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Time-dependent failure mechanisms in silicon carbide composites for fusion energy applications

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Abstract

Silicon carbide has many properties that are attractive for applications in fusion energy systems. The reliability of monolithic silicon carbide, however, is insufficient for its use in large components. Ceramic matrix composites offer greater flaw tolerance and reliability, but their failure mechanisms are less well understood. This work has focussed on studying potential failure mechanisms in silicon carbide fiber-reinforced, silicon carbide matrix (SiC_f/SiC_m) composites.

In the event of pre-existing cracks, subcritical crack-growth may occur due to creep of fibers that bridge the crack faces. Irradiation-enhanced creep will enhance the subcritical crack-growth rate. The presence of oxygen leads to oxidation of the interphase material and subcritical crack-growth controlled by the rate of interphase recession. In addition, fiber shrinkage or weakening due to exposure to radiation can promote additional failure mechanisms, including embrittlement. These mechanisms, the conditions, under which they occur, and the current state of models of the crack-growth mechanisms will be discussed.

1. Introduction

SiC_f/SiC_m composites are candidate materials for high-temperature applications in fusion energy systems because they exhibit desirable thermal, mechanical, and nuclear stability [1–4]. In inert environments, CMCs exhibit greater mechanical reliability than conventional monolithic ceramics [5] that suggests that they can be used safely in demanding environments, such as fusion energy systems. Non-oxide CMCs rely on a material located between the fibers and the matrix, referred to as the interphase, that causes debonding between fibers and the matrix during crack propagation and subsequent toughening due to fibers that apply bridging stresses in the crack wake [6,7]. At high temperatures, bridging fibers can creep and cause subcritical crack-growth [8,9], in the presence of even small amounts of oxygen the interphase can oxidize and also cause subcritical crack-growth [10,11]. Irradiation can embrittle fibers, cause shrinkage, and accelerate creep, all of which would accelerate the rate of subcritical crack-growth. Subcritical crack-growth mechanisms in SiC_f/SiC_m lead to time-dependent failure. In this paper, mechanisms of time-dependent failure and the conditions under which they occur will be described.

2. Fiber relaxation mechanism

CMCs, such as SiC_f/SiC_m, offer potential for use in high-temperature structural applications due to their non-catastrophic failure modes. In order to obtain desirable mechanical properties it is well known that debonding at the interface between the fiber and matrix must occur during crack propagation [6]. If the conditions for debonding are satisfied, fibers bridge the crack faces in the wake of the crack tip, subsequent to matrix cracking (Fig. 1). The stress borne by the bridging fibers applies traction forces to the crack faces that reduce the stress intensity at the crack tip. Under specific conditions crack propagation does not occur without additional applied stress.

Fibers that bridge a matrix crack (cf. Fig. 1) are subject to a bridging stress, σ_b, determined by the

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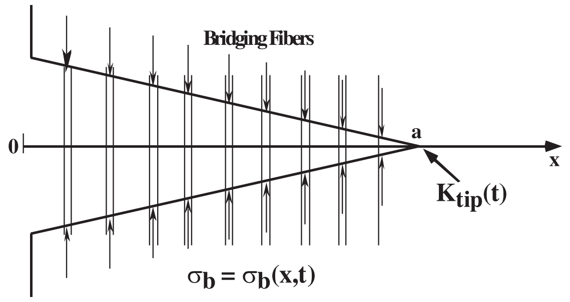

Fig. 1. A schematic of a bridged matrix crack of length a in a composite showing bridging fibers and crack coordinate system.

frictional sliding resistance, τ_0, and the elastic stretching along the unbonded length of the fiber, l_{free}, according to

$$\sigma_{\mathrm{b}}(x)=\left(\frac{4\tau_0 E_{\mathrm{f}}u(x)}{r_{\mathrm{f}}}\right)^{1/2}+\left(\frac{E_{\mathrm{f}}}{l_{\mathrm{free}}}u(x)\right), \quad (1)$$

where r_{f} is the diameter of the fibers, E_{f} the modulus of the bridging fiber, and $u(x)$ is the crack opening displacement. For a cracked specimen it is convenient to write the crack-tip stress intensity as a sum of the K-field from stresses due to remote loads, K_{a}, and the K-field from the bridging stresses, K_{b}.

$$K_{\mathrm{tip}}=K_{\mathrm{a}}+K_{\mathrm{b}} \quad (2)$$

where

$$K_{\mathrm{a}}=2\int_0^a G(x,a)\sigma_{\mathrm{a}}(x)\,\mathrm{d}x \quad (3)$$

and

$$K_{\mathrm{b}}=-2\int_0^a G(x,a)\sigma_{\mathrm{b}}(x)\,\mathrm{d}x. \quad (4)$$

The function $G(x, a)$ is the weight function specific for the cracked specimen geometry and σ_{a} is the stress due to the remote loading. The bridging stresses exert tractions on the crack faces and decrease the crack tip stress intensity.

