Current Status and Recent Research Achievements in SiC/SiC Composites

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CURRENT STATUS AND RECENT RESEARCH ACHIEVEMENTS IN SiC/SiC COMPOSITES

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Abstract

The development and maturation of the silicon carbide fiber-reinforced silicon carbide matrix (SiC/SiC) composite system for fusion applications has seen the evolution from fundamental development and understanding of the material system and its behavior in a hostile irradiation environment to the current effort which essentially is a broad-based program of technology, directed at moving this material class from a laboratory curiosity to an engineering material. This paper lays out the recent international scientific and technological achievements in the development of SiC/SiC composite material technologies for fusion application and will discuss future research directions. It also reviews the materials system in the larger context of progress to maturity as an engineering material for both the larger nuclear community and for general engineering applications.

1 Introduction

Since the early development effort for tri-layered isotropic (TRISO) micro-encapsulated fuels in 1960's for the high temperature gas-cooled reactors [1], silicon carbide (SiC) has been studied as the refractory nuclear material, eventually proving its outstanding high temperature strength, creep resistance [2], oxidation resistance [3], and radiation tolerance [4, 5]. Shortly after the epochal invention of the continuous SiC-based fiber by Yajima et al in mid-1970's at the nuclear materials laboratory of Tohoku University [6], the race for development of ceramic fiber-reinforced ceramic matrix composites (CMC) started to yield various kinds of continuous SiC fiber-reinforced SiC-matrix (SiC/SiC) structural composites, including those through the chemical vapor infiltration (CVI) [7], liquid silicon infiltration (LSI) or melt infiltration (MI) [8], polymer impregnation and pyrolysis (PIP) [9], and transient eutectic phase (TEP) [10] processes.

The CMC's distinguish themselves from the monolithic ceramics for several outstanding features including the damage tolerance, highly predictable failure properties, and the ability of near-net shaping for complex geometries. The application of SiC-matrix composites to fusion reactor blankets was proposed in late 1970's by Riley, et al.[11] The exceptional low activation / low decay heat properties [12] and the lesser impact on the tritium breeding were among the additional factors that SiC/SiC composite may benefit the fusion reactors as a blanket material. However, because of the radiation-instability for the early high oxygen amorphous SiC-based fibers and the second generation carbon-rich, nano-crystalline SiC-based fibers [13, 14], demonstration of the radiation-tolerant SiC/SiC composite had to wait until 1999 when the US – Japan fusion materials collaboration project fabricated the third generation near-stoichiometric SiC fiber, CVI SiC matrix composite tested it following neutron irradiation in the High Flux Isotope Reactor (HFIR) [15].

Succeeding works collectively demonstrated the general radiation tolerance of the third generation SiC fiber composites through the CVI process [16] and the nano-infiltration and transient eutectic-phase (NITE) process [17] in broad ranges of irradiation temperature (300 – 1300°C) and neutron fluence (up to >70 dpa), while the limitations in radiation resistance in certain irradiation conditions are recently being identified for particular materials [18]. While the radiation effects have been considered as the primary technical feasibility factor in the early phase of SiC/SiC study for fusion applications, significant activities have also been undertaken in international fusion and fission materials communities toward advancing the general technological readiness levels (TRL's) [19, 20] for the SiC/SiC materials technologies for those applications, with the efforts ranging from understanding the fundamental radiation damage processes and the environmental effects to the component fabrication and the code qualification. The present paper summarizes the current activities, recent progress, and future opportunities in research and development of SiC/SiC materials science and technology for fusion energy and related application areas.

2 Synergy with non-fusion applications of ceramic matrix composites

The successful development and demonstration of the radiation-tolerant SiC/SiC composites in fusion materials programs attracted significant attentions in fission nuclear energy communities. Currently, the research and development of the CMC for various fission applications and the SiC/SiC studies for fusion energy applications play significant leveraging roles with each other.

2.1 *Advanced Fission Reactors*

The High Temperature Gas-cooled Reactor (HTGR) is the leading runner in the TRL advancement in the nuclear applications of the CMC. The use of SiC/SiC is considered necessary or highly desirable for components such as the control rod sheath that receive significant fast neutron doses at high temperatures, whereas the carbon fiber composites will serve the purposes for the structural core supports where the neutron flux is significantly more moderate [21, 22]. The HTGR's operating at higher temperatures (Very High Temperature Reactors, VHTR's, with the helium coolant outlet temperature up to 950°C) and of the prismatic cores tend to require the radiation-tolerant SiC/SiC core components, whereas the pebble-bed core HTGR's will allow a limited use of the code-qualified high temperature alloys such as Alloy 800H.

