Evaluation on Failure Resistance to Develop Design Basis for Quasi-Ductile Silicon Carbide Composites for Fusion Application

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Abstract. Silicon carbide composites (SiC/SiC) are promising candidate materials for fusion reactors. For the practical application of this class of materials, a design basis for SiC/SiC composites needs to be developed because of inherent brittle-like fracture, i.e., quasi-ductility, which is totally different from ductility of metals. For this purpose, the failure behavior, i.e., matrix cracking behavior, of a new class of radiation-resistant SiC/SiC composites, e.g., a nano-infiltration transient-eutectic phase (NITE) SiC/SiC composites, was evaluated. The single-edge notched bend test results identified notch-insensitivity of the composites regardless of the specimen size. This suggests an apparent correlation between stress and energy criterions, possibly eliminating a parameter required for the mechanical property database development. According to this fact, a fracture resistance as an energy parameter for failure analysis of the composites has been developed based on the non-linear fracture mechanics. A preliminary model could separately distinguish contributions of micro-cracking from macro-crack formation energy. Good crack resistance of pilot-grade NITE-SiC/SiC composites was finally demonstrated.

1. Introduction

SiC/SiC composites are attractive materials for nuclear energy systems due to the inherently good chemical stability at high-temperatures, strength retention, specific strength, low-activation/low after-heat properties. Of many composite types, a NITE-SiC/SiC composite, as well as a chemical-vapor-infiltration (CVI) SiC/SiC composite, is believed to be viable because of excellent baseline mechanical properties [1-3] and proven radiation stability of microstructure and strength under certain irradiation conditions [4]. The good gas tightness of the dense NITE-SiC/SiC composites would provide an additional advantage to apply it to the gas-cooled systems [5]. Accordingly, several fusion blanket designs propose to utilize this class of SiC/SiC composites, e.g., flow channel inserts in the lead-lithium breeder blankets [6, 7] or structural components [8]. With a completion of the “proof-of-principle” phase, the R&D on SiC/SiC composites is shifting to the more pragmatic phase like material data-basing. For instance, generation of engineering database deliverable for design activities of a high-temperature operating advanced DEMO reactor is implemented with a primary priority in the R&D on SiC/SiC composites for the Broader Approach (BA) activity [9].

When considering composite materials, inherent quasi-ductility in fracture gives significant difficulty in component design. Quasi-ductility of composites is totally different from the ductility of metals since this quasi-ductility occurs as a result of cumulative accumulation of irreversible permanent damages. For structural design of the composites, the failure behavior, i.e., matrix crack pop-in and crack extension, should be evaluated since functionality and structural stability of composites often deteriorates by failure initiation. This failure behavior depends significantly on the type and distribution of the internal flaws as a crack origin, probably giving a big difference between dense NITE-SiC/SiC composites and conventional porous composites. The failure initiation behavior would also vary from the choice of fabric architecture and applied loading directions due to inherent anisotropy of the composites and therefore careful discussion is necessary.

From these aspects, the damage accumulation behavior of SiC/SiC composites was first evaluated with a final goal to develop a design basis for generation of practical database with a direction to use this class of composites for structural application. For this purpose, the fracture resistance of composites was evaluated to determine a failure criterion for database development.

2. Experimental

A pilot-grade unidirectional (UD) SiC/SiC composite was fabricated by the nano-infiltration transient-eutectic phase sintering (NITE) method (Institute of Energy Science and Technology, Co., Ltd., Japan). Details of the NITE process are reported elsewhere [1-3]. A highly-crystalline and near-stoichiometric Tyranno™-SA 3rd-grade SiC fiber with a volume fraction of ~0.4 was applied as reinforcements. A ~250 nm-thick pyrolytic carbon (PyC) interphase as a form of the fiber/matrix (F/M) interface was chemically-vapor-deposited on the fiber surface prior matrix densification. A typical micrograph (FIG. 1) shows well-densified matrix (~2.93 g/cm^3) so that the porosity of this material is very low (~7%). For the pilot-grade NITE-SiC/SiC composites, a secondary phase (white contrast in FIG. 1), which is reportedly an oxide composed of sintering additives such as Al_2O_3, SiO_2 and Y_2O_3 [10], was localized in the matrix, specifically within intra-bundles. This oxide phase would however not significantly impact test results at room-temperature.

