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# Issues and advances in SiC<sub>f</sub>/SiC composites development for fusion reactors

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# Abstract

 $SiC_f/SiC$  composites are among the most promising candidate structural materials for fusion because of their potential application for high performance reactors and superior safety characteristics compared to metallic materials. Favourable features of  $SiC_f/SiC$  composites are the high temperature properties and the low activation characteristics at short and medium term. Conversely, the material has some critical issues such as the remarkable gas production due to nuclear transmutation and properties degradation induced by radiation exposure. Continuous progress in R&D and in particular the availability of advanced fibres and improved processing methods, as well as alternative solutions for fibre–matrix interfaces, has led to composites with higher thermo-mechanical characteristics and better radiation stability. This paper reports the issues of fusion reactor studies, the progress in material R&D and the latest results of radiation exposure studies.

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# 1. Introduction

Continuous SiC fibre reinforced-SiC matrix ceramic composites (SiC<sub>f</sub>/SiC) are the primary composite material under development as a structural material for fusion power reactors (FPR). The increasing interest in SiC<sub>f</sub>/SiC is related to its potential high performances at elevated operating temperature. Although the material development presents high risks, mainly connected to the irradiation performances, the potential high pay off is incentive for an expanded R&D effort to assess the material suitability for FPR [1].

Favourable features of SiC<sub>f</sub>/SiC composites are the engineerability to produce a specific component, the material strength and fracture properties retained to high temperature (namely 1000–1200 °C depending on fibre and fibre interphase), the thermal shock and thermal fatigue resistance, the high corrosion resistance, the low activation characteristics at short and medium term and low after heat and the limited saturation swelling values up to 1000 °C. Critical issues of the material concern basic issues, such as the nuclear transmutation and physical and mechanical properties radiation stability, and technological issues such as hermeticity and joining.

The continuous progress in R&D, also driven by the development for different applications such as aerospace and energy conversion systems [2], give rise to some confidence for obtaining a reliable material for fusion.

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Different blanket systems and fusion reactor conceptual studies have considered  $SiC_f/SiC$  composites as structural material for in-vessel components.

This paper first summarizes the issues of fusion reactors studies and the related material requirements. Afterwards, the recent advancement in material development and the recent results of irradiation experiments are reviewed. Finally, some technological issues are presented.

# 2. Design requirements

The use of  $SiC_f/SiC$  composites as structural material for in-vessel components and, in particular for breeding blankets, increases the potential of safety and environmental impact of DT-FPR.

The low activation and afterheat levels associated with SiC<sub>f</sub>/SiC after long-term neutron irradiation lead to FPR components designs showing high safety standards and simplified maintenance schemes. In addition, the excellent SiC<sub>f</sub>/SiC chemical stability at high temperature could lead to minimal mobilization of radioactive products.

Furthermore, the high application temperature of  $SiC_f/SiC$  improve energy handling capabilities, thus allowing the use of high temperature coolant with the potential for high energy conversion efficiency (>50%).

#### 2.1. Blanket design concepts

In recent years, several blanket designs have been studied worldwide in order to exploit the potential of SiC<sub>f</sub>/SiC composites [3], in particular the TAURO blanket concept in the European Union, the ARIES-AT concept in the US and the DREAM concept in Japan. The first two concepts are self-cooled lithium-lead blankets, while DREAM is an helium-cooled beryllium/ ceramic blanket. Both TAURO and ARIES-AT blankets are essentially formed by a SiC<sub>f</sub>/SiC box acting as a container for the lithium-lead which performs the simultaneous functions of coolant, tritium breeder, neutron multiplier and, finally, tritium carrier. The DREAM blanket is characterized by small modules using pebble beds of Be as neutron multiplier material, of Li<sub>2</sub>O (or other lithium ceramics) as breeder material and of SiC as shielding material. The He coolant path includes a flow through the pebble beds and a porous partition wall.

These studies have shown that, although the performances of these blankets are very promising, the properties and behaviour of  $SiC_f/SiC$  composites need to be substantially improved before the feasibility of such blankets can be demonstrated [4].