With the key technical feasibility issues positively addressed, CMC's are now considered the available materials technology for the HTGR applications. The current effort in US focuses on development of the design code and the standards that will be used in the qualification process for the CMC materials and components. The design rules for Composite Core Components and Composite Assemblies for High Temperature Reactors Systems are currently being developed as the American Society for Mechanical Engineers (ASME) Boiler & Pressure Vessel Code, Section III, Division 5 - High Temperature Reactors, Subsection HH – Class A Nonmetallic Core Support Structures, Subpart B – Composite Materials, by the Sub-Group on Graphite Core Components [23]. The core components manufactured from carbon or silicon carbide fiber composites are included in the scope of the code. Moreover, in support of the ASME code development, various standard materials specifications, test methods, and test guides are being developed for the CMC's in the ASTM International's Committee C28 on Advanced Ceramics. The ASTM Standards that are currently being developed include those on specifications for

SiC/SiC and carbon fiber composites for nuclear applications and several test methods for mechanical properties of CMC's [24]. In addition to these ASTM Standards, the ASME design code will adopt supplemental documents such as a compilation of properties and radiation effects for the nuclear grade SiC/SiC composites [16].

For some of the advanced reactor concepts that will demand materials more aggressive high temperature capability and radiation tolerance, SiC/SiC composites are often the sole candidates for certain components. Such components include the fuel assemblies and core supports for the gas-cooled fast reactors (GFR's) [25, 26] and the fluoride salt-cooled high temperature reactors (FHR's) [27]. Moreover, the French sodium fast reactor (SFR) program considers the SiC/SiC as the viable core material option [28].

2.2 Accident-Tolerant LWR Cores

The use of SiC-based materials to the light water reactor (LWR) fuel claddings had been proposed with the primary purpose of achieving a better neutronics and improved safety features [29]. After the Fukushima Dai-ichi accident in 2011, the SiC-based materials started to attract more serious considerations as the enabling materials for the Accident Tolerant Fuels (ATF) and the Accident Tolerant Core (ATC) technologies for the current fleet LWR's. The current areas of active technology development in support of the ATF/ATC programs include the SiC/SiC composite core structures [22], the SiC/SiC composite-based fuel claddings [30], and the SiC-matrix fully ceramic micro-encapsulated (FCM) fuels [31].

Among these, the SiC/SiC composite channel boxes for the boiling water reactors (BWR's) are at the highest TRL, with the prototype fabrication (Figure 1) and the several key evaluations including the handling accident test and thermal shock test completed. The concerns associated

with the water corrosion, the environment-assisted slow crack growth, and the irradiation strain remain to be fully addressed.

Figure 1. SiC/SiC composite components for accident-tolerant light water reactor technologies: prototypical boiling water reactor channel box (left, courtesy of Electrical Power Research Institute) and developmental small diameter tubes (right).

While the LWR fuel cladding application imposes significant more challenges to SiC/SiC composite technologies than the core structure applications, it presents interesting synergy with the fusion blanket applications. For example, the LWR fuel rods require a robust, radiation-tolerant, and hermetic end-plug joining method, which will also be essential for integration of the fusion blankets. Moreover, the heat flux through the LWR fuel clad wall will exceed 0.5 MW/m^2 up to $\sim 1 \text{ MW/m}^2$, comparable with the anticipated heat flux through the first wall of the fusion blankets. Recently, Ben-Belgacem et al [32] shown that the stress in the LWR fuel clad will be dictated by the differential swelling-induced secondary stress, transitioning from tension to compression along the heat flow, through a two-dimensional finite element analysis that takes into account the dynamic evolutions of the thermal conductivity and swelling distributions in a model SiC/SiC clad during its service life. Combining such analyses with the

statistical strength data for the material will enable the probabilistic failure assessment for the components.

Figure 2. Dynamic finite element analysis of thermal-physical-structural evolutions in SiC/SiC light water reactor fuel cladding, showing domination of differential swelling-induced secondary stress in the end-of-life stress state in the cladding as the result of a through-thickness temperature gradient assisted by radiation-induced thermal conductivity decrease [32].

2.3 Non-nuclear applications

Non-nuclear applications of the CMC's are rapidly growing where the high temperature capability beyond the metallic heat resistant alloys and the high specific strength are valued. Besides the wide-spread use of carbon/carbon composites in semiconductor industries, the leading demanding application areas for the CMC's are turbine engines for aircrafts. The use of CMC's for the turbine engines is progress from the less-demanding exhaust components to shrouds/seals, combustors, stators/vanes, then finally rotors/blades in the order of increasing

technical challenges. Replacing the metallic components with CMC's improve the fuel efficiency potentially very significantly through the higher temperature of combustion (that also contributes to the cleaner exhaust), reduced component weight, and the mitigated need for active cooling of the components [33]. SiC/SiC turbine shrouds are currently being tested in the CFM International's LEAP (leading edge aviation propulsion) engine, envisioning the passenger services starting in 2016. GE Aviation plans to start a large scale production of the SiC/SiC combustion liners and the turbine airfoil components for use in their next generation GE9X engines for larger passenger aircrafts. These SiC/SiC are manufactured through the MI process, limiting the synergy with the nuclear CMC development mainly to the test methods. However, expanding synergy into areas like the design methodology and life-limiting properties is anticipated as the target application of SiC/SiC in turbine engines progresses to more demanding components for which the further enhanced high temperature capabilities and creep resistance are required.

Figure 3. Prototypical SiC/SiC components for GE Aviation's next generation GE9X engine for Boeing 777X and other wide-body twin-jet passenger aircrafts (Courtesy, GE Aviation).

