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Delayed failure of ceramic matrix composites in tension at elevated temperatures

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Abstract

Ultimate tensile strength of five different continuous fiber-reinforced ceramic matrix composites (CMCs), including SiC_f/BSAS (two dimensional (2D), 2 types), SiC_f/MAS (2D), SiC_f/SiC (2D), and C_f/SiC (2D, 2 types), was determined as a function of test rate at 1100–1200 °C in air. All five CMCs exhibited a significant dependency of ultimate tensile strength on test rate such that the ultimate tensile strength decreased with decreasing test rate. The dependency of ultimate tensile strength on test rate, the applicability of preload technique, and the predictability of life from one loading configuration (constant stress-rate loading) to another (constant stress loading) all suggested that the overall, phenomenological delayed failure of the CMCs would be governed by a power-law type of slow crack growth.

Keywords: Composites; Strength; Stress–rupture testing; Slow crack growth; Lifetime; SiC/SiC

1. Introduction

The successful development and design of continuous fiber-reinforced ceramic matrix composites (CMCs) are dependent on thorough understanding of their basic properties such as deformation, fracture, and delayed failure (slow crack growth, fatigue, or damage evolution/accumulation) behavior. Particularly, complete evaluation and characterization of delayed failure behavior of CMCs under specified loading-environmental conditions is a prerequisite to ensure accurate life prediction of structural components.

In a previous study,[1] ultimate tensile strength of three SiC fiber-reinforced CMCs (SiC_f/CAS, SiC_f/MAS-5, and SiC_f/SiC) at 1100–1200 °C in air was found to be a strong function of test rate. This rate dependency of ultimate tensile strength, in conjunction with the additional results of both accelerated and stress rupture testing, was found to be attributed to a power-law type of slow crack growth or damage evolution/accumulation that described adequately the phenomenological time-dependent behavior of the CMCs.

This paper, as a continuation of the previous study,[1] describes delayed failure behavior of five different fiber-reinforced CMCs at 1100–1200 °C in air, including three SiC fiber-reinforced CMCs (SiC_f/BSAS (2D, 2 types), SiC_f/MAS (1D) and SiC_f/SiC (2D woven)) and one carbon fiber-reinforced CMC (C_f/SiC (2D woven, 2 types)). Ultimate tensile strength of each composite was determined as a function of test rate in constant stress-rate testing and its rate dependency was analyzed with a power-law type of slow crack growth. Preload testing was also carried out to better understand the governing failure mechanism(s) of the composites. Finally, the results of elevated-temperature constant stress ("stress rupture") testing were obtained for SiC_f/BSAS and compared with those of constant stress-rate testing to verify the overall failure mechanism(s) of the composite and to establish constant stress-rate testing as a means of life prediction test methodology for CMCs.

2. Experimental procedure

Five different CMCs—four SiC fiber-reinforced and one carbon fiber-reinforced—were used in this study, includ-

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ing Nicalon™ or Hi-Nicalon™ SiC crossply (2D) fiber-reinforced barium strontium aluminosilicate (designated Nicalon/BSAS and Hi-Nicalon/BSAS), Nicalon™ unidirectionally (1D) reinforced magnesium aluminosilicate (designated SiC_f/MAS), Nicalon™ plain-woven (2D) silicon carbide (designated SiC_f/SiC: "enhanced"), and T-300 carbon fiber-reinforced plain-woven (2D) silicon carbide (designated C_f/SiC: "standard" and "enhanced"). The matrices of the composites, except for C_f/SiC, were reinforced by Nicalon™ or Hi-Nicalon™ fibers with a fiber volume fraction of about 0.40. The unidirectional, crossply or plain-woven laminates of the SiC fiber-reinforced composites were typically 12–18 plies thick with a nominal thickness of around 3–3.5 mm, depending on material. The C_f/SiC composite had a total of 29 plies, a fiber volume fraction of 0.46, and a nominal laminate thickness of 3 mm. The enhanced SiC_f/SiC composite was modified from its standard matrix by a proprietary process to increase the oxidation resistance of the composite. SiC was also chemically vapor deposited on the composite panels to cover the residual porosity. The enhanced C_f/SiC composite had a boron carbide that was introduced into the composite prior to deposition of pyrolytic carbon interface to protect the carbon fibers from oxidation. Both Nicalon/BSAS and Hi-Nicalon/BSAS were fabricated at NASA Glenn Research Center,[2] SiC_f/MAS by Corning, Inc. (Corning, NY),[3] SiC_f/SiC by DuPont Company (Newark, DE),[3] and C_f/SiC by Honeywell Advanced Composites, Inc. (Newark, DE).[4] Detailed information regarding the composites and their processing can be found elsewhere.[2–4]