These conclusions have been confirmed by the most recent study on  $SiC_f/SiC$ -based blanket, the SCLL (self-

cooled lithium–lead) blanket developed in the framework of the on-going EU Power Plant Conceptual Study [5]. The SCLL blanket concept is based on the most attractive features of TAURO and ARIES-AT. The corresponding power plant thermal efficiency has been estimated to be 61%. SiC<sub>f</sub>/SiC composite structures have been assumed also for shield and divertor. The assumed properties of SiC<sub>f</sub>/SiC are shown in Table 1 where the corresponding value shown by present-day composites are given.

# 2.2. Required properties and performances for $SiC_f/SiC$ composites

The development of conceptual design has identified a set of satisfying SiC<sub>f</sub>/SiC properties which can be used as guidelines for defining R&D priorities. Table 1 gives an idea of the kind and level of properties improvement which are required. Other improvements concern functional aspects such as irradiation resistance, compatibility and fabrication techniques. In general, the most important requirements identified are the following:

- improvement of thermal conductivity, especially through the thickness, at high temperature and under neutron irradiation;
- determination and possible improvement of maximum working temperature under irradiation (swelling, compatibility with Pb–17Li);
- confirmation of the out-of-pile mechanical and thermo-mechanical properties after beginning of irradiation;
- determination and improvement of the lifetime; in particular, the effects of C and Si transmutation burn-up needs to be experimentally assessed;
- capability of fabrication of components with homogeneous properties at large dimensions, with a particular attention to the minimum thickness (especially for the divertor design) and maximum thickness (especially for the blanket back-plate);
- development, testing and validation of acceptable joining techniques;
- determination and possible reduction of the electrical conductivity under irradiation;
- establishment of the maximum interface temperature with Pb–17Li under representative flowing conditions and irradiation level; in particular verification that no Pb–17Li infiltration through the SiC<sub>f</sub>/SiC surface will occur, the major risk being an increase in the wall electrical conductivity;
- improvement of hermeticity to Pb–17Li and high pressure helium, which could imply the use of coatings;
- compatibility of brazing material with Pb–17Li.

Moreover, because of the proposed use of  $SiC_f/SiC$ as structural material for a nuclear component having to ensure a reasonably long lifetime is fairly recent, no Table 1

SiCr/SiC properties and data base: comparison between assumed values and typical present-day measured values

Key $SiC_f/SiC$ properties and parameters <sup>a</sup>	Assumed values [1] in the design analyses	Commercial material value (SNECMA)	
Density	$\approx 3000 \text{ kg/m}^3$	$\approx 2500 \text{ kg/m}^3$	
Porosity	≈5%	≈10%	
Young's modulus	200–300 GPa	≈200 GPa	
Poisson's ratio	0.16-0.18	0.18	
Thermal expansion coefficient	$\approx 4 \times 10^{-6}$ /°C	$4 \times 10^{-6}$ /°C	
Specific heat	190 J/kg K	190 J/kg K	
Thermal conductivity in plane (1000 °C)	$\approx 20 \text{ W/m K} (\text{EOL})^{a}$	$\approx 15 \text{ W/m K} (\text{BOL})^{a}$	
Thermal conductivity through thickness (1000 °C)	$\approx 20 \text{ W/m K} (\text{EOL})^{a}$	$\approx$ 7.5 W/m K (BOL) <sup>a</sup>	
Electrical conductivity	$\approx 500 \ (\Omega m)^{-1}$ (under irradiation,	$\approx 500 \ (\Omega m)^{-1}$ (before irradiation)	
	EOL <sup>a</sup> value)		
Tensile strength	300 MPa	300 MPa	
Trans-laminar shear strength	_	200 MPa	
Inter-laminar shear strength	_	44 MPa	
Maximum allowable tensile stress	Not used <sup>a</sup>	Unknown <sup>a</sup>	
Max. allowable temperature (irradiation swelling basis)	≈1000 °C	≈1000 °C	
Maximum allowable interface temperature with breeder	$\approx 1000$ °C (flowing)	$\approx 800$ °C (static)	
Min. allowable temperature (thermal conductivity basis)	≈600 °C	$\approx 600 \ ^{\circ}\text{C}$	
Cost	≪\$400/kg	$\approx 10$ times larger	

<sup>a</sup> BOL = beginning of life, before irradiation; EOL = end of life, after irradiation.

adequate modelling and design criteria are yet available. Some preliminary work has been performed [6] aimed at identifying appropriate models available in aerospace research field and at theoretically defining sound design criteria to improve the thermo-mechanical design analyses. The analyses performed for the SCLL design described above are based on these preliminary findings, however a significant improvement is still required.