3 Requirements for fusion SiC/SiC

The potential applications of SiC/SiC composites currently considered for the in-vessel components of the magnetic confinement fusion devices may be classified into three categories: blanket structures including the first wall (FW), flow channel inserts (FCI) for the liquid metal (LM) blankets, and the plasma-facing components (PFC's). However, there currently are no relatively mature PFC concepts that assume the use of the CMC's, despite the favorable compatibility of the tungsten – SiC system (in terms of thermal expansion, joining technology [34], and the operating temperature windows [35]) and the CMC's likely adequacy as the materials for the certain helium-cooled PFC parts and the radiatively cooled high temperature surfaces in more aggressive PFC concepts. Below, the performance requirements to the SiC/SiC composites as the blanket structural and FCI materials in the recent design concepts are briefly discussed.

3.1 Blanket structures

The conceptual fusion power reactor blanket designs that assume the use of SiC/SiC composites as the structural and FW materials have not significantly evolved since the last review by Snead et al [36], except that the ARIES-ACT1 self-cooled lead-lithium (SCLL) blanket design [37], Figure 4, evolved from the preceding ARIES-AT concept [38]. The refinement in the newer ARIES-ACT1 design involves the slightly narrowed operating temperature window, complete removal of the gas tightness requirement for the SiC/SiC walls, and the simplified structural design, all of which help reduce burdens to the structural material. Selected operating parameters for the SiC/SiC blanket/FW structures in some of the proposed fusion breeding blanket concepts are compared in Table 1.

The SCLL is considered the leading concept that may use SiC/SiC as the structural material, mainly because of the relatively low coolant pressure, less demanding hermeticity requirement, and the proven static compatibility between the lead-lithium eutectic (LLE) and SiC up to very high temperatures [39]. However, the chemical compatibility in a flow condition with the temperature gradient and the fusion-relevant electromagnetic environment is yet to be clarified. The allowable stresses in some conceptual designs appear moderately challenging for the present nuclear-grade SiC/SiC, considering the long-term hermeticity requirement. The design-assumed through-thickness thermal conductivity values are slightly optimistic for the required in-plane strength, presenting the need for further design refinements and the opportunities for further materials development.

The helium-cooled ceramic breeding (HCCB) blanket also shown in Table 1 is another concept that may utilize the SiC/SiC structures. The selection of chemically controlled helium coolant likely eliminate any serious concern for the chemical incompatibility as assessed in the HTGR and GFR studies [40, 41]. The chemical compatibility of SiC with certain lithium oxides has been demonstrated in a high temperature, high radiation environment [42]. The helium tightness requirement under a significant stress loading due to high pressure helium containment remain a leading critical issue, for which a research synergy with the fission product gas containment in the LWR clad development is anticipated. Since the ceramic structure HCCB designs have not significantly evolved, there are significant rooms for the design refinement for this blanket concept.

Figure 4. A section of self-cooled lead-lithium blanket, divertors, and structural ring (left) and a section of the outboard blanket (right) for ARIES-ACT1 fusion power reactor concept [37] (courtesy, The ARIES Team).

Table 1. Operating conditions and design requirements for SiC/SiC structures in some of proposed blanket concepts.

	<i>ARIES-ACT1</i>	<i>EC PPCS</i>	<i>Dream</i>
	<i>SCLL</i>	<i>Model D</i>	<i>HCCB</i>
		<i>SCLL</i>	
Coolant / breeder	Pb-Li	Pb-Li	He / Li ₂ TiO ₃
Coolant temperature, inlet/outlet (°C)	740/1030	700/1100	600/900
Structural material temperature, min./max. (°C)	740/960	700/990	600/1000
Max. SiC/coolant interface temperature (°C)	<960	915	900
Fluence lifetime	3% burn-up	N/S	N/S
Stress loading (MPa)	Combined	Normal <109	FW Tresca
	<190	Shear <57	<60
Thermal conductivity at operating temperature, transverse (W/m-K)	10 (600°C)	15	15
	20 (1000°C)		
Thermal expansion coefficient, in-plane (ppm/K)	4	4	3.3

Reference	[37]	[43]	[44]
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3.2 LM blanket FCI

The main functions of the LM blanket FCI are to electrically and thermally insulate the reduced-activation ferritic/martensitic steel structures from the flowing liquid metal that is of high temperature and highly conductive. Therefore, the primary requirements to the FCI materials (or component) are adequately low electrical and thermal conductivities in the through-thickness orientation of the wall. The FCI is obviously a less demanding application for SiC-based ceramics and composites than the blanket/FW structures, as the key material requirements are summarized in Table 2. The materials and design requirements have been identified to fair details [45] and several SiC-based FCI concepts including those using the two-dimensional (2-D) SiC/SiC [46] and engineered porous SiC structures [47] have been proposed. The previous studies have identified that today's nuclear grade 2-D SiC/SiC composites meet most of the required properties including the thermal and electrical insulating properties and the ability to survive the temperature drop exceeding 200K across the thickness [46], whereas the SiC in other forms may provide some benefit-disadvantage trade-offs [47]. The remaining critical issues include the chemical compatibility for the SiC – steel – LLE ternary system in the practical temperature and flow conditions, the potential effects of the aggressive nuclear transmutation during the power reactor operation on various properties (also true for structural applications), and the hermetic joining that withstand both the radiation and the environment.

