sub>f</sub>/SiC composites and monolithic SiC material as a function of temperature. (3)

The CVR-SiC<sub>f</sub>/CVR-SiC composite, 4 mm thick had an RT through-the thickness thermal conductivity of 75 W/m K. This represents almost a seven-fold increase over the state-of-the-art SiC<sub>f</sub>/SiC composite thermal conductivity. The thermal conductivity experiences a parabolic behavior reaching 35 W/m K at 1000°C. The importance of this result can be further emphasized by comparing it with the 6 W/m K, 1000°C thermal conductivity exhibited by the state-of-the-art CVI-matrix SiC<sub>f</sub>/SiC composite. The parabolic decay of the CVR-SiC<sub>f</sub>/CVR-SiC coupon's thermal conductivity is typical of the higher thermal conductivity matrix. (23)

## 5. BENDING STRENGTH

The results of the four point bend tests on all composites fabricated are given in Table 3. For the case of the CVD SiC/Nicalon based fiber composites, a single interfacial coating thickness of 0.3 μm was chosen. This has been shown previously to yield the maximum bend strength for the ceramic grade Nicalon fiber. From the table it is seen that the low-oxygen-content Hi-Nicalon yielded an ultimate bend strength of 348 ± 27 MPa versus the 292 ± 26 MPa achieved for standard Nicalon. The CVD SiC/MER converted fiber system had a substantially lower bend strength than the Nicalon composite. From Table 3

it can be seen that increasing the interfacial thickness from 0.15 to 0.3  $\mu\text{m}$  increased the measured strength from  $123 \pm 6$  to  $144 \pm 9$  MPa.

Fig. 2b. Flexure curves of MER fabric composites (19)

Table 3. The results of the four point bend tests on  $\text{SiC}_f/\text{SiC}$  composites(19)

Fiber	Interface ( $\mu\text{m}$ )	Volume Fraction of Fiber (%)	$\sigma$ UTS (MPa)	Std. dev. (MPa)	Density (g/cc)	Void (%)
Nicalon	~0.3	41	292	26	2.5	~14
Hi-Nicalon	~0.3	42	348	27	2.6	~13
Nicalon-MER	~0.3	34	221	24	2.5	~19
MER T-300	~0.15	~33	123	6	2.3	~20
MER T-300	~0.3	~34	144	9	2.4	~19
MER T-300	~1.0	~28	123	18	2.1	~30
MER T-300 <sup>a</sup>	~0.3	~32	152	1	2.1	~15
Amoco k1100	~0.3	44	283	30	2.1	~20

The CVD  $\text{SiC}/\text{K1100}$  graphite fiber system yielded quite good bend strength considering no process optimization was conducted (due to limited availability of material). The bend strength in this case was found to be  $267 \pm 26$  MPa with the major fiber direction parallel to the axis of the bend bar. The flexure curves for all materials tested are shown Fig. 2(19).

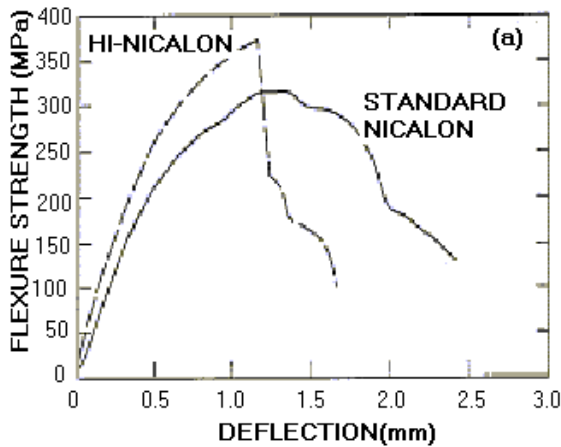


Fig. 2 a. Flexure curves of Nicalon fabric composites (19)

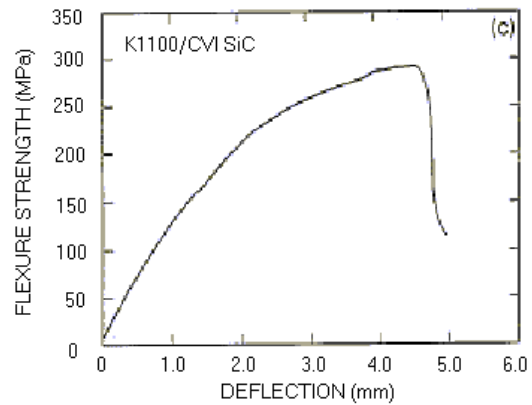
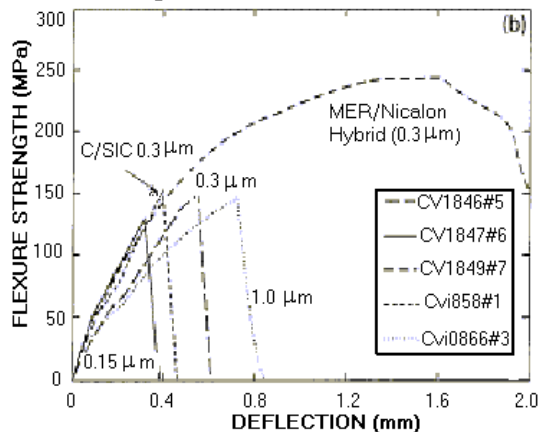