Fracture resistance was evaluated by the single-edge notched-bend (SENB) test technique. Various size specimens with a different initial notch depth were applied (FIG. 2). Note that the width to length ratio was fixed for all specimen types. For comparison, un-notched bend specimens with a size of 20×4×1.5 mm^3 were tested. The fiber longitudinal direction is set parallel to the specimen longitudinal direction. Test coupons including an artificial notch were machined from the composite plate by a diamond saw. The specimen surfaces were then polished by the standard metallographic technique with a surface finish of ~1μm for the crack extension observation. The radius of the notch root was approximately ~150 μm. The SENB tests were conducted at room-temperature using an electromechanical testing machine. Test specimens were loaded using a three-point bend fixture with a support span of 16 mm for SENB-1~3 and 32 mm for SENB-4. The crack opening displacement (COD) was measured by the clip-on type gauge. A constant crosshead displacement rate was 0.1 mm/min. The unloading/reloading sequences were applied to evaluate the damage accumulation behavior during the tests.

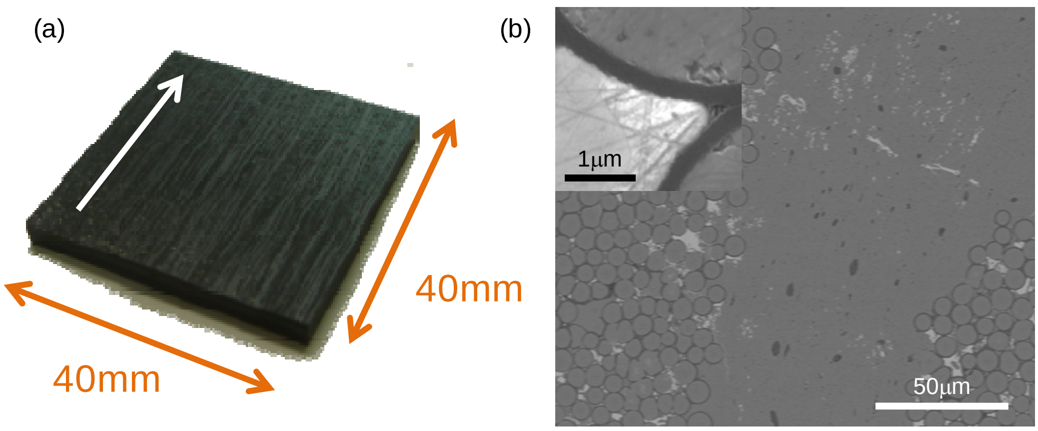
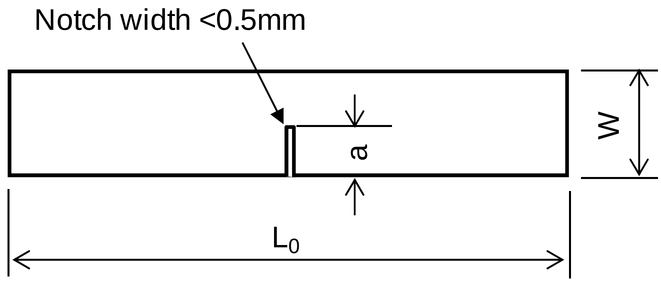


FIG. 1. (a) Outlook and (b) typical microstructures of pilot-grade NITE-SiC/SiC composites.



ID	Total Length, L_0 [mm]	Support Length, L [mm]	Width, W [mm]	Thickness, t [mm]	Notch Depth, a [mm]	a/W
SENB-1a	20	16	4	1	1	0.25
SENB-1b	20	16	4	1	2	0.50
SENB-2a	20	16	4	2	1	0.25
SENB-2b	20	16	4	2	2	0.50
SENB-3a	20	16	4	4	1	0.25
SENB-3b	20	16	4	4	2	0.50
SENB-4	40	32	8	4	4	0.50

FIG. 2. Schematic illustrations of single-edge notched-bend specimens.

A crack extension length from the initial notch was measured by the optical microscopy. For that purpose, the replica films of the surface microstructure of the specimen were prepared at each unloading point.

3. Results and Discussion

3.1. Fracture Behavior in SENB tests

FIG. 3 shows a typical load vs. crack opening displacement curve with micrographs of the specimen surfaces in each loading stage. In this figure, crack length measured by the replica method was also plotted. According to the microstructural observation, apparent three damage stages in fracture behavior were identified: 1) initial elastic segment followed by non-linear stage due to micro-crack formation, 2) macro-crack extension from the notch root with a rapid load drop, and 3) load transferring by friction at the fiber/matrix interface, coupled with crack branching and fiber breaks. Of particular emphasis is that many micro-cracks were formed along the fiber longitudinal direction near the root of the notch in the first stage however no macro cracking in the loading direction from the root of notch was observed. In the second stage, macro-crack length linearly increased with increasing crack opening displacement. Compared with the rapid crack extension in the second stage, a mild increase of the crack length was obtained in the third stage, i.e., in the mixed failure accumulation process.