Table 2. Operating conditions and design requirements for flow channel inserts for some of the proposed fusion power reactor liquid metal blanket concepts.

	<i>US DEMO</i>	<i>ARIES-ACT2</i>	<i>EC PPCS</i>
	<i>DCLL</i>	<i>HCLL</i>	<i>Model C HCLL</i>
Coolant / breeder	He & Li-Pb	He / Li-Pb	He / Li-Pb
Max. SiC / breeder interface temperature (°C)	700	640	700
Fluence lifetime	N/S	N/S	N/S
Local stress loading (MPa)	Design		Tensile <110
	allowable 120		Von Mises <140
Thermal conductivity, transverse (W/m-K)	1 – 5	2 (all temp.)	2 (600°C)
Electrical conductivity, transverse (S/m)	<~20		4
Additional note			Joining assumed
Reference	[45]	N/A	[43]

4 Recent progress in fusion SiC/SiC R&D

In this section, some of the recent progresses in the research and development of SiC ceramics and composite materials technology in fusion or related programs are highlighted. These developments are considered to belong to TRL's 2 (e.g., targeted materials development), 3 (e.g., basic radiation effects and joining technologies), through 4 (e.g., evaluations for design-limiting factors).

4.1 Joining development and evaluation

Technology development for joining SiC/SiC to itself or other materials is essential for the blanket integration and at least preferred for the FCI assembling. The requirements for the joints will include the mechanical robustness, tolerance against neutron irradiation, chemical

stability in the operating environment, and the hermeticity to liquid metal or helium depending on the design. While there have been developed a wide variety of methods for joining SiC to self or various metals, none of them has been developed or designed to operate in nuclear environments. Recently, several candidate SiC joining methods for nuclear applications had been down-selected, optimized for the radiation stability, and evaluated after neutron irradiation up to ~ 5 dpa at elevated temperatures in an international collaboration among US, Japan, and Italy [48].

The joining methods evaluated were 1) a diffusion bonding with the titanium insert resulting in a $\text{Ti}_3\text{SiC}_2/\text{Ti}_5\text{Si}_3$ in the bonding layer, 2) a displacement reaction bonding with the TiC/Si green tape producing a $\text{Ti}_3\text{SiC}_2/\text{SiC}$ bonding layer, 3) SiC-based transient eutectic-phase (TEP) joining with the mixed oxide additives for bonding with a seamless NITE-like SiC using either a mixed powder slurry or a green tape, and 4) CaO- Al_2O_3 glass-ceramic tailored to produce a fully ceramic CaO/ Al_2O_3 /SiO₂ bonding layer. The substrate materials that were joined together included the high purity CVD SiC, the NITE-like sintered SiC, and the Tyranno-SA3/NITE SiC composite. After neutron irradiation to ~ 3 to ~ 5 dpa at 500 or 800°C in HFIR, all the evaluated joints exhibited no or only slight decrease in the torsional shear failure resistance, demonstrating the encouraging radiation tolerance. At the same time, microcracks were often observed in the non-SiC bonding materials as shown in Figure 5, implying potential hermeticity issues likely arising from the differential swelling between SiC and the bonding layer. A follow-up program extending the irradiation temperature range and fluence level is in progress.

One of the difficulties in this irradiation program was the serious lack of commonly used test methods for determination of strength of the ceramic joints in an adequately defined stress state. For this reason, a test method for torsional shear failure resistance of ceramic joint test

articles was newly developed as a part of the collaboration program. As the result, a test method that determines the failure resistance of the miniaturized ceramic joint specimens in solid or perforated hourglass geometries was successfully established [49], enabling evaluation of the effects of neutron irradiation (and environmental exposure) on the joint strength.

Figure 5. Scanning electron micrographs of some of radiation-tolerant silicon carbide joints after neutron irradiation in High Flux Isotope Reactor.

4.2 Alternative composites

The CVI SiC matrix composites with the third generation near-stoichiometric SiC fibers are considered the reference nuclear-grade SiC/SiC composites because of the perceived radiation tolerance and the chemical compatibility of the high purity, stoichiometric SiC matrix with various harsh environments. However, performances of the CVI SiC matrix composites involve certain limitations including the relatively low thermal conductivity due to their inherent porosity, the moderate proportional limit stresses for the same reason, and the gas

permeability due to the process-induced matrix microcracking. Moreover, the current near-stoichiometric SiC fibers and the certain pyrocarbon (PyC) interphases are rather unlikely to take full advantage of the inherent high radiation tolerance of high purity SiC, as elaborated later. Therefore, there will be opportunities for the composite materials made of the alternative fibers, interphases, and/or matrices to complement or replace the today's reference SiC/SiC.

