Journal of Nuclear Materials 386–388 (2009) 622–627

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Recent advances and issues in development of silicon carbide composites for fusion applications

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A B S T R A C T

Radiation-resistant advanced silicon carbide (SiC/SiC) composites have been developed as a promising candidate of the high-temperature operating advanced fusion reactor. With the completion of the 'proof-of-principle' phase in development of 'nuclear-grade' SiC/SiC composites, the R&D on SiC/SiC composites is shifting toward the more pragmatic phase, i.e., industrialization of component manufactures and data-basing. In this paper, recent advances and issues in (1) development of component fabrication technology including joining and functional coating, e.g., a tungsten overcoat as a plasma facing barrier, (2) recent updates in characterization of non-irradiated properties, e.g., strength anisotropy and chemical compatibility with solid lithium-based ceramics and lead-lithium liquid metal breeders, and (3) irradiation effects are specifically reviewed. Importantly high-temperature neutron irradiation effects on microstructural evolution, thermal and electrical conductivities and mechanical properties including the fiber/matrix interfacial strength are specified under various irradiation conditions, indicating seemingly very minor influence on the composite performance in the design temperature range.

1. Introduction

A new class of high-temperature structural materials, silicon carbide (SiC/SiC) composites, has been developed for fusion and other nuclear systems incorporating high performance/high radiation-resistant SiC fibers and matrix, and specially tailored fiber/matrix (F/M) interphases [1–3]. While the development efforts carried out over the past decade has resulted in a radiation-resistant 'nuclear-grade' SiC/SiC composite [1], development continues to improve on engineering properties of this composite. An example of such continued development is the nano-infiltration transient-eutectic-phase sintering (NITE) SiC/SiC composites [2,3]. With the completion of the 'proof-of-principle' phase, the R&D on SiC/SiC composites is shifting to the more pragmatic phase of development of industrial foundation and material data-basing, which is recognized as an essential tool for engineering design of SiC/SiC components. For that purpose, standardization of the material and test methodology is of particularly importance and will be discussed in the framework of international collaborations such as Broader Approach (BA) activities toward the DEMO design [4].

Of many proposed DEMO concepts utilizing SiC/SiC composites, a dual-coolant lead–lithium (Pb–17Li) breeder blanket system is particularly attractive and has been developed as a primary option in US and EU [5,6]. In this system, SiC/SiC composites are used as flow channel inserts (FCI), which act as a thermal and electrical insulator for steel structures from Pb–17Li. Understanding the irradiation effect on electrical conductivity is specifically important for this application because of the paucity of data. Application of SiC/SiC composites in the solid-breeder blanket is still viable and limited number of researches has been conducted.

This paper reviews recent advances in component production technology including joining and coating, research activities for the development of the design basis and mechanical property database, high-temperature irradiation effects on SiC and SiC/SiC composites, and chemical compatibility with either solid or liquid breeder.

2. Advances in component fabrication technology

2.1. Composite fabrication

With many efforts on material development in the past decade, fundamental technologies for production of 'nuclear-grade' SiC/SiC

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doi:10.1016/j.jnucmat.2008.12.305

composites have been established [1]. Specifically, chemical vapor infiltration (CVI) method is a well-developed composite fabrication technology and various components have been produced. For instance, a four inch-diameter braided composite tube has been successfully fabricated by CVI [7]. Presently, design-oriented tune of the composites is specifically addressed. To assist densification of the SiC matrix, nano whiskers were applied as seeds for the CVI-SiC growth [8]. In contrast, aiming to have thermal and electrical insulation for the FCI application, EU parties have developed very porous CVI SiC/SiC composites by controlling the mid-plane structure of the composites replaced by porous layers [9].

A NITE SiC/SiC composite as another candidate is promising to meet various industrial demands, i.e., mass production of various types of components with reasonably low production cost. Three types of NITE-SiC/SiC composites: (1) high-ductility, (2) high-strength, and (3) high-thermal conductivity types are presently available [10]. In accordance with commercialization of NITE-technology, various shapes and sizes of components (plates, thick-bricks, thin-wall tubes, large-diameter cylinders, etc.) can also be produced by the near-net shaping method [11].