Fig. 2c. Flexure curves of K1100 fiber composite (19)

**6. HERMETICITY**

Processing methods during manufacture are responsible for about 10 to 15% porosity in most CMCs (ceramic matrix composites). These materials are also prone to matrix cracking in response to applied stress, thermal cycles or radiation. As a structural material,  $\text{SiC}_f/\text{SiC}$  must perform as a pressure boundary and must therefore maintain a minimal in-leakage of coolant into the plasma chamber. Jones, Conn and Shafer(3) estimated that a maximum helium in-leakage rate of 0.1 mol/s would be tolerable. However, Jones estimated that microcracking in a  $\text{SiC}_f/\text{SiC}$  first wall could allow as much as  $7 \times 10^5$  mol/s for the case where all the pores in the composite are linked by channels of a dimension equal to the average pore size. A leak rate of 0.1 mol/s is achieved for the condition where all pores are connected by cracks opened in a direction

perpendicular to the crack to a width of 0.3 nm. This small crack opening will increase with application of a stress and subsequent creep processes.

Potential solutions to this problem include the application of a low-activation, low-strength metal layer or a self-healing glass layer on the coolant side of the first wall. The low-strength metal layer must exhibit significant irradiated ductility such that cracks do not form. Likewise, the self-healing glass layer must have a sufficiently low viscosity to prevent cracking. The seal coat of SiC commonly applied to the surface of SiC<sub>f</sub>/SiC composites could also help reduce the coolant in-leakage. However, a brittle layer will probably crack during reactor start-up and shut-down cycles, or during radiation so the net effect on leak rate is uncertain. (3)

## 7. ENVIRONMENTAL EFFECTS

### 7.1. Oxidation

Several mechanisms to explain environmentally induced crack growth of SiC<sub>f</sub>/SiC at elevated temperatures, with varying amounts of oxygen have been proposed. 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 SiO<sub>2</sub> 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 O<sub>2</sub> where the SiO<sub>2</sub> 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(2). A microstructural study on crept SiC fiber and SiC<sub>f</sub>/SiC composites in argon gas containing low partial pressure of oxygen using high-resolution electron microscope was reported by Shibayama(2). 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<sub>f</sub>/SiC composites assumed that IRM was operative. However, it will be necessary to verify

the specific crack growth mechanism for SiC<sub>f</sub>/SiC under fusion relevant conditions(2).

### 7.2. Compatibility

The compatibility of SiC<sub>f</sub>/SiC commercial composites with solid breeder beds in fusion relevant conditions was investigated. Solid Li<sub>4</sub>SiO<sub>4</sub> and Li<sub>2</sub>TiO<sub>3</sub> 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 Li<sub>4</sub>SiO<sub>4</sub> and Li<sub>2</sub>TiO<sub>3</sub> up to 10 000 h at 800°C in flowing He containing 1000 ppm H<sub>2</sub> gas. After 10 000-h exposure to Li<sub>4</sub>SiO<sub>4</sub>, lithium metasilicate was found on the samples. 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 Li<sub>2</sub>TiO<sub>3</sub>, the protective coating was partially eroded after 10 000 h of exposure. The SiC layer was full 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<sub>f</sub>/SiC composites with Be and Li oxide breeder materials was studied by Kleykamp. 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 Be<sub>2</sub>C-Si was observed on the SiC surface of the pellet, because Be<sub>2</sub>C and Si are in thermodynamic equilibrium at 1000°C. Powder reaction experiments were also made between α-SiC and Li<sub>4</sub>SiO<sub>4</sub>, LiZrO<sub>3</sub> and LiTiO<sub>3</sub> 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<sub>f</sub>/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<sub>f</sub>/SiC with respect to Pb-17Li. The data are from a test performed at 800°C in stagnant liquid Pb-17Li using 2D SiC<sub>f</sub>/SiC composites(2).