From FIG. 3, two characteristic parameters: a proportional limit strength (PLS) as an initiation load of micro-cracking and a maximum flexural strength (UFS) as an initiation load of macro-cracking with cumulative micro-crack formations were identified. FIG. 4 shows both PLS and UFS normalized as a flexural stress form with respect to a function of the notch depth to width ratio. An important remark from this figure is that pilot-grade NITE-SiC/SiC composites are notch insensitive. This means that the slopes of the linear fit give unique PLS and UFS regardless of the presence of initial notches.

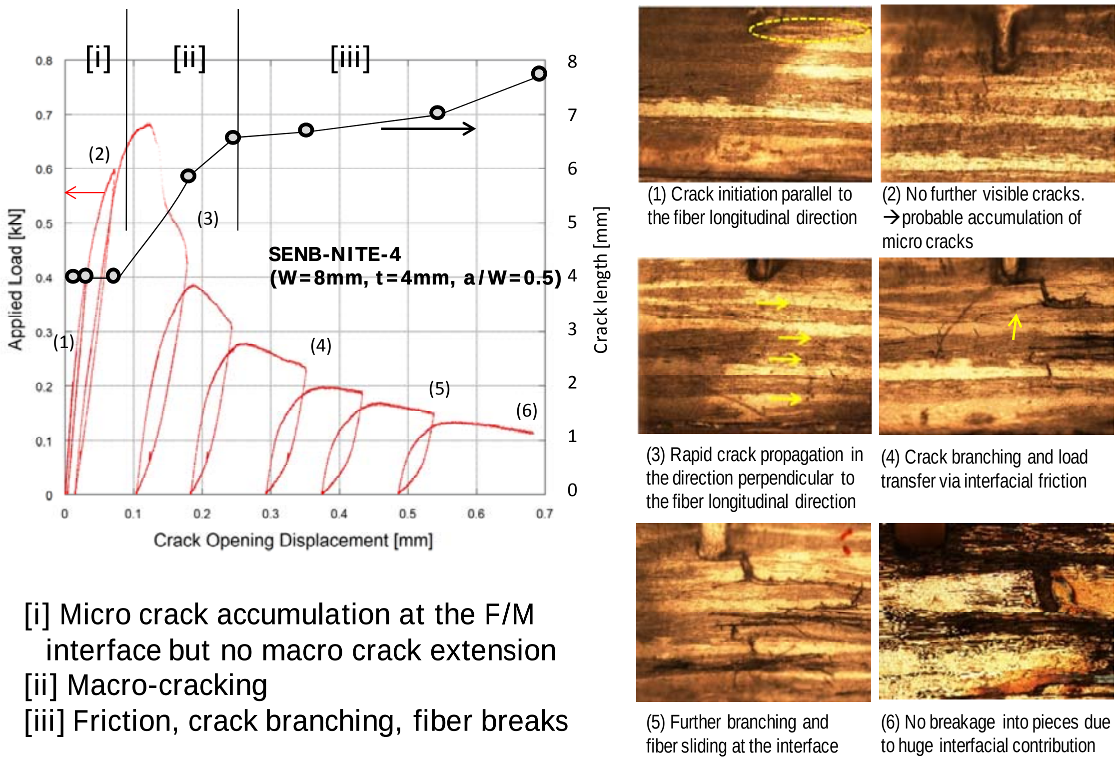


FIG. 3. Typical SENB behavior of pilot-grade NITE-SiC/SiC composites.