The dogboned tensile test specimens measuring 152 mm (length) by 12.7 mm (width) were machined from the composite laminates, with the gage section of about 30 mm long, 10 mm wide, and 3.0–3.5 mm thick (as-furnished). The C_f/SiC test specimens were supplied with a notch machined (in depth = 2.5 mm and root radius = 1.2 mm) at one side of gage section at the longitudinal center of each test specimen. After machining, the C_f/SiC test specimens were seal coated with SiC by the chemical vapor infiltration method.[4]

Monotonic tensile testing was conducted in air at 1100 °C for Nicalon/BSAS, Hi-Nicalon/BSAS and SiC_f/MAS-5 and at 1200 °C for SiC_f/SiC and C_f/SiC, using a servohydraulic test frame (Model 8501, Instron, Canton, MA). A schematic test setup is shown in Fig. 1. Each test specimen, located inside of a SiC susceptor via two upper and lower water-cooled hydraulic grips, was induction-heated by radiation through a 15-kW power supply. Two high-temperature extensometers were placed on edges of each test specimen to measure tensile strain. Detailed descriptions on test setup and induction heating equipment were found in a previous study.[3] A total of three different test rates in force control, corresponding to stress rates of 5, 0.16, and 0.005 MPa/s, were employed for a given composite. This test method, when applied to advanced monolithic ceramics, is called constant stress rate or "dynamic fatigue" testing. Typically, one to three test specimens were tested at each test rate for a given composite. Tensile testing was performed in accordance with ASTM Test Standard C 1359.[5]

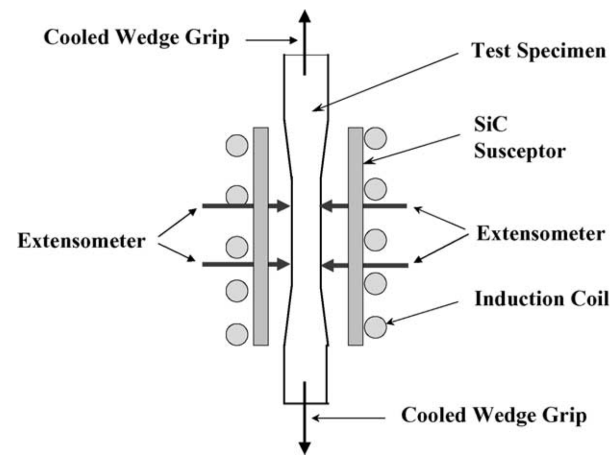

Fig. 1. Schematics of experimental setup used in tensile testing for ceramic matrix composites at elevated temperatures in air.

Preload or accelerated tensile testing, primarily applied to monolithic ceramics in order to save test time,[6] was also conducted at 1100 or 1200 °C using the lowest test rate of 0.005 MPa/s in an attempt to better understand the governing failure mechanism(s) of the composites. A predetermined preload, corresponding to an 80% of ultimate tensile strength of each composite that was determined at 0.005 MPa/s with no preload, was applied quickly (∼100 MPa/s) to the test specimen prior to testing, and monotonic tensile testing at 0.005 MPa/s started and continued until the test specimen failed. The corresponding ultimate tensile strength was determined. One test specimen was used in preload testing for each composite.