## 8. THERMAL FATIGUE AND SHOCK BEHAVIOR

Structural materials in fusion reactors will experience thermal fatigue from reactor start-up and shut-down cycles and thermal shock from plasma discharge. Thermal fatigue and thermal shock are important properties of ceramic composites for fusion energy systems. The fatigue and thermal shock behaviors of ceramics have been reviewed by Jones et al.(24). The thermal fatigue of a Nicalon-fiber-reinforced SiC<sub>f</sub>/SiC composite was evaluated by Jones and Henager(25) using low-cycle mechanical fatigue. It was found that the crack velocity decreases with increasing fatigue cycles in a manner similar to that observed for subcritical crack growth with time. There was a factor of 10 decrease in the load cycle between the 1<sup>st</sup> and 25<sup>th</sup> cycles at a stress intensity of 18 MPam<sup>1/2</sup>. Subcritical crack growth decreases with time because additional bridging fibers accumulate behind the crack tip. Fiber bridging is a toughening mechanism in fiber-reinforced composites that delays the crack growth. At higher stress intensities the crack velocity after 1 and 25 load cycles converges. There have been several studies of high cycle fatigue of continuous fiber reinforced composites(26-28), that support the observation that these materials exhibit a fatigue stress threshold below which crack growth does not occur. This stress threshold is approximately equal to the matrix cracking stress. Above this threshold stress, Holmes(26) has shown that, at 1200°C and 10Hz, the number of cycles to failure is a function of the R ratio and peak stress. For the material studied by Holmes(26), the matrix cracking stress was 200 MPa and the ultimate strength was 380MPa at 1200°C.

The thermal shock response of materials is very dependent on the temperature change and change rate and the constraint on the sample or component. For continuous fiber-ceramic matrix composite, the thermal shock behavior also depends on whether the temperature change is positive or negative. Most thermal shock data are for samples quenched from an elevated temperature; however, in a fusion reactor a plasma discharge will result in rapid heating. Ceramic composite properties are not strain rate dependent, other than in the creep strain rate range, so the

heating rate should be a concern. Thermal shock tests with a positive temperature change were performed by Eckel(29) and Wang and Singh(30). Eckel measured the thermal shock of monolithic and composite material heated from 1300°C to 2300°C in a burner rig. The monolithic material failed after 1.5 cycles while the SiC<sub>f</sub>/SiC composite survived 25 cycles without loss of strength. However, with a heating rate of 1900°C/s the composite exhibited a 35% strength loss as compared to no strength loss at a heating rate of 1700°C/s(31).

## 9. IRRADIATION EFFECTS

Neutron irradiation effect studies on SiC<sub>f</sub>/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(2), reported that the fracture strength decreased rapidly with increasing fluence plateau at about 5 dpa with little further decrease up to 80 dpa.

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. 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(2).

Crystalline, β-SiC has a well established response to radiation where the dimensional or

density change is temperature dependent and saturates at a relatively low fluence. The dimensional change at 200°C is ~0.3% (linear expansion), decreasing to nearly zero dimensional change at 1000°C. The lone exception to this is Sylramic which swells like crystalline SiC. Crystal growth and oxygen loss are two processes that have been proposed to explain the radiation-induced changes in density of SiC fibers. The polymer derived Nicalon fibers are not fully crystallized but have nanosized crystals embedded in an amorphous Si-C-O matrix. Nicalon-CG has the highest concentration of oxygen and exhibits



the greatest radiation-induced shrinkage. Hi-Nicalon has lower oxygen concentrations; however, Hasegawa et al. (7) observed a growth in crystal size from 1 to 5 nm in Nicalon-CG following irradiation with a fast neutron fluence of  $3.4 \cdot 10^{26} \text{ n m}^{-2}$  (43 dpa) at 1040°C, while the crystal size of Hi-Nicalon remained constant at ~5 nm following irradiation at the same conditions. Hi-Nicalon is not fully crystallized so density changes and crystal growth could be expected with increasing dose(32).

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.

## 10. HELIUM EFFECTS ON SiC<sub>f</sub>/SiC COMPOSITES

In a fusion neutron spectrum, helium will be produced at the rate of about 1500 - 2000 appm MWy/m<sup>2</sup> in SiC by transmutation reactions, with a concurrent displacement damage of 10-15 dpa/(MWy/m<sup>2</sup>), depending on the details of the blanket structure and neutron spectrum. In the end of its life (about 100 dpa), the He concentration will be 15000-20000 at.ppm. As the solubility of He is almost zero in all materials, and it stabilizes vacancy clusters, microstructural changes such as swelling and strength degradation may be accelerated in SiC<sub>f</sub>/SiC composites under fusion reactor conditions. The impact of this large helium transmutation rate on the properties of SiC<sub>f</sub>/SiC composites is a significant issue in their application(33).

## 11. CONCLUSIONS

The use of low activation SiC<sub>f</sub>/SiC composite as structural material in a fusion reactor is attractive based on its low induced radioactivity, low afterheat, high temperature properties and excellent corrosion resistance.

The improvement of both thermal conductivity and stability of thermo-mechanical properties after irradiation remain the main issue of SiC<sub>f</sub>/SiC research and development. The constant progress in fiber quality which led to the fabrication of almost stoichiometric fibers is a good premise for reducing such concern. However, a large effort has to be made for the matrix-fiber interface and matrix processing itself in order to

reduce the differences in performances with respect to the bulk CVD SiC. This objective may probably be achieved by using processing parameters.

Joining and coating techniques and hermeticity need further developments to study their compatibility with fusion environments and to improve their performance. And also lifetime of modern SiC<sub>f</sub>/SiC composites that requires the necessary database has to be determined.

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