2.4 EVALUATION OF DAMAGE TOLERANCE OF ADVANCED SiC/SiC COMPOSITES AFTER NEUTRON IRRADIATION—K. Ozawa, Y. Katoh, L.L. Snead (Oak Ridge National Laboratory), T. Nozawa (Japan Atomic Energy Agency), T. Hinoki (Kyoto University)

OBJECTIVE

The main objective of this study is to determine the neutron irradiation effects on damage tolerance of SiC/SiC composites with near-stoichiometric fibers and two different matrix types. In the current study, we applied non-linear fracture mechanics, based on actual crack increment measurements.

SUMMARY

The effect of neutron irradiation on damage tolerance of two nuclear grade SiC/SiC composites (plain-woven Hi-Nicalon™ Type-S fiber-reinforced, CVI SiC matrix composites with a multilayer interphase and unidirectional Tyranno™-SA3 fiber-reinforced, NITE matrix with a carbon mono-layer interphase) was evaluated by means of miniaturized single-edged notched-beam test. No significant changes in crack extension behavior and in the load-loadpoint displacement characteristics such as the peak load and hysteresis loop width were observed after irradiation to 5.9×10^{25} n/m^2 (E > 0.1 MeV) at 800°C and to 5.8×10^{25} n/m^2 at 1300°C. The global energy balance analysis based on non-linear fracture mechanics estimated the energy release rate contributed by macro-crack initiation to be 3±2 kJ/m^2 for both the unirradiated and irradiated composites. The effects of neutron irradiation on fracture resistance of these composites appeared insignificant for the conditions examined.

PROGRESS AND STATUS

Introduction

Silicon carbide fiber reinforced silicon carbide matrix (SiC/SiC) composites are attractive candidate materials for structural and functional components in fusion energy systems due to the good radiation stability coupled with inherently low induced radioactivity and after-heat [1, 2]. For the new class of "nuclear grade" SiC/SiC composites, determining fracture resistance is one of the most important issues to be investigated. However, this has been challenging because composites consist of several constituents and hence the fracture resistance of composites with non-uniform properties cannot be determined by applying common methods for the linear-elastic plane-strain fracture toughness determination (such as ASTM E-399) or the compliance method based on linear-elastic fracture mechanics (LEFM) for a homogeneous material. Since no full-consensus test standards are available for damage tolerance determination of ceramic continuous fiber reinforced ceramic matrix composites, even though some ASTM standards for polymer composites (D 5528 for Mode I Interlaminar toughness and D 6671 for Mixed I and II Mode) are available, various methods of experiments and analyses have been proposed by researchers [3-8]. Moreover, there are no published results on the effects of irradiation on damage tolerance, while there have been extensive efforts to study the irradiation effects in the SiC/SiC composite system [9-13]. There has been a report on fracture toughness of Hi-Nicalon™ Type-S fiber reinforced SiC matrix composites after neutron irradiation to 4.3×10^{24} n/m^2 at 40°C, unfortunately with no detailed information disclosed [14]. The main objective of this study is to determine the neutron irradiation effects on fracture toughness of SiC/SiC composites with near-stoichiometric fibers and the matrices. In the current study, we attempted to apply non-linear fracture mechanics, based on actual crack increment measurements.

Experimental

Hi-Nicalon™ Type-S plain-woven fabric-reinforced CVI matrix composites with $(PyC_{20}/SiC_{100})_5$ multilayer interphase composite (HNLS-CVI-ML) and unidirectional Tyranno™-SA3 reinforced with 500 nm PyC mono-layer interphase and nano-infiltration and transient eutectic-phase (NITE) SiC composite (TySA-NITE) were prepared. Detailed information about the composites is given elsewhere [15, 16]. The materials were machined into miniature single-edged notched-beam (SENB) specimens for in-plane