Constant stress ("stress rupture") tensile testing was conducted at 1100 °C in air for the Nicalon/BSAS (with two different batches "A" and "B") composite using test specimens with the same geometry and the same test frame and equipment that were employed in constant stress-rate tensile testing. The limited availability of test materials confined the testing to three to four test specimens, depending on batch. Two to three different constant stresses were applied to test specimens and corresponding times to failure were determined.

3. Results and discussion

3.1. Ultimate tensile strength

The results of ultimate tensile strength as a function of test rate determined for the aforementioned CMCs are presented in Fig. 2, where ultimate tensile strength was plotted as a function of applied stress rate using log–log scales. Each solid line in the figure represents the best-fit regression based on the log (ultimate tensile strength) versus log (applied stress rate) relation. The strength data determined

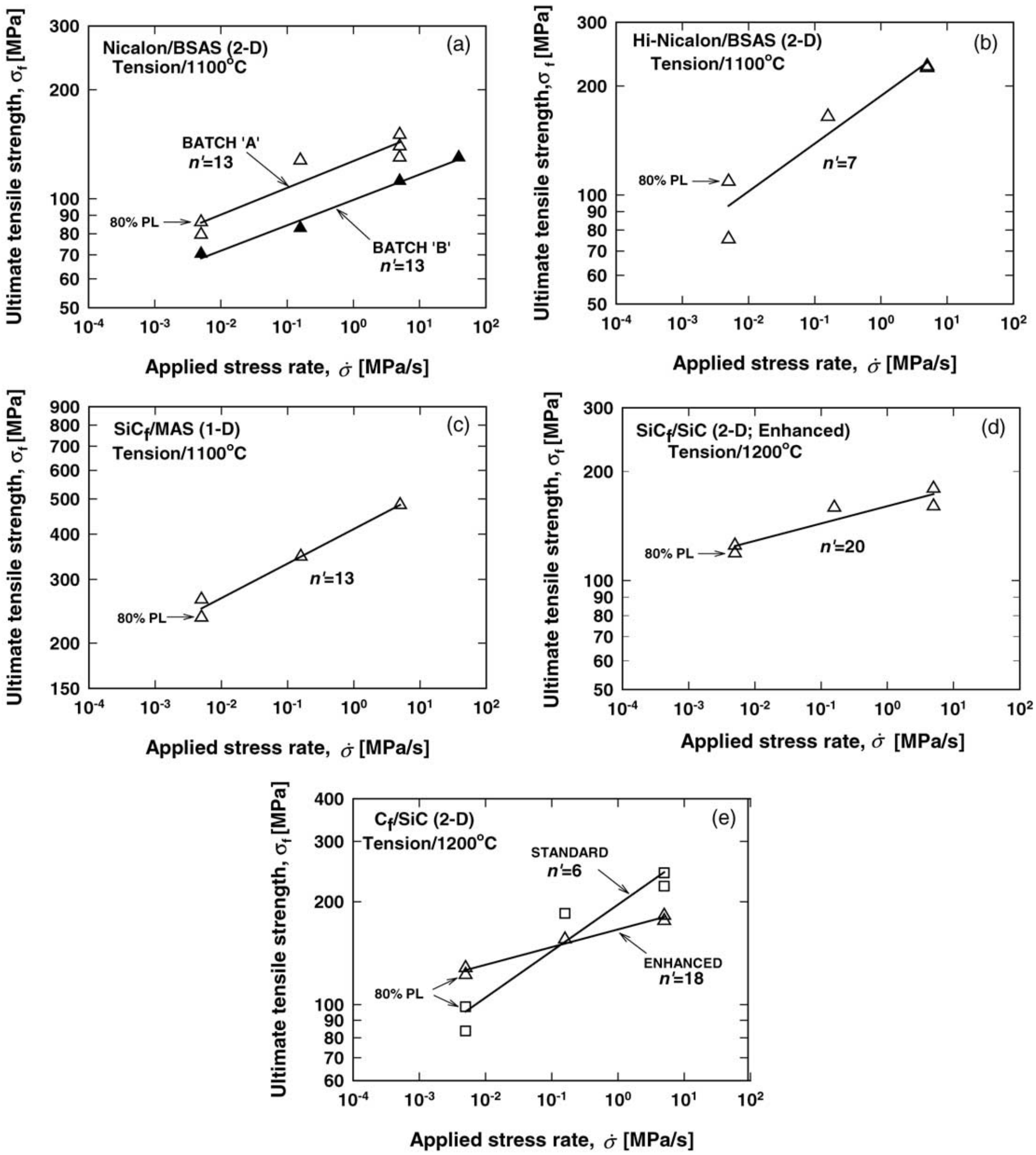

Fig. 2. Results of ultimate tensile strength as a function of applied stress rate determined for (a) Nicalon/BSAS, (b) Hi-Nicalon/BSAS, (c) SiC_f/MAS, (d) SiC_f/SiC, and (e) C_f/SiC ceramic matrix composites at elevated temperatures in air. The solid lines represent the best-fit regression lines based on Eq. (1). The delayed failure (or slow crack growth) parameter n' was also included for each material.