With the recent progress in component fabrication technology, development of in-service inspection technology becomes an important technical issue for quality assurance of the components. For instance, many attempts to detect internal flaws using a non-distractive ultrasonic or an X-ray technique have been conducted [12].

2.2. Joining

Of many joining techniques [13–23], NITE-joining is very attractive, since it allows a robust structure with the same composition of the matrix of NITE-SiC/SiC composites [15]. A 200 mm-long NITE-tube joint was successfully manufactured (Fig. 1(a)). Additionally, good applicability of mechanical joints was demonstrated using screw-threaded tubes (Fig. 1(b)) [2,3]. With advances in NITE-joining and good machinability of dense and robust NITE-SiC/SiC composites, the channel structure of a heat exchanger unit was successfully produced. With the multiple-joining steps applied, the well-controlled microstructure of the joint was obtained [16]. One drawback to apply NITE-joining is a strict requirement of pressure under controlled environments. Alternatively, either high-strength joints using solid-state displacement reactions [17–22] or preceramic polymer joints [23], which are easily applicable and that could be used in field repair situations, have been developed. In parallel, to obtain reliable strength data of these joints, the torsional shear testing method, as one of the perceived

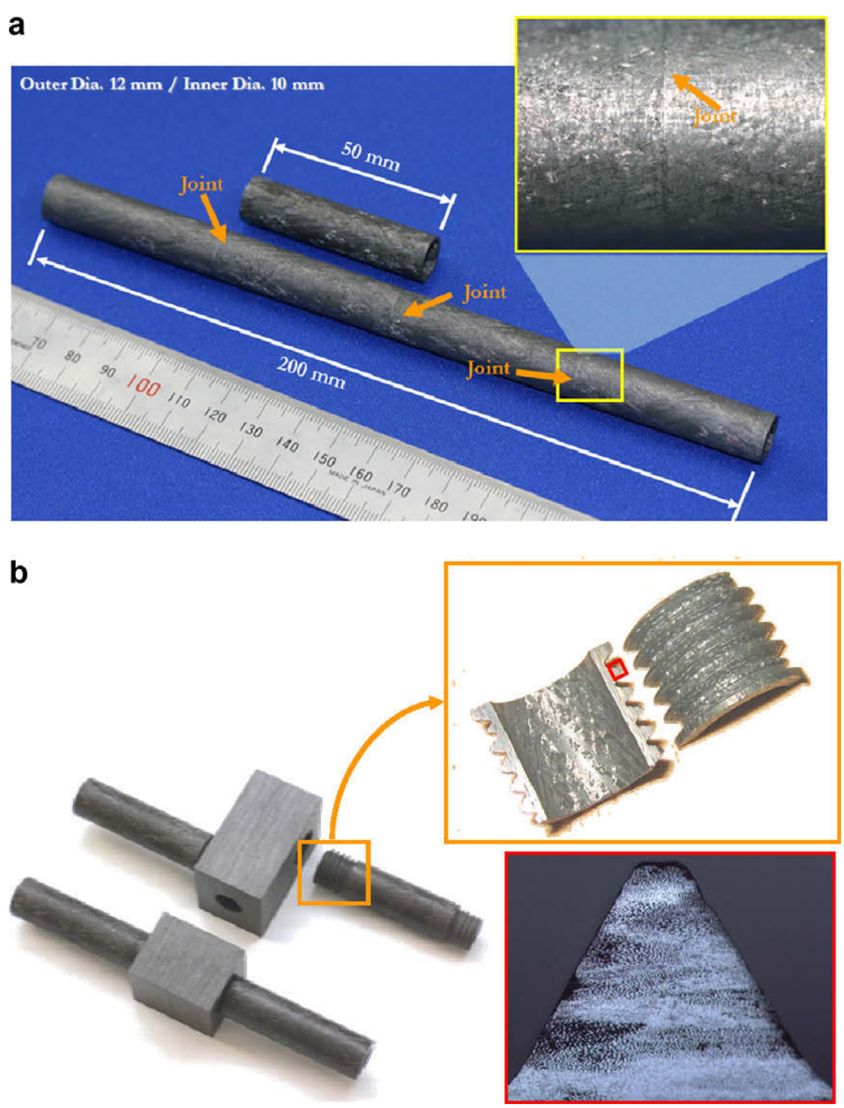

Fig. 1. Various types of joint components: (a) a 200 mm-long NITE-joined tube and (b) a screw-threaded NITE-SiC/SiC composite joint [3].

reliable test methods, has been developed under the international collaboration.

2.3. Coating

Metal tungsten coating on the surface of SiC/SiC composites has been developed for the plasma facing application of fusion [24–26]. A developmental hot pressing technique using nano-powders enables to provide a good bonding at the W/SiC joint interface [25]. To assess quality of the W-coating, in situ observation of crack propagation at the W/SiC joining interface was conducted using a high voltage electron microscopy with a piezoelectric driven indentation system [26].

3. Status of characterization of mechanical properties