In case of modelling, several theories developed for aerospace applications are under evaluation and comparison; the implementation of the most appropriate one in available design codes will allow, in the short term, one to obtain more realistic composites structures models and, consequently, more reliable thermomechanical analyses. However, it is already known that the new models will require a large set of experimental data concerning the composite actually being used as structures which is not available at present. Also, for the validation of appropriate design criteria, a large number of tests are required to be performed under relevant geometry and operating conditions. It must be noted that the design criteria presently used [6] for the analyses have been derived from established design criteria for metals and adapted for composite structures without submitting them to any experimental validation.

# 3. Material status

#### 3.1. Manufacturing

The SiC<sub>f</sub>/SiC composite R&D for fusion reactors is focused on the development of advanced composites

with high density, high purity and improved temperature limits and their characterization in terms of mechanical properties, lifetime and irradiation performance.

 $SiC_f/SiC$  composite performance is highly dependent on properties of the fibre reinforcement. Moreover, fibre texture characteristics are important for the composite loading capability. SiC fibre properties have been improved by reducing the content of oxygen and the excess of carbon [7]. Recently, stoichiometric and high crystallinity SiC fibres have been developed (Hi-Nicalon S type, Tyranno SA) by reducing the excess of carbon up to a C/SiC ratio close to the stoichiometric composition [8]: in this way high elastic modulus, high thermal conductivity and excellent creep resistance have been obtained. The properties of such fibres have been reported elsewhere [1,9]. It has been proved that also complex 3D architectures can be made by using such fibres [10,11].

The composite mechanical behaviour can be controlled by the deposition on the fibres of a coating (fibrematrix interphase) of convenient thickness and strength [12]. For fusion applications, carbon (graphitic or glassy) is the most frequently used interphase material. It is well understood that graphite is much less tolerant to neutrons than SiC and the irradiation effects are severe [13]. The influence of interphase damage on the composite performances may be minimized by minimizing the use of carbon. SiC-based interphases, e.g., pyrolithic carbon–SiC multilayered interphase  $(PyC/SiC)_n$  and pseudo-porous SiC interphase, have been proposed for fusion  $SiC_f/SiC$  with this purpose [14]. The multilayered interphase was originally developed to improve oxidative resistance and is reported to be effective for prolonged static rupture time and fatigue lifetime [15]. Tyranno SA/CVI SiC matrix composites have been manufactured with PyC or  $(PyC-SiC)_n$  interlayers [16]. The results indicate that PyC-SiC multilayers cause no obvious change in the Tyranno SA fibre reinforced composites, although these composites showed slightly larger interfacial shear strength compared with those of PyC layered ones. Beside these complex interphases, a simpler 'very thin' carbon interphase appears to be applicable to advanced SiC fibre composites [17]. Structural optimization and extensive characterization of very thin carbon interphase composites are presently underway.

Several processes are available for the production of SiC<sub>f</sub>/SiC composites, which include chemical vapour infiltration (CVI) polymer infiltration and pyrolysis (PIP) reaction sintering (RS) and melt infiltration (MI).

CVI is the leading process to fabricate SiC<sub>f</sub>/SiC composites [2], in spite of the slow deposition rate and residual porosity. This process allows for the production of radiation-resistant stoichiometric crystalline β-SiC matrix and has other important advantages such as a modest deposition temperature and a high flexibility. Moreover, different versions of the technique are available and they provide efficiency improvement of the basic CVI process [18]. Tyranno SA-composites have been manufactured also by means of forced flow-thermal gradient chemical vapour infiltration (FCVI), which allows for the fabrication of composites in relatively short time [19]. The composites manufactured showed a density up to 2.7 g/cm3 and mechanical properties comparable with those obtained by CVI.