with an 80% preload were also included. The decrease in ultimate tensile strength with decreasing stress rate, which represents a susceptibility to delayed failure (or slow crack growth or damage accumulation), was significant for all the composite materials tested, consistent with the previous results determined with other CMCs such as 1D SiC_f/CAS (calcium aluminosilicocate), 2D SiC_f/MAS-5, and 2D woven SiC_f/SiC (standard).[1] The strength degradation was about 43, 59, 48, and 28%, respectively, for Nicalon/BSAS (batch A), Hi-Nicalon/BSAS, SiC_f/MAS, and SiC_f/SiC, when the stress rate decreased from the highest (=5 MPa/s) to the lowest (=0.005 MPa/s). The corresponding strength degradation for C_f/SiC was 61 and 30%, respectively, for the standard and enhanced composites. It is noteworthy that although Hi-Nicalon/BSAS exhibited higher strength than Nicalon/BSAS at test rates $\geq$0.16 MPa/s, the former exhibited a greater susceptibility to delayed failure than the latter, viewed from the degree of strength degradation with decreasing test rate. Also note that the enhanced C_f/SiC composite showed lower strength at high test rates but greater resistance to delayed failure than the standard counterpart, thus achieving more improved resistance to strength–environmental degradation by the boron carbide enhancement.

Typical examples of fracture pattern of each composite tested at the highest (=5 MPa/s) and lowest (=0.005 MPa/s) test rates are shown in Fig. 3. The mode of fracture for both Nicalon/BSAS and Hi-Nicalon/BSAS composites showed fiber pullout with zigzag matrix cracking through the