There are two design concepts for engineering applications of composites: (1) 'damage tolerant' composites and (2) 'cracking-resistant' composites. The former composites are traditional ones with comparably high-ductility due to weak interfacial strength, while the latter is composites with high proportional limit stress (PLS) due to the strong F/M interface. For the primary safety issue, considering the failure behavior associated with matrix cracking is more important rather than composites' ultimate fracture since the matrix cracking is closely linked to the durability of the component functionality, e.g., structural stability, permeability and thermal/electrical insulation. In short, the high PLS, i.e., the high cracking-resistance, is essentially important for the structural/functional design of the composites. The damage tolerance is also important for the secondary safety purpose, i.e., functionality durability, proposing a leak before break concept for instance. The damage tolerance and crack tolerance issues are then being discussed because of their significance [27]. Also, establishment of such design basis for composite materials is targeted as a primary goal in the BA activity [4]. For that purpose, standardization of test methodology and development of a reliable and reproducible database deliverable to the DEMO design are ongoing.

Strength anisotropy is an important composites' specific issue for the component design. The strength anisotropy of advanced SiC/SiC composites was determined by the off-axial tensile test [28] or analytically estimated from the combination of in-plane shear and transverse-thickness test results [29]. Preliminary test results imply the importance of in-plane shear strength to improve off-axial strength for two-dimensional composites and inter-laminar shear strength for unidirectional composites. It is recognized that these shear properties depend significantly on the F/M interfacial strength. The off-axial strength was also evaluated for the braided tube and seemingly a good correlation with the plate results was identified [9]. In support of an improved dynamic crack growth model of SiC/SiC composites at elevated temperatures [30,31], time-dependent deformation of off-axial strength was also evaluated (Fig. 2) [32]. In the figure, experimental data was obtained at ambient temperature using single-edge-notched beams of each material. Note that snubbing friction was ignored or was set to a ratio of 200 compared to the interfacial shear friction. The model was also used to simulate a case where matrix spallation due to fiber bending would increase the fiber bending moment and reduce the toughness. The data point at 35° falls below all reasonable model assumptions. An inclined fiber bridging model captures a toughness decrease with increasing fiber inclination angle.