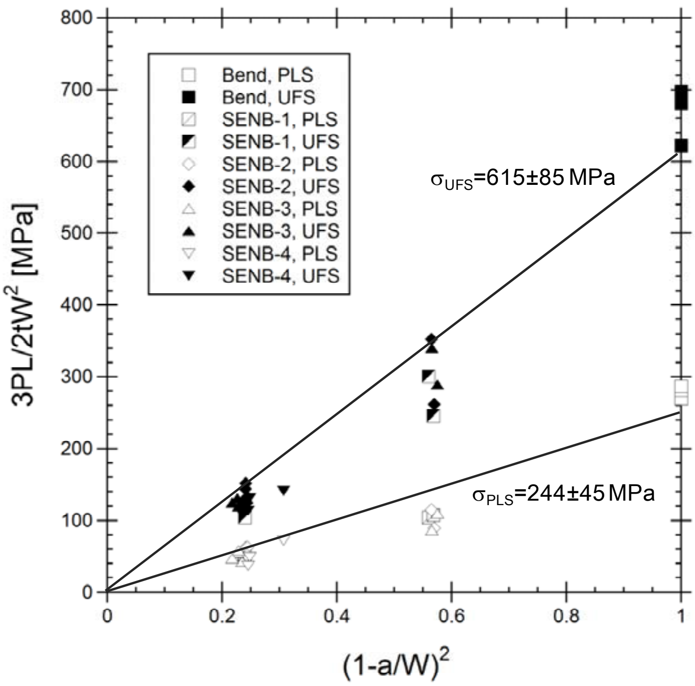


FIG. 4. Notch Sensitivity of pilot-grade NITE-SiC/SiC composites.

As a supplemental finding, FIG. 4 also identified that the notch insensitivity was independent of specimen size. Developing a small specimen test technique is one of important technical issues for the irradiation effect studies. Considering a notched specimen is beneficial to control the fracture location we desire, the design of miniature notched specimens should be essentially important and definitely effective.

3.2. Fracture Resistance

The fact of notch insensitivity is somehow surprising when considering that ceramic composites generally fail in a brittle-like manner. When developing a design basis of this class of composites, this notch insensitivity becomes very important since this implies an apparent correlation between stress and fracture energy criterions in fracture of the composites. When this relationship is clearly identified, unique failure criterion can be utilized for the component design. An energy-based criterion is proposed and preliminarily evaluated hereafter.

Test results indicate an apparent improvement of ductility, i.e., energy consumption during irreversible damage accumulation beyond matrix cracking due to the high interfacial friction on the rough-surface of highly-crystalline SiC fibers. Because of this quasi-ductility, an analytical model based on the non-linear fracture mechanics [11, 12] was applied to separately discuss the effect of irreversible energies such as interfacial friction, thermal-residual strain energy and fiber breaks. In this analysis, the total work during the SENB test (w) is expressed as:

$$w = U_e + U_{fr} + U_r + \Gamma \, , \qquad (1)$$

where elastic energy (U_e), friction energy at the interface (U_{fr}), residual strain energy (U_r), and crack surface formation energy (Γ) are defined in FIG. 5 and they are plotted as a function of crack opening displacement (FIG. 6). Note that the crack surface formation energy includes micro- and macro-crack forming energies together. Then, the fracture resistance (G) can be defined as:

$$G = \frac{\partial \Gamma}{t \partial a} = \frac{1}{t}\frac{\partial \Gamma}{\partial x}\frac{\partial x}{\partial a} \, , \qquad (2)$$

where the crack length (a), specimen thickness (t) and crack opening displacement (x).

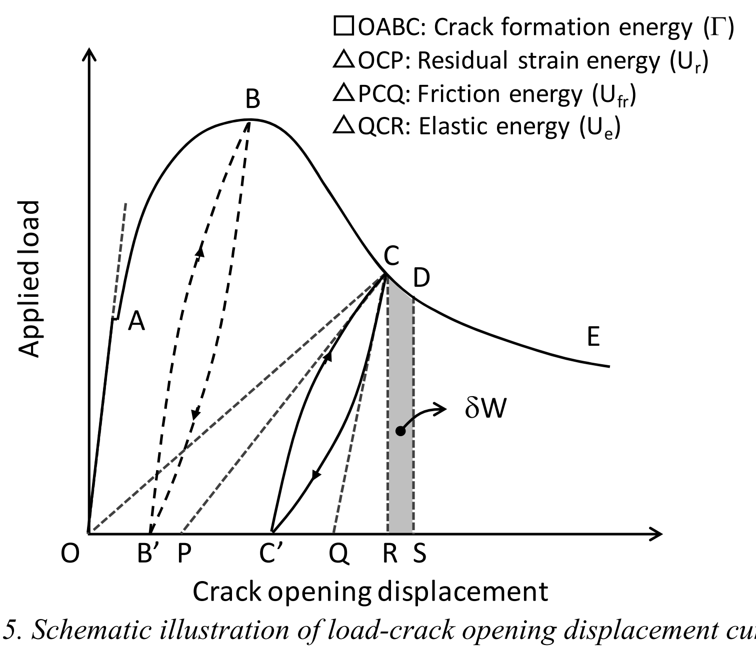


FIG. 5. Schematic illustration of load-crack opening displacement curve.