mode-I fracture behavior evaluation. Effects of the test specimen size on the fracture behavior have been studied and are discussed elsewhere [17, 18]. Neutron irradiation was conducted in the High Flux Isotope Reactor (HFIR) at the Oak Ridge National Laboratory as a part of RB*-18J campaign. The peak neutron fluence and the nominal irradiation temperature were ~5.9×10^{25} n/m^2 (E > 0.1 MeV) at 800°C and ~5.8×10^{25} n/m^2 at 1300°C. An equivalence of one displacement per atom (dpa) = 1×10^{25} n/m^2 (E > 0.1 MeV) is assumed. The SENB tests were conducted at room-temperature using an electromechanical testing machine with a load capacity of 1 or 10 kN. Test specimens were loaded using a three-point bend fixture with a support span of 16 mm under a constant crosshead displacement rate of 0.05 mm/min. Loadpoint displacement was measured using a clip-on crack opening gauge. The unloading-reloading sequences were applied to evaluate the damage accumulation behavior during testing. Crack length at each loading-unloading cycle was evaluated by optical microscope observations of the replica films. Detail experimental procedure is given elsewhere [19].

Results and Discussion

Typical load-loadpoint displacement (P-u) curves of the composites before/after neutron irradiation are reproduced in Figure 1. Both composites exhibited typical quasi-ductile behavior. No significant changes in various features of the P-u curves such as peak load and hysteresis loop width following neutron irradiation were observed for either composite type.