4. Irradiation effects

4.1. Microstructural evolution and swelling

Irradiation-induced microstructure of chemical-vapor-deposited (CVD) β-SiC has been investigated by neutron and ion irradiation [33–35]. The recent updates [36–38] specified the irradiation temperature dependence of void formation as (1) sparse formation of voids at 1050 °C, (2) relatively dense and small voids preferentially formed at stacking faults above 1300 °C, and (3) significant growth of voids at 1460 °C. It is worth noting that the voids appear to be faceted with {1 1 1} planes. In contrast, the dominant dislocation structure at high-temperatures is concluded as (1) detectable Frank loops and (2) black spot defects which were generally dominant at lower temperatures. When T_{irr} = 1460 °C, corresponding with the temperature at where steep increase of void size commences, the dominant defects turn into dislocation networks to the fluence of $\sim 8.5 \times 10^{25}$ n/m^2 due to the progressive growth and development of defects. Of particular importance is that the effect of cavity swelling <1300 °C is negligibly small. Fig. 3 shows a resultant map of irradiation-induced microstructure of SiC. There seems no significant impact on the application of advanced SiC/SiC composites in the assumed operating temperature range.

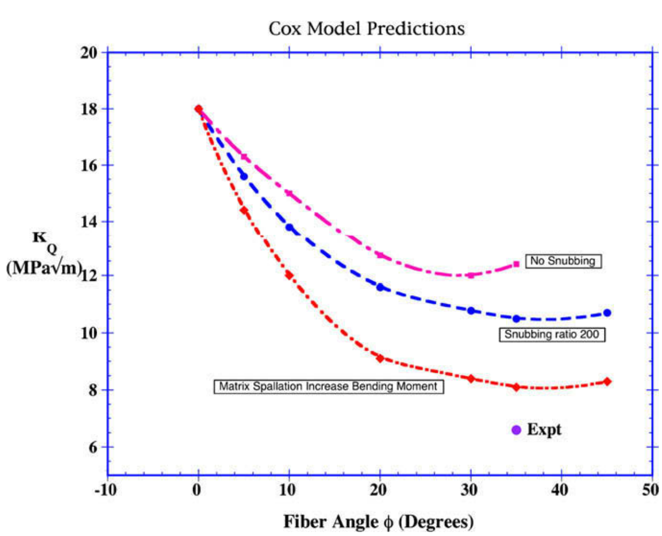

Fig. 2. Model calculations of composite toughness, reported as K_Q values, for static loading at ambient temperature as a function of fiber inclination angle [32].

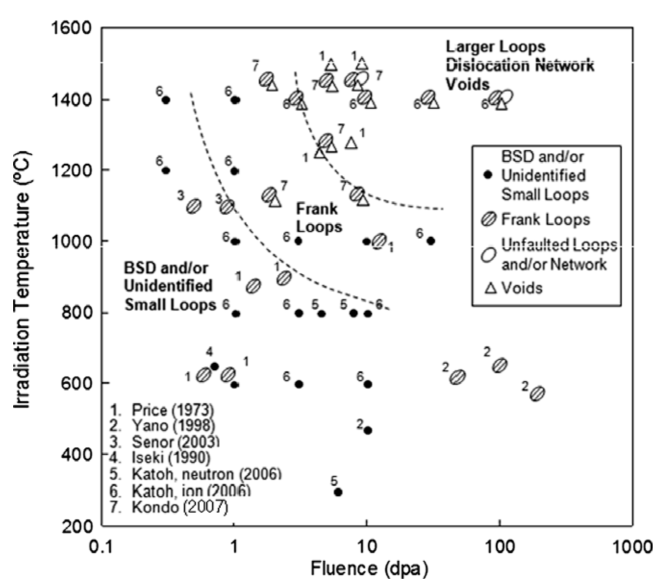

Fig. 3. Summary of irradiation-induced microstructure of CVD-SiC [36].

4.2. Thermal conductivity

Thermal conductivity of neutron-irradiated SiC has been reported by many authors [36,39–41]. The rapid decrease and saturation of the thermal conductivity of CVD-SiC in the point-defect regime was analyzed in terms of the thermal resistance induced by neutron irradiation [41]. From the recent updates at higher-temperature irradiation (Fig. 4), it is clearly shown that as the microstructure makes a transition from the point-defect swelling regime to that associated with void swelling that the thermal conductivity, as governed by the phonon scattering from these irradiation-induced defects, undergoes a fundamental shift when T_{irr} = 1000 °C. For irradiation above this temperature, the thermal conductivities are not expected to saturate due to the formation of voids and other complex defects in the high-temperature regime.