As mentioned earlier, the NITE SiC/SiC composites have demonstrated the stability under neutron irradiation up to intermediate fluence levels [17]. The current efforts in advancing this processing technique in Japan are focused on the manufacture of thin walled, small diameter tubes for the fuel cladding applications [50] and the development of interphase-less porous NITE-like SiC matrix composites toward the reduced cost manufacturing [51].

In the European program, the slip infiltration-based SITE processes were developed as alternative process for matrix densification of SiC/SiC composites with 3-dimensional architecture. The process utilizes a combination of two techniques: 1) infiltration of the fabric preform with fine (nano-sized and submicron) SiC particles from aqueous suspension by electrophoretic (infiltration) deposition, EP(I)D, and 2) densification with the aid of a pre-ceramic polymer by the PIP [52]. During the EP(I)D, the porous preform is infiltrated with a large amount of crystalline β -SiC powder, which serves as a passive filler during the PIP densification steps. In contrast to pressure-assisted infiltration of the fabric with SiC powder, EP(I)D is effective in achieving a homogeneous infiltration through thickness of the preform with a good infiltration of inter- as well as intra-bundle areas. Due to large amount of SiC powder introduced into the fabric, the densification of final material is enhanced and presence of large voids, typical for the conventional PIP process, is avoided.

Densification of the green composite is achieved by vacuum infiltration of pre-ceramic polymer followed by pyrolysis and crystallization. After six PIP cycles with final thermal treatment at 1600 °C, 87 % of density is achieved, the material is composed of highly crystalline β -SiC, possessing a relatively high thermal conductivity (65 W/m-K at RT and 30 W/m-K at 1000 °C) [52, 53], which is partly the consequence of absence of oxide phases and relatively large grain size. In comparison to the standard PIP processing, a more homogeneous matrix with much smaller pores and a similar density is obtained with fewer densification steps [54], as shown in Figure 6. Due to higher maximum temperatures used in the crystallisation step, the SITE matrix exhibits higher crystallinity in comparison to standard PIP processing, where lower temperatures were used in order to achieve densification.

Figure 6. Typical scanning electron microscopy images of a SITE composite at different magnifications.

As the SITE-SiC/SiC composite is composed only of highly crystalline SiC with no sintering additives, it relates to the European target - low -activation material with relatively high thermal conductivity. The main objectives of further work are to improve mechanical strength by using better interphase layer and to apply a W-rich surface layer which will serve as

impermeable coating and will widen the application area. The SITE process has not been demonstrated beyond a laboratory scale production, but due to the nature of the involved procedures, the process itself has good potential for up-scaling.

4.3 Chemical compatibility

Chemical compatibility of the SiC – LLE system in non-static conditions is being studied in the Japan-EU Broader Approach (BA) program, specifically addressing the effects of surface conditions (fiber penetration, oxidation, etc.), coating on fibers, and impurities (e.g., trace elements that exist in irradiated environment and ferritic-martensitic steel-corrosion products). The recent experiment in Japan employing the rotating disc technique indicated the lack of obvious reactions or corrosion at the CVD SiC disc surfaces after an exposure to LLE for 1000 h at 900°C [55].

A thermal convection LLE loop facility has been constructed at Oak Ridge National Laboratory. This new facility enables a critical evaluation of the materials compatibility in an LLE flow environment at a temperature gradient of ~150°C while the peak temperature is currently limited at 550°C, making it suitable for evaluating the FCI materials for steel-structure LLE blankets. The future plans include gradual increases of the peak temperature as performance data justifies.

4.4 Electronic properties

In the BA program, the in-situ volume electrical conductivity measurements were carried out for SiC under various irradiation sources (gamma-ray, X-ray, 14 MeV fusion neutrons) in various atmospheres (air, He and vacuum), in order to understand the radiation effects on electrical

properties of SiC. The radiation-induced conductivity (RIC), which is an instantaneous increase in the electrical conductivity by electronic excitation, occurred in the beginning of ionizing dose [56]. The RIC values gradually increased with an increase of the dose and subsequently became almost constant. As for the base conductivity in the absence of radiation, the similar radiation-induced phenomenon was observed as the rapid increase at the lower dose of approximately 10 kGy and the gradual increase at the higher dose. Therefore, the dose dependence of RIC was associated with the degradation in base conductivity with dose: radiation-induced electrical degradation (RIED)-like behavior. On the other hand, the RIED-like behavior may be caused by a change of electronic structure due to the electronic excitation essentially, resulting from the covalent nature of SiC. The preliminary XPS results suggest the dissociation of carbon from $\text{SiO}_x\text{C}_{3-x}$ composition existing on the top-most surface. The dependence of dose rate was summarized in Figure 7. It was found that the RIC values for the mono-crystalline 4H-SiC and CVD (poly-3C) SiC under X-ray irradiation at room temperature were proportional to the dose rates and 3-4 and 5-6 orders of magnitude higher than those in oxide materials, respectively. Figure 7 also summarizes the temperature dependence of electrical conductivity of various types of SiC materials, indicating that the increasing electrical conductivity at high-temperatures is due primarily to the thermal effect with the impact of RIC negligible.