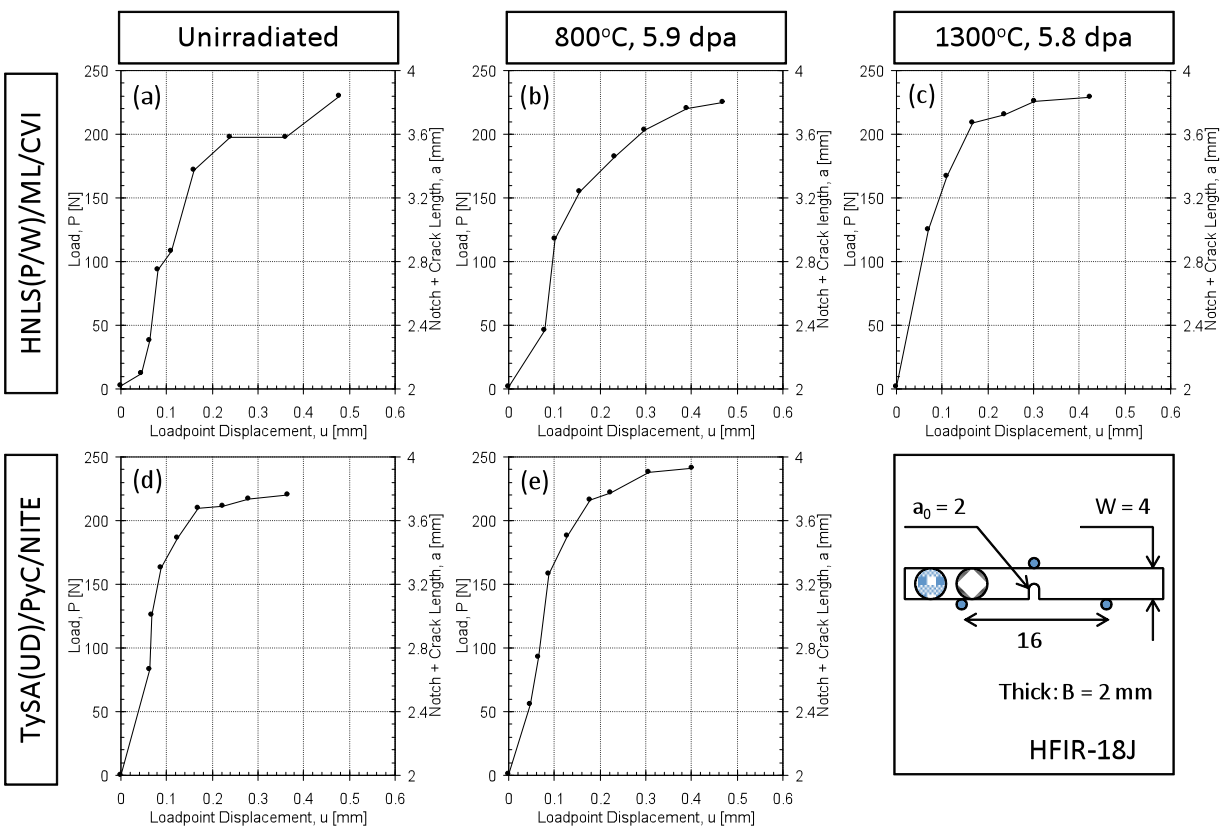

Figure 1. Typical load-loadpoint displacement curves for the unirradiated and irradiated HNLS-CVI-ML and TySA-NITE comoposites.

Because of the apparent quasi-ductility of the composites, application of an analytical model based on non-linear fracture mechanics [20, 21] for determining fracture resistance is considered suitable. In the present work, the analytical method developed by Nozawa *et al.* [17, 18] has been applied. The definition of each energy is shown in Figure 2 and the detailed analytical procedure is given elsewhere [19]. In brief,

the apparent fracture resistance (G_{total}) and energy release rate for micro-crack formation (G_{micro}) were calculated from the following equations:

$$G_{total} \equiv \frac{1}{B}\frac{\partial \Gamma_{total}}{\partial a} = \frac{1}{B}\frac{\Delta \Gamma_{total}}{\Delta u}\frac{\Delta u}{\Delta a} \tag{1}$$

$$G_{micro} \equiv -\frac{1}{B}\frac{\Delta \Gamma_{micro}}{\Delta a} \cong \frac{1}{WB}\sum_{i=1}^{n} iC_i\left(1-\frac{a}{W}\right)^{i-1} \tag{2}$$

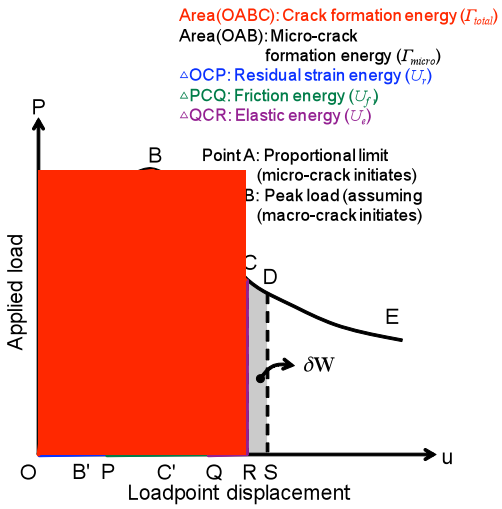

Figure 2. Schematic illustration of P-u curve and definition of each energy.

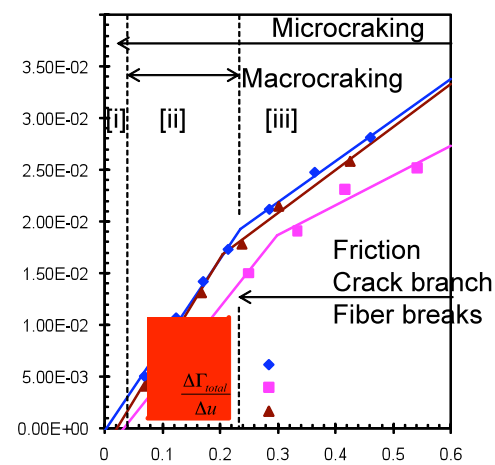

Figure 3. Total crack formation energy (Γ_{total}) and definition of $\Delta\Gamma_{total}/\Delta u$.