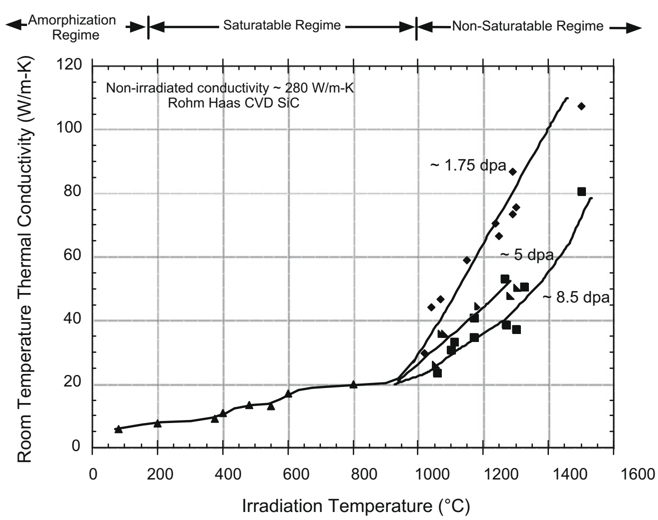

Fig. 4. The thermal conductivity change of SiC by neutron irradiation at high-temperatures [36].

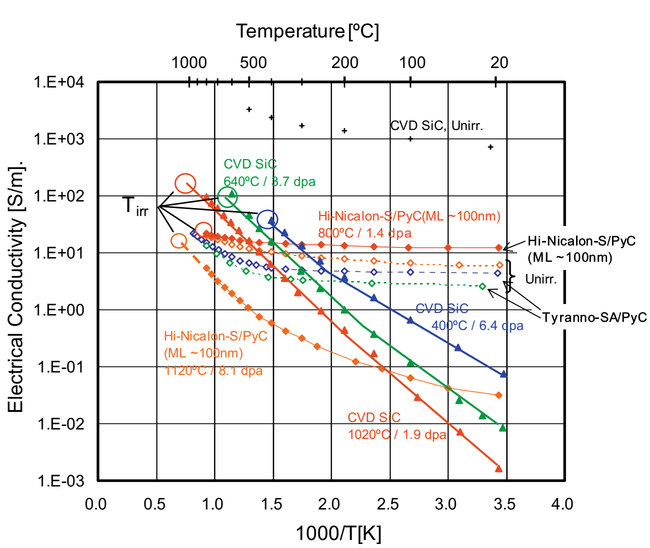

Fig. 5. Neutron irradiation effects on electrical resistivity of SiC and CVI-SiC/SiC composites [43].

4.3. Electrical resistivity

To enable adequate MHD/thermo-mechanical design of dual-coolant lead-lithium breeder blankets and to develop appropriate FCI materials, it is essential to understand the irradiation effect on the electrical resistivity of SiC ceramics and composites. Post-irradiation electrical conductivity for CVD-SiC and CVI-SiC/SiC composites were identified up to 1.4–8.1 dpa at 800–1120 °C (Fig. 5) [42,43]. Through-thickness electrical conductivity of neutron-irradiated 2D SiC/SiC composites with thin pyrolytic carbon (PyC) interphase appeared to be in the order of 10 S/m in the typical operating temperature range for FCI. Applying SiC over-coating appeared beneficial in reducing electrical conductivity at relatively low temperatures in expense of more significant irradiation effect and steeper temperature dependence.

The experimental results were successfully analyzed by constitutive modeling of the anisotropic electrical transport in two-dimensional woven fabric composites. It was revealed that the interphase network provides imperfect bypass through fabric layers for through-thickness conduction, while axial interphase conduction dominates in most conditions for in-plane conductance. Specifically, over-coating or internal layer of semi-conducting SiC add a serial resister to the circuit. Electrical conductivity tailoring by engineered interphase structure and configuration in composite materials appears feasible with the aid of modeling. Under neutron irradiation, PyC interphase conductivity may increases, while SiC semi-conducting properties become dictated by radiation defects. Transmutation plays a minor role in fission neutron irradiation, however, that will unlikely be the case for fusion neutrons.