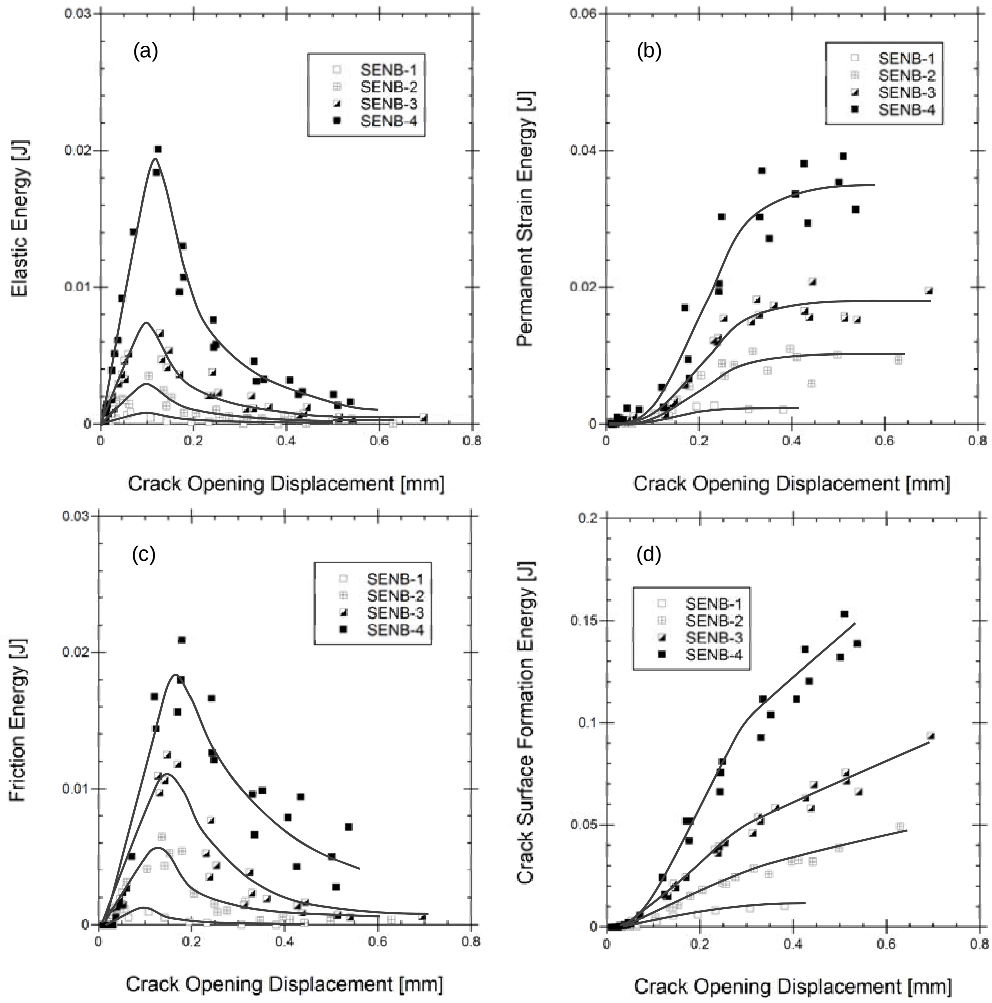


FIG. 6. Crack surface formation energy vs. crack opening displacement.

From FIG. 6, it is apparent that the elastic energy and small portion of friction energy were dominant in the first stage. Whereas, the crack surface formation energy rapidly increase with a release of residual thermal strain energy by interfacial debonding in the second stage. In the third stage, crack branching and fiber breaks consumed much energy to form new crack surfaces. Of particular emphasis is that the crack surface formation energy seems proportional to the crack opening displacement in the second stage. Accompanied with the result of the crack length change obtained from FIG. 3, a fracture resistance of ~5 kJ/m^2 was finally obtained for pilot-grade NITE-SiC/SiC composites using Eq. (2). Specifically no significant size effect was obtained for certain test conditions.