Figure 7. Radiation-induced conductivity of SiC and ceramic oxides: (a) dependence of dose rate [3] and (b) dependence of temperature.

4.5 Interfacial characterization

The mechanical properties of the fiber-matrix interfaces are critical in designing and determining the properties of CMC's [57]. A novel experimental technique to determine some of the interfacial properties was recently developed in the US fusion program [58]. In this exercise, micro-pillar samples involving the fiber-matrix interfaces at varied slant angles are prepared by the focused ion beam micro-processing followed by the compressive failure load measurements using a nano-indentation device, so that the slant angle dependence of the interfacial shear failure load explicitly gives the cohesive shear debond stress and the internal debond friction coefficient per the Mohr-Coulomb criterion. These data also enables more credible determination of the interfacial sliding stress when combined with conventional techniques such as the single filament push-out method.

Figure 8. A slant interface micro-pillar prepared for interfacial mechanical properties determination [58].

4.6 Fundamental radiation effects

The exceptional radiation stability of SiC is often attributed to the presence of very high density point defects and clusters, suppressing the net fluxes and the long-range diffusion of migrating point defects, consequently retarding or eliminating the progressive evolutions of various radiation-induced phenomena that are commonly seen for many other materials at elevated temperatures. This self-stabilizing effect in SiC, or the radiation-induced radiation insensitivity, may be visualized in Figure 9, where the estimated effective sink strength in the first order effective medium approach for the radiation-produced matrix defects in SiC is compared to the geometrically estimated sink strengths for engineered ceramic nano-features in some of the advanced radiation-tolerant steels [59]. The sink strength of 10^{16} m^{-2} is considered a rather conservative criterion for the very effective suppression of the migrating defect flux.

Figure 9. Comparison of estimated equivalent sink strength (SS) of self-defects in irradiated SiC in an intermediate temperature range (~200 - ~800°C) with those estimated for engineered dispersants in advanced radiation-tolerant alloys.

While it is convincing that the matrix defects that account for the saturatable swelling for SiC in the intermediate temperature range are very effectively suppressing the migrating defect fluxes, detailed physical processes of the formation of the extremely highly defected matrix that self-stabilizes remain to be explored through both simulations and experiments. Among the interesting results from the recent atomistic simulation on the radiation-induced defect productions and evolutions is the finding of very limited in-cascade recombination in SiC.

Samolyuk et al [60] reported that only ~50% of the Frenkel pairs produced in cascade events annihilate through the in-cascade recombination (Figure 10), in contrast to ~99% typical for bcc iron, in the molecular dynamics (MD) simulation. The outstanding feature at the MD cutoff time is the closely separated vacancy – interstitial pairs that do not recombine for both carbon and silicon sublattices, attributed to the significant recombination barrier for the Frenkel pair recombination. Hu et al. has demonstrated presence of such a high recombination threshold

energy for the silicon Frankel pair in SiC in a DFT calculation [61], further implying the important role of recombination barrier in the self-stabilization mechanism of SiC.

Figure 10. Time evolutions of the numbers of Frenkel defects, antisites, and recombinations during a 10 keV Si primary knock-on event as calculated by a molecular dynamics model using the Gao-Weber potential [60].

4.7 High dose irradiation effects

The recent data from a high dose irradiation of a reference SiC/SiC composite in HFIR presented both promise and limitations [62]. In this experiment, the Hi-Nicalon Type S, CVI SiC matrix composite with the PyC/SiC multilayered interphase was irradiated to 71 – 74 dpa at 300, 500, and 800°C. The results demonstrated the continued saturations (after the saturation was reached at <2 dpa) in swelling, thermal conductivity, and dynamic Young's modulus, implying the lack of progressive radiation effects mainly in the CVI SiC matrix up to these very high fluence levels. The proportional limit stress was retained at the pre-irradiation level after irradiation at 800°C, confirming the integrity of the matrix. However, the ultimate flexural strength decreased moderately at 500 and 800°C and very significantly at 300°C, with the proportional limit stress somewhat reduced at 500°C and significantly reduced at 300°C. The

fracture surfaces with some fiber pull-out remain unchanged after irradiation except for the 300°C case where both the composite and fiber fracture surfaces appeared unusually flat. These observations collectively convince one that the Hi-Nicalon Type S fiber deteriorated, more significantly at lower irradiation temperatures, and the interphase compromised its function after the 300°C irradiation. The transmission electron microscopy analysis revealed evidences for the significant microstructural modifications around the interphase [62]. The current effort is focusing on identification of the cause for fiber degradation with the radiation instability of the second phases is suspected to be the culprit. Moreover, new irradiation campaigns to determine the high fluence effects on the Tyranno-SA3 and the SCS-Ultra SiC fiber composites have been initiated in the US program.