4.4. Mechanical properties

The radiation tolerance of strength of high-crystallinity and near-stoichiometric SiC and SiC/SiC composites has been proven at intermediate temperatures by many authors [33,36,44–49]. Specifically, high-temperature irradiation stability of CVI-SiC/SiC composites has been proven up to T_{irr} = 1100 °C. Furthermore, good radiation stability of liquid-phase-sintered (LPS) SiC as a corre-

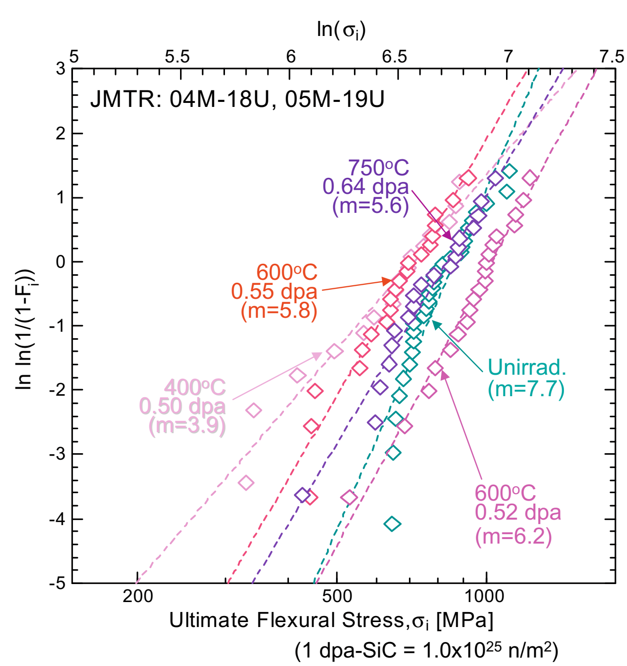

Fig. 6. The effect of neutron irradiation on the Weibull statistical bend strength of LPS-SiC as the corresponding matrix phase of NITE-SiC/SiC composites [47].