As to matrix densification processes for SiC<sub>f</sub>/SiC other than CVI, polymer impregnation and pyrolysis (PIP) as well as a variety of direct conversion processes (meltinfiltration MI, reaction sintering RS, hot pressing HP, etc.) have long been studied and - to some extent industrialized [20-22]. These techniques have demonstrated respective advantages and disadvantages which arise from their processing routes. A major disadvantage for the fusion application lies in the irradiation instability of the matrix second phases such as silicon, carbon, silica and amorphous (-like) SiC. Although it may be very difficult to overcome this issue, there are good premises in order to minimize the influences of irradiation damage to second phases [23]. In particular, the availability of advanced fibres, which allows for a higher pyrolysis temperature with modest fibre degradation and improved preceramic polymers (AHPCS-Starfire Systems, USA) [24], able to be converted in stoichiometric crystalline SiC, makes the PIP process quite attractive. The development of 2D and 3D composites by hybrid CVI/PIP process has also been carried out [25]. Densities up to 2.7 g/cm<sup>3</sup> and high thermal conductivity have been obtained. A similar activity was carried out on composites produced by a hybrid CVI/PIP process and SiC powders injection [26].

A novel SiC<sub>f</sub>/SiC composite has been recently developed by means of 'nano-infiltration and transient eu-

Fig. 1. Microstructures of innovative SiC<sub>f</sub>/SiC ceramic composite by NITE process.

tectic-phase (NITE)' process [27,28]. The NITE process makes use of so called liquid phase sintering for silicide ceramics by using small amount of metal oxide additives, that is one of the common production processes for monolithic SiC. The liquid phase sintering has long been considered inapplicable to  $SiC_f/SiC$  processing due to the sintering conditions being too harsh for SiC-based fibres. The successful development of NITE owes to sintering condition optimization, appropriate fibre protection, and emergence of the advanced SiC fibres [29]. In this process, pyrolytic carbon coated Tyranno SA fibre performs are infiltrated with β-SiC nano-powders and small amounts of sintering aids (Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub>) and afterwards a thermal process is carried out at high temperature (1750-1800 °C) under pressures ranging from 15 to 20 MPa (Fig. 1). The matrix of NITE  $SiC_f/SiC$  is comprised of polycrystalline SiC and small amount of isolated oxide grains. The characterization of laboratory grade material has shown that the composite has interesting characteristics (see Table 2): very high density (from 0.92 to almost 1 of the theoretical density) promising mechanical properties (tensile strength of about 400 MPa), interesting thermal conductivity and very low gas permeability. Pilot commercial production is in progress. Neutron irradiation programs are being planned for year 2004 as a part of JA-EU IEA collaboration and US-JA JUPITER-II collaboration. A commercialization of Tyranno<sup>TM</sup>-SA NITE SiC<sub>f</sub>/SiC is being promoted at Ube Industries, Ltd. (Japan).

# 3.2. Thermal conductivity

Thermal conductivity and particularly throughthickness conductivity plays a fundamental role for the



Material	GA Hi-Nicalon <sup>TM</sup> /FCVI (conventional CVI)	ORNL Tyranno <sup>TM</sup> -SA/FCVI (advanced fibre CVI)	Kyoto University NITE
Reinforcement			
Architecture	2D P/W, 0/90°	2D P/W, 0/90°	Unidirectional
Fibre volume fraction	35–40%	35–40%	20–25%
Tensile properties			
Ultimate stress	$\sim 200 \text{ MPa}$	~250 MPa	~400 MPa
Proportional limit	$\sim 70 \text{ MPa}$	$\sim 70 \text{ MPa}$	~220 MPa
Modulus	~150 GPa	~200 GPa	~300 GPa
Thermal conductivity <sup>a</sup>			
20 °C	$\sim 4 \text{ W/m K}$	$\sim 15 \text{ W/m K}$	$\sim$ 30 W/mK
1000 °C	$\sim 2 \text{ W/m K}$	$\sim 10 \text{ W/m K}$	$\sim 20 \text{ W/m K}$

Representative mechanical	property data f	or FCVI and	NITE SiC <sub>f</sub> /SiC	composites

<sup>a</sup> Through thickness.

application of  $SiC_f/SiC$  composites for fusion reactor high heat flux components. The thermal conductivity degradation due to neutron irradiation increases the criticality of this property.