4.8 *Irradiation creep*

Irradiation creep is an important phenomenon that often contributes to define the design windows for nuclear structural materials. The previous in-pile bend stress relaxation (BSR) creep study by Katoh et al. revealed the linear couplings of the initial transient irradiation creep with the stress magnitude and the transient swelling for various grades CVD SiC [63]. The relatively small coupling coefficients between the creep and swelling strains were determined, denying the possibility of significant creep-enabled mitigation of the swelling-induced stress that develops in temperature-graded and/or flux-graded structures made of SiC-based materials. Such stress is anticipated to dominate in the SiC-based fusion FCI [45] and the fission fuel clads [32]. A companion study by Koyanagi et al. for the NITE SiC/SiC composite and its matrix material revealed the creep strains that are greater than for CVD SiC and slightly non-linear in stress dependence, implying the significant contributions from both thermal and

irradiation creep at the impure grain boundaries [64]. The NITE SiC/SiC composite exhibited the creep behavior close to that for CVD SiC, indicating the decent creep resistance for the Tyranno-SA3 fiber.

These earlier low fluence BSR experiments not only revealed the initial transient irradiation creep properties of these materials but proved applicability of the stress relaxation technique to study the irradiation creep behavior at higher fluence levels, because only small fractional relaxations have taken place during the first dpa or two. Based on this a set of BSR experiments were added toward the irradiation to fluences beyond the initial transient creep regime. The first data that were recently made available in the ~ 10 dpa range, Figure 11, likely indicate the slow progression of creep deformation with the ever-decreasing instantaneous creep compliances for the CVD SiC. The lack of any symptom of the onset of steady-state irradiation creep becoming apparent at the ~ 10 dpa fluence levels adds a unique feature to the irradiation response of SiC. These irradiation creep data will be fully analyzed after completion of the planned irradiation experiments that extend to 30 dpa.

Figure 11. Fluence-dependent stress relaxation behavior of CVD SiC during neutron irradiation at 380 – 1180 °C, showing minor temperature dependence of irradiation creep behavior. Dashed lines are for hypothetical steady-state creep for the constant creep compliance indicated by the labels.

4.9 Ion irradiation studies

The irradiation creep behavior of SiC and the effect of helium on it were recently studied by means of ion irradiation (self-ion and dual-beam with helium) that offers the precise control of the irradiation conditions [65]. The tension surfaces of elastically bent thin specimens were irradiated with 5.1 MeV Si^{2+} ions up to 3 dpa at various temperatures, which is referred to as a single-ion experiment. Additional He^+ ions were irradiated simultaneously in the dual-ion experiment to simulate the (n, α) reaction. Results for cases of 150 MPa stress are shown in Fig. X for both (a) single-ion and (b) dual-ion experiments. Irradiation creep was detected above 400 °C in the single-ion case, where a linear relationship between irradiation creep and swelling was observed at 400–800 °C for all stress levels studied. The proportional constant of the linear relationship was strongly dependent on temperature and stress. A rapid reduction in creep strain was observed above 1000 °C, indicating thermal creep is more operative. On the basis of the microstructural analysis in this work, anisotropic distribution of self-interstitial atom (SIA) clusters was suspected to be one of the key creep mechanisms. Some interesting results were obtained from re-irradiation under stress after the irradiation without stress. The creep strain was significantly retarded by pre-irradiation to even 0.01 dpa at 400 and 600 °C. This implies that the loop orientation, and/or the population of other key contributors, was determined very early in the irradiation regime. For the dual-ion cases, irradiation creep was seemed to be absent or very limited at all irradiation temperatures studied (400–800 °C). Although further

data may be required for determining the helium effects, microstructural analysis indicated that helium inhibited the stable growth of SIA clusters and prevented them from exhibiting anisotropic distribution.

Figure 12. Relationship between the irradiated curvatures of stressed and unstressed specimens at various irradiation temperatures in (a) single ion and (b) dual ion experiments. The radii of irradiated samples were measured at room temperature using the AFM after samples were released from the straight or curved fixtures. [65]

4.10 Thermal conductivity modeling

Recent progress in SiC/SiC thermal conductivity modeling [66] explores the analogy between the variables used in elasticity and those in steady-state heat conduction established by Hatta and Taya [67]. This analogy allows the use of the formulation established for the elastic stiffness of a unidirectional (UD) composite using the Eshelby equivalent inclusion method to obtain the expression for the composite thermal conductivity. To date, the Eshelby's equivalent inclusion

method [68] combined with a Mori-Tanaka type model [69] presents one of the most efficient and accurate approaches for the prediction of the composite elastic properties [70]. To fully exploit this analogy, a Mori-Tanaka type model combined with the Eshelby's equivalent inclusion method has been explored to predict the thermal conductivities of these composites as a function of temperature and the constituent parameters such as thermal properties of the fiber and matrix phases, fiber volume fraction, fiber orientation, and fiber/matrix interfacial thermal conductance [67-70]. A computational tool named EMTA (Eshelby-Mori-Tanaka approach) was developed to implement the EMTA models for the computation of thermal conductivities of fiber-reinforced composites [71].