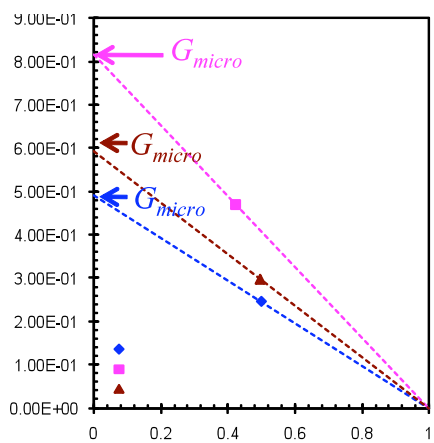

Figure 4. G_{micro} determination.

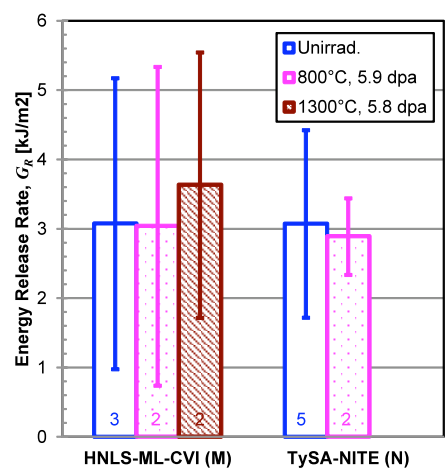

Figure 5. Non-linear energy release rate of unirradiated and irradiated (a) HNLS-CVI-ML and (b) TySA-NITE composite. Error bars, and numbers in each bar denote one standard deviation, and the number of valid tests.

with Γ_{total}, being the apparent surface crack formation energy, Γ_{micro} the micro-crack formation energy, a the crack length, B the specimen thickness, W the specimen width, u the loadpoint displacement, C_i the constants. The $\Delta\Gamma_{total}/\Delta u$ values were obtained from the slope of Γ_{total}-u graph at the stage [ii], corresponding to the macro-crack extension, as shown in Figure 3. The $\Delta u/\Delta a$ values were measured from the plots of the actual crack length change in the stage [ii] in Figure 1. The G_{micro} value in Eq. (2) was obtained by linear-fitting (Figure 4). The actual energy release rate was given by $G_{macro} = G_{total} - G_{micro}$. Eventually, the energy release rate contributed only by macro-crack initiation of 3±2 kJ/m^2 was estimated for either type of composite before and after irradiation (Figure 5).

Significant scatter was observed in the energy release rate analysis, likely due to the limited specimen dimensions as compared to the weave unit cell size. However, the damage tolerance properties of these composites did not appear to have changed remarkably, based on the present result on the energy release rate analysis. The previous results of the tensile tests after neutron irradiation and the hysteresis loop analysis estimating the interfacial properties further support this conclusion [16,22]. As shown in Figure 6, no significant changes in tensile stress-strain curves was observed for either type of composite irradiated in identical conditions. In addition, it is previously pointed out by Droillard *et al.* that energy release rate can be strongly influenced by interfacial sliding stress (and hence matrix crack density) from the viewpoint of frontal process zone size [7]. Since sliding stress was not directly measured, it was estimated by the interfacial sliding stress parameter at around same matrix damage parameter proposed by Katoh *et al.* [23] for qualitative comparison. From the result in Figure 6, almost no change or the tendency of slight increasing "effective" sliding stress was also estimated (except NITE 1300°C, 5.8 dpa for which evaluation is underway). Hence, it is concluded that the effect of neutron irradiation on fracture resistance of the SiC/SiC composites is not significant for the conditions studied.