Therefore, a substantial effort on material development has focused on the improvement of this property. Thermal conductivity of unirradiated silicon carbide is determined by phonon-phonon interaction and phonon scattering by various defects like lattice defects, interfaces and impurities [12]. However, the irradiated thermal conductivity is primarily governed by phonon scattering at irradiation-produced defects. In composites, thermal resistance factors arising from porosity, matrix second phases and interfaces/interphases are added. Stoichiometric fibres with three-dimensional texture, optimized interphases and dense matrices (with very low and favourable porosity distribution) allowed for a relevant improvement of conductivity. Today, composites with high conductivity (interesting for fusion first wall application) are available: they are manufactured by PIP-chemical vapour reaction technology [25] generally through high temperature processes. Unfortunately, these composites exhibit low mechanical properties and a matrix that is not fully stoichiometric and crystalline. The thermal conductivity performances of composites manufactured by means of PIP-CVR are reported to be as high as 80 W/m K at RT and 40 at 1000 °C [30], but the irradiation behaviour needs to be assessed. Superior thermal conductivity performances have also been reported for composites with Hi-Nicalon S and Tyranno SA fibres, manufactured by means of PIP technique [25]. These composites reached about 50 W/m K thermal conductivity at RT and 20 W/m K at 1000 °C. The conductivity of NITE composite was found to be 30 W/m K at RT, notwithstanding a density as high as 3 g/cm<sup>3</sup> [29] (Table 2). An alternative route, incorporating heat-piping media along the heat flux direction is considered to be an effective scheme for improved thermal conductivity. As an example, orthogonal 3D composite with graphite z-fibres (through-thickness fibres) is being evaluated [31].

# 3.3. Electrical conductivity

In addition to the above mentioned fusion reactor studies, recently SiC<sub>f</sub>/SiC composites have been also proposed for non-structural application in advanced dual coolant Pb-17Li blanket concept [32] as thermal shielding and electrical insulation material. In all cases, low electrical conductivity composites are desirable in order to minimize the magnetohydrodynamic effect (MHD) that results from the interaction of the moving liquid metal and reactor high magnetic flux [33]. The electrical conductivity of SiC<sub>f</sub>/SiC composites differ significantly from that of monolithic SiC due to the combined effect of fibres, C interphase and matrix. Scholz [34] measured the electrical conductivity of different types of fibres and composites in the temperature range 20-1000 °C. The fibres tested were Nicalon CG, Hi Nicalon, Hi-Nicalon S and Tyranno SA; the composites were made with Nicalon CG or Hi-Nicalon fibres with their matrices produced by CVI or PIP. The results of this study show that electrical conductivity increases within the temperature range analyzed; in particular, more stoichiometric and crystalline fibres showed lower conductivity values (Hi-Nicalon S and Tyranno SA have values lower than 300  $(\Omega m)^{-1}$ . Because of the effect of carbon interlayer, the composites electrical conductivity is higher than the fibre one (up to 550–600  $(\Omega m)^{-1}$  at 1000 °C). Multilayer interphase composites exhibit lower electrical conductivity [35].

# 4. Irradiation studies

### 4.1. Radiation damage in $SiC_f/SiC$

Neutron irradiation induces the displacement of Si and C atoms, with formation of defects such as

Table 2

vacancies and antisite and interstitial defects. Depending on irradiation temperature and dose, the effects of irradiation are amorphisation, point-defect swelling and cavity swelling; these phenomena strongly affect the stability of thermal and mechanical properties of SiC and SiC<sub>f</sub>/SiC composites [9]. A significant amount of data has been produced on the swelling of SiC by early studies in support of gas fission reactors [36].

SiC<sub>f</sub>/SiC composites swelling, thermal conductivity and specially mechanical properties changes have been the subject of a specific study for fusion application. A determined development program has shown that nearstoichiometric SiC fibre composites exhibit very good mechanical property stability up to  $\sim 8$  dpa for temperatures as high as 800 °C [37]. Moreover, single fibre strength [38] and strength of monolithic material [39] has been measured to this dose level, also indicating no significant degradation in mechanical properties. Noting that swelling and mechanical properties for ceramics tend to saturate by this dose level, these materials show promise to much higher dose levels. These higher dose studies are underway. (PyC/SiC)<sub>n</sub> multilayered interphase composites have been proven to have superior microstructural stability under high fluence ion irradiation [40]. Bending test of irradiated and unirradiated composites provided of multilayered and porous SiC interphases applied on old generation fibres has been carried out [41]. The results evidenced that the radiation stability of multilayer C-SiC interphase composite is higher than the porous SiC interphase composite.