Based on the EMTA method that successfully modeled the thermal conductivities of 2-D woven Hi-Nicalon Type-S CVI SiC/SiC [72], a more fundamental approach has been developed for predicting the thermal conductivities of the composites subjected to neutron irradiation in the point defect swelling regime. The effects of grain size and temperature were incorporated into computations for CVD-SiC to compute the thermal conductivity of unirradiated SiC constituents with high accuracy. Molecular dynamics (MD) data is used from simulations performed to compute the thermal defect resistance as a function of vacancy concentration in perfect crystal β -SiC via the Green-Kubo or non-equilibrium MD (NEMD) methods [73]. The computed defect thermal resistances as functions of vacancy concentration (atomic fraction) and irradiation dose are then used to compute the thermal conductivities of polycrystalline SiC including CVD-SiC, nearly stoichiometric SiC fibers, and the ICVI-SiC matrix. Finally, the computed fiber and ICVI-SiC matrix thermal conductivities are used by EMTA, together with a pyrocarbon (PyC) interphase, to compute the thermal conductivities of 2D woven SiC/SiC composites subjected to neutron irradiation within the same irradiation dose range corresponding to the point defect swelling

regime. The predicted thermal conductivity results for irradiated 2D Tyranno-SA/ICVI-SiC and Hi-Nicalon-Type S/ICVI-SiC composites are compared with the available experimental data for similar composites ICVI-processed with these fibers.

Figure 13 shows the transverse thermal conductivities predicted for the two composites as a function of temperature and damage dose (dpa) using only the experimental thermal resistance data as a function of dose from [74]. This simulation result demonstrates the full power of this approach in being able to separate out the fiber and thermal effects. The beneficial effect of operating at 1073 K for SiC/SiC composites is clearly seen in this prediction. Physically, this is due to the close-pair recombination that results from the elevated temperature radiation damage and this data corresponds to MD results qualitatively.

Figure 13. Transverse thermal conductivity vs. dose predicted for the 2D Tyranno-SA/ICVI-SiC and Hi-Nicalon Type-S/ICVI-SiC composites irradiated at 473 K and 1073 K.

4.11 Transmutation effects

The nuclear transmutations induced by the fusion spectrum neutron irradiation are considered to be the most critical unknown among the key service life-limiting factors for SiC ceramics and composites. The latest neutronics work by Sawan et al.[75] predicts the production rates of 50 – 180 appm He/dpa and 20 -70 appm H/dpa for gaseous transmutations and 10 – 45 appm Mg/dpa, 5 – 18 appm Be/dpa, 3 – 14 appm Al/dpa, and 0.2 – 1.5 appm P/dpa, depending on the blanket concept and magnetic or inertial confinement, for the most notable solid transmutations. The high production rate for helium is known to cause enhanced swelling due like to stability of the interstitial helium [76] at intermediate temperatures during ion irradiation [77] and through stabilization of vacancy clusters at high temperatures ($>\sim 1000^{\circ}\text{C}$) during ion [78] and neutron irradiation [79]. The recent triple-ion irradiation study implies the minor effects of hydrogen implantation in addition to helium on the irradiated microstructural evolutions [80].

The implications of the significant solid transmutations are largely unknown. Aluminum is known to alter the oxygen transport in SiC at high temperatures making its burn-in a significant concern in terms of corrosion resistance [75]. Moreover, most of the burn-in elements will modify the electronic properties of SiC as an impurity semiconductor. In lack of an intense fusion neutron source today, the ion implantation and the atomistic simulations are useful tools for exploring the solid transmutation effects [81]. The recent theoretical study by Hu et al. employing the density functional theory indicates significant Mg mobility and that Mg clustering is energetically favored with end result forming Mg_2Si [61]. Further theoretical and experimental investigations into the Mg burn-in are in progress in the US fusion materials program.

5 Conclusions and path-forward

As reviewed earlier, the nuclear-grade SiC/SiC composites are currently establishing a position as a new class of materials that bring in revolutionary high temperature capability, accident tolerance, and radiation tolerance to today's fission energy systems such as the LWR's and HTGR's. In particular, the ASME design code development for CMC core components for high temperature reactors and the associated ASTM standards development will pave a way for qualification of critical nuclear components that are manufactured with these engineered brittle-matrix damage-tolerant composite materials. The long-range development of the accident-tolerant SiC/SiC-based fission fuel clad technology presents an important leverage with fusion structural SiC/SiC research because of the challenging requirement for gas tightness under a significant stress loading for both applications. Moreover, the ongoing development and deployment of CMC's in passenger transportation systems present synergy in terms of both the risk management for the critical safety components and operations in extreme high temperature conditions.

Despite the favorable environments for the CMC research and development in fission and non-nuclear application areas, the international fusion materials programs are up to overcome the significant roadblocks and address various design-limiting factors toward establishing more credible fusion blanket (and possibly PFC) designs with the SiC/SiC components. The critical research areas toward narrowing the technology gaps, or advancing the TRL's, include the high fluence radiation effects on the current and developmental composite materials, robust integration technologies that withstand the harsh operating conditions, chemical compatibility with and tightness against permeation of gaseous or liquid coolant and/or breeding materials, and the effects of gaseous and solid transmutations on a whole range of properties. Moreover,

advancing the basic understandings of the foundations for the design-limiting phenomena, including the physical processes of radiation damage and the constitutive theories for physical, thermal, mechanical, and fracture properties, are essential.

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