At elevated temperatures, fibers subject to stress will undergo creep deformation. Creep causes the stress in the fibers to relax and, hence, the bridging traction forces, or σ_{b}, will decrease. It is assumed that the unbonded length of the fiber does not change during creep. This assumption is reasonable since additional stress would be required to initiate further debonding, but the stress on the fibers decreases as a result of creep. As σ_{b} decreases, K_{b} increases according to Eq. (4) and K_{tip} increases (K_{b} becomes less negative). Nicalon fibers have been shown to undergo non-linear creep of the form [12]

$$\dot{\varepsilon}=A_0\sigma^{1.2}t^{0.4}\exp(-Q/RT), \quad (5)$$

where t is time and Q is an activation energy for creep. If sufficient creep occurs, the stress intensity at the crack tip will exceed that required to cause matrix cracking and crack propagation will occur (Fig. 2). If crack propagation occurs, however, additional bridging fibers enter the crack wake and they impose additional bridging traction forces that inhibit crack-growth. Under the conditions of power-law creep, subcritical crack-growth in this manner occurs with a continuously decreasing crack velocity (Fig. 2). This mechanism of crack-growth is referred to as the fiber relaxation mechanism (FRM).

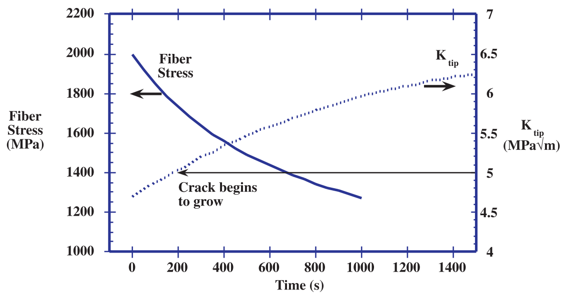

Fig. 2. Computed stress relaxation of a Nicalon fiber at 1373 K from an initial load of 2 GPa using non-linear creep equation (Eq. (5)) shown together with the computed rise in K_{tip} for a bridged crack initially containing 10 bridging fibers. K_{tip} increases from a value of 4.6 MPa$\sqrt{\mathrm{m}}$ to 6.0 MPa$\sqrt{\mathrm{m}}$ over 1000 s. A matrix material with a critical-K of 5 MPa$\sqrt{\mathrm{m}}$ would transition from a stable crack to a propagating crack during this time.

Predicted crack velocities from a micromechanical model describing this behavior agree well with experimental measurement [8]. In addition, the activation energy for crack-growth in argon [9] matches that reported for fiber creep [12]. Therefore, at elevated temperatures, under stress, in inert environments, the time-dependent failure of SiC_f/SiC_m is likely to be controlled by fiber creep.

3. Interface recession mechanism

Infiltration of oxygen into cracked SiC_f/SiC_m composites can lead to additional time-dependent failure mechanisms. Oxidation of SiC_f/SiC composite with a carbon interphase can result in both removal of the interphase and formation of SiO_2 from reaction with the fiber or the matrix. The temperature dependence of subcritical crack-growth in the presence of oxygen (47 kJ/mol) [10] is similar to that measured for weight loss in TGA experiments (50 kJ/mol) [13]. Subsequent to these weight loss experiments, analysis of the microstructure revealed that the carbon interphase between the fibers and matrix was progressively removed from surfaces or cracks that were exposed to oxygen. These results suggest that the observed subcritical crack-growth in oxygen is controlled by interphase oxidation. Although subcritical crack-growth occurred in specimens tested at 1100°C in 2000 ppm O_2, the residual toughness was not affected by this type of crack-growth [10]. In addition, the residual toughness was not significantly different when measured at 800°C or 1100°C. On the other hand, the residual toughness measured after a severe exposure to 500,000 ppm O_2 at 800°C was significantly lower than the intrinsic toughness of the material. These results suggest that the fiber strength, which defines the peak load that the specimen can support, is not strongly affected by exposure to low oxygen concentrations but it is degraded by exposure to high oxygen concentrations.

At relatively low temperatures, low oxygen concentrations (such as those anticipated in fusion energy systems), and short times, oxidation of the carbon interphase occurs faster than growth of the oxidation reaction product on the fibers and matrix of SiC_f/SiC_m. Oxidation of carbon yields gaseous products leaving a gap between the fibers and matrix. Single fiber push-in experiments indicate that, for a given applied load, fibers with partially oxidized interfaces appeared more compliant (due to a longer gauge length) than those with unoxidized interfaces [14]. The compliance is an inverse function of the stiffness, or elastic moduli. Furthermore, as oxidation progresses, the apparent compliance of the fibers increases. Intuitively, it is clear that a material with bridging fibers that are more compliant will have a lower resistance to crack-growth than one with stiffer bridging fibers.