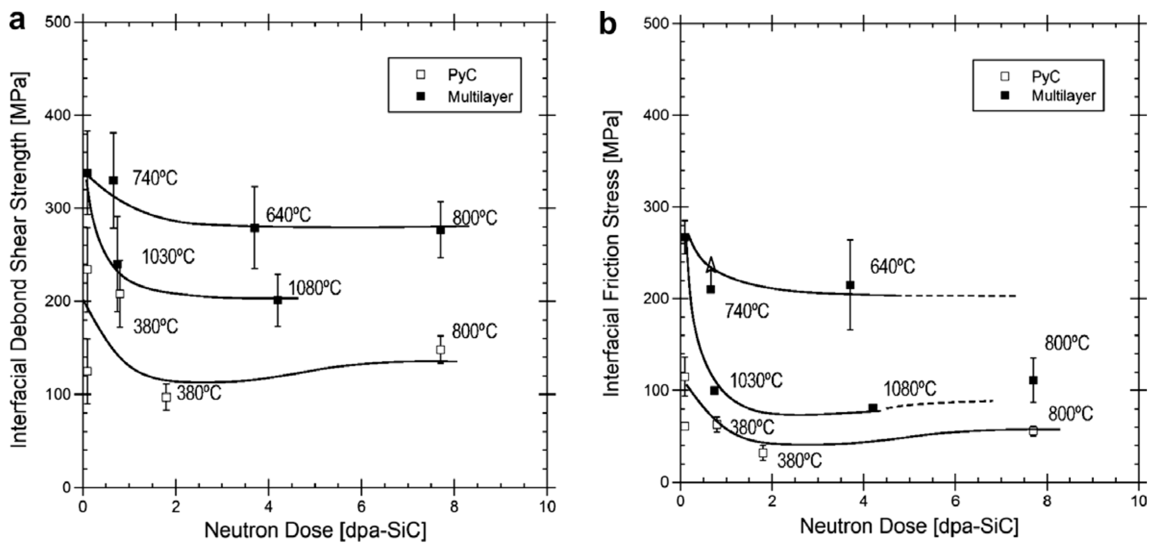

Fig. 7. The effect of neutron irradiation on (a) interfacial debond shear strength and (b) interfacial friction stress at the fiber/matrix interface of CVI-SiC/SiC composites [51]. Irradiation temperatures are noted in the figure.

sponding matrix phase of NITE-SiC/SiC composites has been demonstrated up to 0.5 dpa at intermediate temperatures (Fig. 6) [47], although some degradation had been concerned by the presence of sintering additives as inferred from the previous results for impure SiC materials [36].

Irradiation effect on the F/M interfacial strength was evaluated by applying analytical models to the fiber push-out test results [50,51]. It was obvious that both an interfacial debond shear strength and an interfacial friction stress slightly decrease by irradiation depending on neutron dose rather than irradiation temperature for both PyC monolayer and PyC/SiC multilayer interphases when T_{irr} < 1000 °C (Fig. 7). Contrarily, when T_{irr} > 1000 °C, further deterioration of interfacial shear properties and bulk strength was identified under certain irradiation conditions. However, reasonably high interfacial shear strength even after irradiation results in very minor influence on the bulk strength [48,49]. Note that the high interfacial debond shear strength closely link to the high PLS. As aforementioned, this results in improved cracking-resistance rather than damage tolerance. In contrast, the damage tolerance depends on interfacial friction, crack density and others. For further understanding at the interfacial stress interaction, the effect of stress-relief at the interface by irradiation creep needs to be discussed [52].

5. Chemical compatibility

Chemical compatibility of SiC/SiC composites with coolant and breeding materials is of great importance to ensure the reliability of the fusion blanket system. Supposing a helium-cooled solid-breeder blanket, compatibility between CVD-SiC and Li-based ceramics ($LiAlO_2$, Li_4SiO_4, Li_2ZrO_3, and Li_2TiO_3) was evaluated at 900 and 1000 °C for 100 h in helium [53]. The preliminary result specifies formation of (1) an adhering substance of Li_2SiO_3 on the SiC surface except for the SiC–$LiAlO_2$ pair at 900 °C and (2) a reaction phase only in the SiC–Li_4SiO_4 pair at 1000 °C. Oxidation by a limited amount of oxygen contained in a helium coolant is another potential concern. Oxidation kinetics of SiC and PyC on a state of the F/M interface is being identified [54]. In contrast, good chemical stability of SiC with Pb–17Li as a breeding material in the liquid blanket system was previously demonstrated <1100 °C [55]. Presently erosion/corrosion behavior of SiC/SiC composites in a Pb–17Li fluid is being investigated [56].

6. Other critical issues toward DEMO

Critical issues still remain for this class of materials, including (1) determination of the strength limit, correlated with the failure behavior, including a strength anisotropy map and lifetime evaluation, i.e., fatigue and creep, (2) high-neutron fluence and high-temperature irradiation effects with considerations about the He/H synergistic effects and dynamic deformation behavior, e.g., irradiation creep, (3) He/H permeability and retention, (4) influence of nuclear transmutation other than He/H with an origin of metal impurities, and (5) component performance characterization including joining and coating under fusion relevant environments.

7. Conclusions

With advances in development of radiation-resistant 'nuclear-grade' SiC/SiC composites, the R&D on SiC/SiC composites is shifting toward the more pragmatic phase, i.e., industrialization of component manufactures and data-basing. This paper reviewed recent advances in (1) component production technology development including joining and coating, (2) characterization of mechanical properties, (3) irradiation effect studies and (4) chemical compatibility with breeding materials. It is concluded that considerable progress has been made in all 4 domains.

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