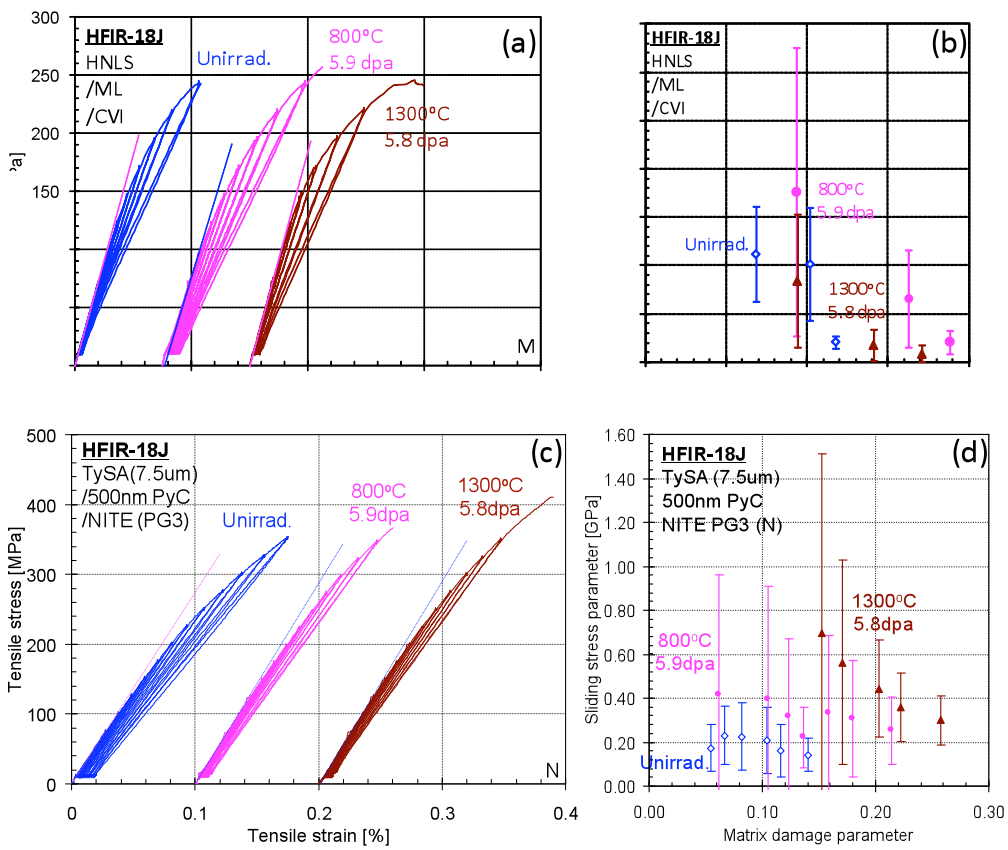

Figure 6. Tensile stress-strain curves for (a) HNLS-CVI-ML [16] and (c) TySA-NITE composite [22], and interfacial sliding stress parameter for (b) HNLS-CVI-ML and (d) TySA-NITE composite.

Conclusions

The effect of neutron irradiation on damage tolerance of the nuclear grade SiC/SiC composites was evaluated by means of miniaturized single-edged notched-beam test. No significant changes in crack extension behavior and the load-loadpoint displacement characteristics such as the peak load and hysteresis loop width were observed after irradiation to 5.9×10^{25} n/m^2 (E > 0.1 MeV) at 800°C and to 5.8×10^{25} n/m^2 at 1300°C. The global energy balance analysis based on non-linear fracture mechanics estimated the energy release rate contributed by macro-crack initiation to be 3±2 kJ/m^2 for both the unirradiated and irradiated composites. According to this analytical result, it was included that the tensile properties, and the hysteresis loop analysis of the tensile tests, the effects of neutron irradiation in conditions studied on fracture resistance of these composites are insignificant.

Acknowledgement

The authors would like to thank Ms. A.M. Williams, Ms. P.S. Tedder, Mr. L.E. Reid, Ms. S.G. Beck and Ms. B.D. Johnson for the post-irradiation experiments. Irradiation was carried out in the Department of Energy High Flux Isotope Reactor User Facility. This research was sponsored by the Office of Fusion Energy Sciences, US Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC. This study was also a part of TITAN US Department of Energy/Japanese Ministry of Education, Culture, Sports, Science and Technology (MEXT) collaboration for fusion material system research.

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