In reality, interphase recession increases the free length of the bridging fibers, which makes them appear more compliant. During push-in testing, fibers that have undergone interphase recession will exhibit a greater displacement for a given applied load, relative to fibers that have not undergone interphase recession, because the frictional sliding resistance fiber is lower and the fibers behave more like an elastic spring in compression. Likewise, after interphase recession, fibers that bridge matrix cracks will exert lower bridging tractions, as expressed by Eq. (1), than those that have not undergone interphase recession. The net result, as inferred from experimental observations, is that the K-field from the bridging stresses, K_b increases (Eq. (4)) and the stress intensity at the crack tip increases (Eq. (2)). Therefore, the stress intensity at the crack tip increases leading to the possibility of subcritical crack-growth. This mechanism is referred to as the interphase recession mechanism (IRM). The crack-growth rate can be calculated from the relationship between the crack opening displacement and time-dependent interphase recession distance due to oxidation. Accurate prediction of subcritical crack-growth rates due to interphase recession requires incorporation of interphase recession kinetics (considering transport phenomena) as a function of position and time in a matrix crack, the relationship between crack opening displacements and the interphase recession distance, and micromechanical modeling to solve for the distribution of bridging stresses and resultant crack tip stress intensity. Interphase recession is expected to control time-dependent failure in SiC_f/SiC_m at intermediate temperatures and oxygen concentrations, which may exist in fusion energy systems.

4. Oxidation embrittlement mechanism

If interphase oxidation occurs, extended oxidation can lead to the formation of brittle, solid reaction product on either the matrix, the fibers, or both. The formation of a brittle oxidation product between a crack-bridging fiber and the matrix prohibits the matrix from sliding along the fiber as the crack opening increases. In this case, as crack opening increases, due to crack propagation, the stress in the fiber rapidly increases until the fiber fails. Another possible result of oxygen ingress after interphase recession is the formation of a brittle oxidation product on fiber surfaces. If cracks occur in this oxidation product they may act as Griffith type flaws and lower the stress required for fiber failure. In either case, fiber failure reduces the crack bridging tractions and causes crack propagation (Eq. (4)); hence the rate of subcritical crack-growth continuously increases as a function of time, eventually leading to catastrophic failure. This mechanism is referred to as the oxidation embrittlement mechanism (OEM). Although the oxygen concentration required to cause OEM is unlikely to occur under normal operating conditions of a fusion energy system, OEM may occur in accident situations or after extremely long times.

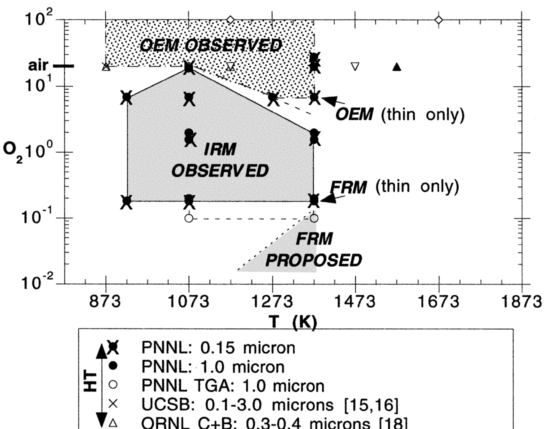

Fig. 3. A failure mechanism map for CVI SiC_f/SiC_m composites.

5. Failure mechanism mapping

Results reported by other investigators [9,13,15–20], for SiC_f/CVI-SiC_m composites reinforced by Ceramic-grade™ Nicalon fibers (manufactured by Nippon Carbon, Japan), were combined with the results of previous studies in our laboratory, using composites reinforced with Hi-Nicalon fibers and a 1-μm-thick carbon interphase, to construct a failure mechanism map (Fig. 3). Although the failure mechanism map that is presented contains data gathered from slightly different materials, the relationships among the boundaries separating distinct failure mechanisms is expected to be similar for other SiC_f/SiC_m composites.

6. Summary and conclusions

SiC_f/SiC_m composites have desirable properties for fusion energy system applications. Several time-dependent subcritical crack-growth mechanisms occur in SiC_f/SiC_m composites: the fiber relaxation mechanism, the interphase recession mechanism, and the oxidation embrittlement mechanism. It is important to understand the physical mechanisms and parameters that control these mechanisms. Experimental data and reports from the literature can be combined to create a failure mechanism map that indicates the conditions under which each failure mechanism operates.

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