Recently, particular emphasis has been placed on the subjects of understanding the degradation in monolithic and composite thermal and electrical conductivity. Thermal conductivity study has included modelling of the composite taking into consideration architecture, porosity and neutron irradiation [42,43] as well as the degradation of high-purity materials [44]. Fig. 2 shows a comparison of thermal conductivity degradation and swelling as a function of irradiation dose. Two interesting conclusions can be drawn from this work. First, the room temperature thermal conductivity has been reduced from about 400 to 20 W/m K at 4.5 dpa for the fusion relevant irradiation temperature of 800 °C. Second, the swelling at 800 °C does not appear to be saturating in contrast with the earlier literature. As the material from Fig. 2 can be considered a near ideal one, the conductivity of irradiated composite will necessarily be lower than this. In fact, plane-weave Nicalon Type S fibre-CVI SiC composite irradiated in HFIR reactor (in the damage dose and irradiation temperature range of 2-4 dpa and 300-800 °C) showed a through-thickness RT temperature conductivity variable from 1.5 to 2.5 W/m K [35]. Similarly, infiltrated Sylramic fibre composite resulted in room temperature conductivity up to  $\sim 5$  W/m K [42].

The study of the effect of ionizing radiation on the electrical conductivity of monolithic SiC has shown that

Fig. 2. Room temperature thermal conductivity and swelling as a function of neutron damage for CVD SiC irradiated in HFIR [35].

- for fusion relevant ionizing dose rates up to  $\sim$ 5 Gy/s the conductivity increase is appreciable only for highly resistive forms of SiC [35]. More conductive forms such as CVD SiC exhibit no 'in situ' increase. In fact, there appears to be no increase in conductivity under ionizing irradiation for materials with base conductivity greater than  $\sim 1 \ (\Omega m)^{-1}$ . Comparing this to the recommended upper conductivity limit of  $<500 \ (\Omega m)^{-1}$  [3], radiation enhanced conductivity does not appear to be an issue. Measurements have been carried out on 2D Nicalon CG-CVI composite subjected to a neutron irradiation at 750 °C up to 5 dpa [34]. This composite showed an unirradiated electrical conductivity of 350  $(\Omega m)^{-1}$  at room temperature and 550  $(\Omega m)^{-1}$  at 1000 °C. The results showed a 20% reduction of the electrical conductivity measured at RT with an almost linear trend with increasing dpa.

### 4.2. Transmutation gases

Neutron irradiation of SiC also give rise to material burn out and transmutation products. Burn out rates of Si and C, estimated for the He-cooled ARIES IV reactor first wall neutron spectrum, are 0.0047/efpy for Si and 0.0042 for C [4]. Transmutation products include gaseous (H, He) or solid atomic species (Mg, Al, Be) and have been estimated also for ARIES-IV reactor. H and He production rate in first wall region are about 800 and 1500–2000 appm/MWa/m<sup>2</sup> corresponding to a gas/



dpa ratio of 50 appmH/dpa and 130 appmHe/dpa [45,46]. Although a high concentration of transmutation elements cannot be produced by fission reactor due to the threshold energy required, the problem has been approached by studying the effect of H and He implantation by accelerator irradiation.

Potentially significant effects of helium on swelling in SiC both in point-defect swelling and cavity swelling temperature regimes have been shown by a dual-beam ion irradiation study [47]. The quantitative swelling enhancement by helium within a point-defect swelling regime and the responsible mechanism need more study to be better understood. The fact that point-defect swelling is increased by helium suggests a helium effect on thermal conductivity degradation in irradiated SiC. The cavity swelling behaviour of SiC appears similar to that in irradiated metals in the presence of helium; at temperatures where vacancies are readily mobile, complex clusters of helium and vacancies form helium-filled bubbles and grow as helium is accumulated, and then convert into unpressurized voids that exhibit unstable growth when certain conditions are met. However, the magnitudes of cavity swelling determined by TEM are one or two orders smaller than the total hard sphere volume of implanted helium, due to helium transport along dislocations and (sub-) grain boundaries to surfaces.