The fracture resistance defined in Eq. (2) however cannot perfectly distinguish contributions from micro- and macro-crack formations. This issue is discussed by considering an energy release rate by micro-crack accumulations until macro-crack causes. FIG. 7 shows crack surface formation energy with respect to the initial notch depth to width ratio using various specimens. Micro-crack surface formation energy (Γ_m) was then empirically obtained as:

$$\Gamma_m \cong \sum_{i=1}^{n} C_i \left(1 - \frac{a}{W}\right)^i , \tag{3}$$

where C_i (i=1,2, …, n) are constants. Then, an energy release rate (G_c) is then defined as:

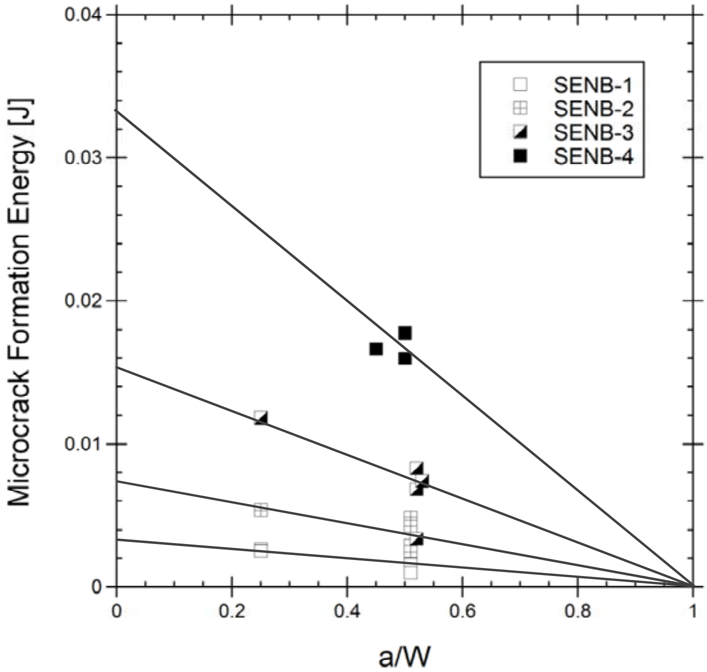


FIG. 7. Micro-crack formation energy vs. initial notch depth.

$$G_c \equiv -\frac{\Delta \Gamma_m}{t\Delta a} \cong \frac{1}{Wt}\sum_{i=1}^{n} iC_i\left(1-\frac{a}{W}\right)^{i-1}, \tag{4}$$

Simply applying linear fit in FIG. 7, we obtained an energy release rate of ~1.2 kJ/m^2 for each specimen type. This value is quite lower than that of conventional polymer-impregnation-pyrolysis (PIP) SiC/SiC composites, ~4.8 kJ/m^2 [13]. This implies more crack resistant for pilot-grade NITE-SiC/SiC composites, since this energy release rate means crack density in damage process. In contrast, it is concluded that PIP-SiC/SiC composites are more damage tolerant. Eventually, the actual fracture resistance for macro-cracking of ~3.8 kJ/m^2 was estimated for pilot-grade NITE-SiC/SiC composites.

Recent cyclic unloading/reloading test result [14] also demonstrated that NITE-SiC/SiC composites are crack resistant compared with the conventional porous chemical-vapor-infiltrated (CVI) SiC/SiC composites. No fatigue cracks critical to deteriorate composite lifetime were identified for NITE-SiC/SiC composites, while CVI-SiC/SiC composites show a rapid modulus decrease upon crack initiation, i.e., progressive damage accumulations. The improved crack resistance of NITE-SiC/SiC composites thus results in better helium gas tightness (10^{-10}~10^{-9} m^2/s) than that of CVI-SiC/SiC composites (10^{-6}~10^{-4} m^2/s). Very minor deterioration of the gas permeability was also identified for NITE-SiC/SiC composites damaged by mechanical loading beyond a matrix cracking stress.

4. Summary

Silicon carbide composites are promising candidate materials for fusion reactors with advanced features such as high thermal efficiency. This paper provided a present status in development of design basis for quasi-ductile SiC/SiC composites as a structural application. For that purpose, failure behavior, i.e., matrix cracking behavior, of a new class of radiation-resistant SiC/SiC composites, e.g., a NITE-SiC/SiC composite, was evaluated. With a fact of the notch-insensitivity, a correlation between the stress and energy criterions was implied. Accordingly, this study provided a rigorous solution to distinguish contributions of micro-cracking from macro crack extension energy, giving a fracture resistance as an energy failure criterion. Applying this method, good crack resistance of pilot-grade NITE-SiC/SiC composites was demonstrated.

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