Recently, the synergistic effect of displacement damage and He implanted atoms on the microstructure of SiC<sub>f</sub>/SiC composites has been undertaken at He/dpa ratios comparable with those of fusion reactors [48]. Single-, dual- or triple-beam experiments have been carried out at irradiation temperature below 800 °C on advanced fibre composites [48]. Helium diffusion behaviour and microstructural observation have been reported for SiC<sub>f</sub>/SiC composites, SiC fibres and monolithic SiC [49]. In this study, helium was found mobile in the carbon phase between 500 and 800 °C; in SiC phase helium become mobile above 900-1000 °C. On 10000 appm RT He implanted composites and annealed at 1400 °C He bubble were observed only at grain boundaries in SiC matrix and not at fibre-interphasematrix interface and in SiC fibre; high temperature He desorption may account for this distribution [50].

A study of the synergetic effect of triple irradiation of He, H and Si ions on the microstructural changes of SiC<sub>f</sub>/SiC composites (Hi-Nicalon S type and Tyranno SA fibre with C interphase composites manufactured by means of forced CVI process) performed at 1000 °C with a He/dpa and H/dpa respectively equal to 60 and 50 appm/dpa and a damage dose rate of 10 dpa has been carried out [51]. He bubbles were observed in the SiC matrix but no bubbles were found in both types of the SiC fibres. Carbon interphase showed no evidence of microstructural changes and no He bubbles, due to the larger He diffusion coefficient in carbon. More recently the role of H on the synergistic effects of displacement damage, helium and hydrogen implantation on microstructure and mechanical properties of ultra-high-purity stoichiometric beta-SiC and Hi-Nicalon S-CVI composite has been investigated qualitatively [52-55]. Microstructural observations of composites irradiated to 10 dpa at 800, 1000 and 1300 °C with a He/dpa and H/dpa respectively equal to 130 and 40 appm/dpa showed that special distribution of cavities in SiC composites depended on grain structure of SiC and irradiation conditions in this temperature region [52]. Cavities size in SiC matrix resulting from triple ion beam irradiation (Si, He, H) tended to be greater than that in the specimen irradiated by dual ion beam (Si, He) at 800 °C. Moreover cavities were observed on SiC fibres after triple-beam irradiation and not after dual-beam irradiation at 1000 °C [52,53]. In the case of 1300 °C irradiation, cavities on grain boundaries became larger and fine cavities appeared in grains but synergetic effect of H was not obvious.

#### 5. Other material issues

SiC<sub>f</sub>/SiC composites are an intrinsically porous material; therefore there are issues with its use for actively cooled components to be installed in a plasma vacuum chamber. In fact, He or liquid metal vapours can permeate the wall towards plasma and increase radiation losses and cool the plasma. The He permeability of PIP composites has been reported [56] to be in the order of  $10^{-5}$  (Pa s)<sup>-1</sup> for uncoated specimens. In a different work [57], the permeation constant for different composites including PIP, HP, PIP + RS based and NITE composites was reported. In that study the highest value (order of 10<sup>-4</sup> m<sup>2</sup>/s) was reported for PIP composites and the minimum for NITE composites (order of 10<sup>-11</sup> m<sup>2</sup>/s). As a result, depending on coolant operating conditions and porosity distribution, the permeability value may also increase with the same material; therefore, an efficient densification or sealing coatings are needed for blanket applications. The primary and most effective candidate as SiC<sub>f</sub>/SiC coating is CVD SiC itself [12]. Recently the feasibility of a high thickness-tight coating has been proved (up to 200 µm) [58] but this process is time consuming and expensive. The possibility to use glass ceramic coatings and Si-Cr or Si-Ti eutectics [59,60] is under investigation. In particular the first offers a materials which can be in a non-brittle state at the operating temperatures, while the second offers an excellent sealing capability. Conversely, the neutron irradiation performances of these coatings still are to be assessed.

 $SiC_f/SiC$  composites can be produced as half finished products and only in simple shapes (flat or curved panels, tubes etc...). Therefore, one of the leading technological issues is the availability of a robust low activation joining technique suitable for fusion environment. Several joining techniques have been proposed but most of them refer to adhesion by preceramic polymers, reaction bonding and brazing processes. Each of these techniques shows potential advantages and disadvantages [4] and different mechanical performances have been reported. It is worth noting that a direct comparison of these performances is not always possible because their strength is estimated by different test methodologies. Moreover, their irradiation degradation has to be assessed. Preceramic polymer joining features and performances have been described and reviewed [61,62]: this technique shows a large range of performances depending on polymer type and composition, pyrolysis temperature, powder insertion and surface conditioning. The outcome of this study was that, although the method is attractive for its simplicity and the low activation composition, this technique shows modest strength caused by the polymer shrinkage after pyrolysis and imperfect ceramic conversion. Reaction bonding joining exhibited high performances but large scattering of strength values [63]. Moreover, a source of concern is the chemical composition of the joining layer and in particular the amount of oxygen, free silicon and carbon that can limit the irradiation performances. The 'in situ' displacement reaction joints performances have also been reported [4]: in this case, the joining layer consists of a complex compound such as  $Ti_x Si_y C_z$  + SiC. Recently, the use of Si–Ti and Si– Cr eutectics has been proposed for brazing of SiC and  $SiC_f/SiC$  composites [60]. The joints obtained by means of these brazing alloys showed high pure-shear strengths (up to 80 MPa). The feasibility of a brazing technique which employs alloys without a free Si phase has been studied by using Si-44Cr alloy and CrSi<sub>2</sub> [64]. The joints thus obtained exhibited levels of strength comparable with those obtained by the Si-16Ti and Si-18Cr. The European Fusion Technology Programme foresees the characterization of these joints following neutron irradiation.

The compatibility between SiC<sub>f</sub>/SiC and the liquid Pb-17Li is under investigation because TAURO blanket and ARIES AT reactor design studies foresee the use of this metal as a coolant, breeder and neutron multiplier. Pure SiC was found chemically stable in static Pb-17Li and its stability is expected up to 900 °C based on Gibbs free energy consideration [65]. The corrosion is not the only potential limiting factor of SiC in Pb-17Li, since the liquid metal flows at different velocities through the blanket system, and its velocity is higher in the first wall region, where the temperature is higher too. Therefore, the combined effect of corrosion-erosion has to be assessed. SiC CVD coated-CVI SiC<sub>f</sub>/SiC composites were found to have good stability in Pb-17Li flowing at 0.5 m/s at 550 °C up to 3000 h exposure [66]. Corrosionerosion tests have been performed by using CVD SiC coated-CVI SiC<sub>f</sub>/SiC composites in dynamic conditions.

The material was kept in rotation for 3000 h in a Pb– 17Li bath at 800 °C [67,68]. The liquid metal-specimen relative velocity was about 1 m/s. Post exposure examination showed that, although metal infiltration occurred in the composite, no significant corrosion–erosion occurred. As manufactured CVI SiC<sub>f</sub>/SiC composites CVD coated were also exposed to isothermal static Pb– 17Li at 1000 °C up to 2500 h. Chemical analysis of Pb– 17Li before and after exposure and post exposure examinations suggested that no or negligible dissolution occurred during testing, probably due to a low Si solubility in the metal. A complementary test will be the investigation of the occurrence of an erosion-enhanced SiC dissolution in Pb–17Li at 1000–1100 °C.

# 6. Conclusions

A specific effort is on going in order to develop and characterize SiC<sub>f</sub>/SiC composites for fusion reactor application. Conceptual design studies have continued, but suitable design criteria still have to be set up in order to perform more detailed design activities. The feasibility of a high performance reactor can be demonstrated if some material properties, including irradiation performances, are substantially improved. The study of irradiation behaviour has been continued, but the fundamentals still need to be fully understood.

The thermal conductivity is a key property to be improved: the modelling of this property has provided useful information to predict the degradation induced by neutron irradiation. Material transmutation in fusion environment is another key issue; the study of gaseous transmuted element effect on microstructure and on some mechanical property has been carried out by ion beam implantation experiments. The electrical conductivity has been studied in detail; the potential radiation enhancement does not seem to be an issue for liquid metal blankets. Promising new materials with favourable unirradiated properties are available and are going to be characterized with respect to irradiation performances. Permeability issues have been addressed and quantified. A liquid lithium lead compatibility study has been performed at elevated temperature: in the experimental conditions analyzed no relevant concerns were noticed.

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