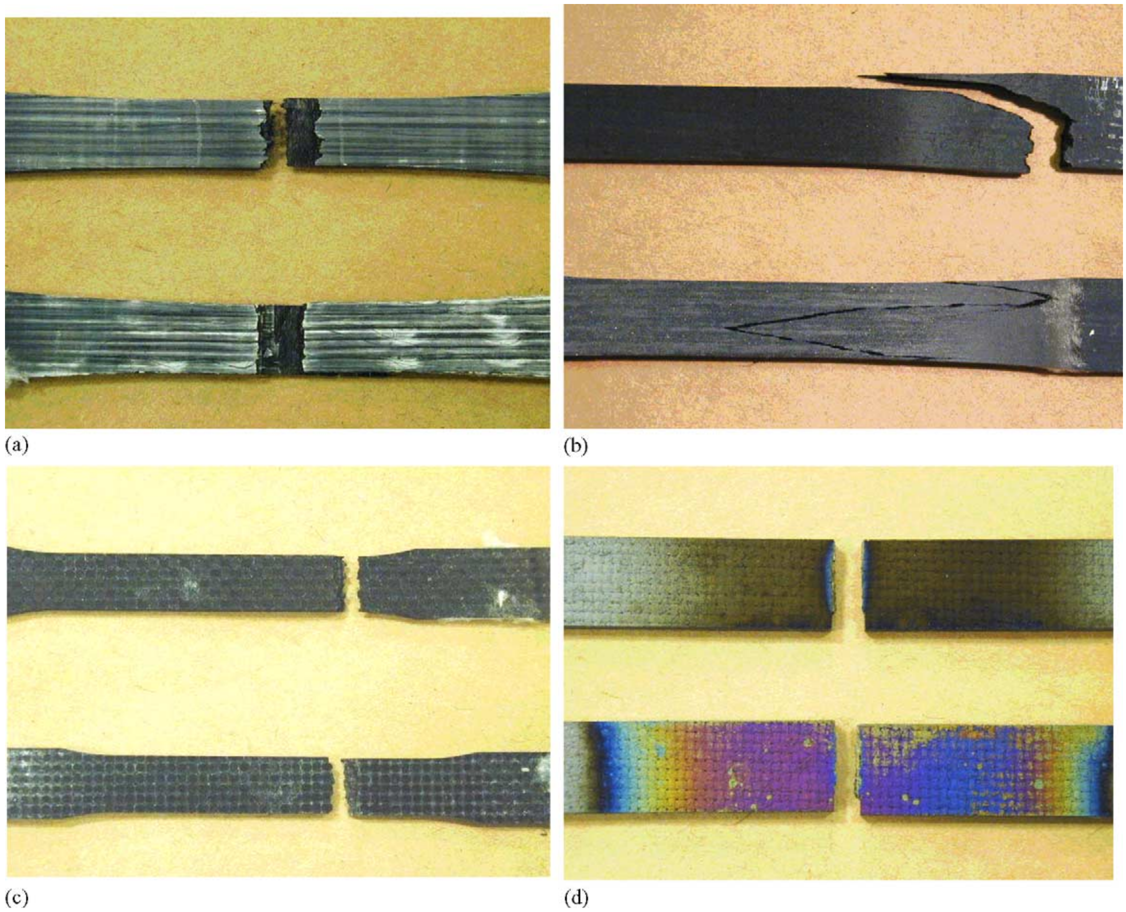

Fig. 3. Fracture patterns for (a) Nicalon/BSAS, (b)SiC_f/MAS, (c) SiC_f/SiC ("enhanced"), and (d) C_f/SiC ("enhanced") ceramic matrix composites tested in tension at elevated temperatures in air. The upper and lower pictures for a given composite material indicate the specimens tested at the lowest (=0.005 MPa/s) and the highest (=5 MPa/s) test rates, respectively.

specimen-thickness direction. A change in surface (matrix) color of test specimens from dark grey to white was more pronounced at 0.005 MPa/s than 5 MPa/s, an evidence of more aggressive high-temperature reaction/oxidation involved at the lower test rate, attributed to increased test time. Fracture patterns for the SiC_f/MAS composite indicated some fiber pullout with jagged faceted matrix cracking often propagating along the test-specimen length. One specimen tested at the fast test rate of 5 MPa/s failed close to the transition and grip regions. No significant difference in the mode of fracture was observed between SiC_f/SiC (enhanced) and C_f/SiC (standard or enhanced), where almost all the specimens tested at either a high or low test rate exhibited relatively flat, straight fracture surfaces with little fiber pullout, termed brittle fracture. A similar brittle mode of fracture was also observed previously for the 2D standard SiC_f/SiC composite.[1] Black to bluish discoloration in the heated region of tested specimens was obvious for either standard or enhanced C_f/SiC, accompanying a weight loss after testing due to oxidation: the more weight loss occurred at the lower test rate, and vice versa. Detailed oxidation and stress rupture/life behaviors of this C_f/SiC composite system have been explored previously in a low partial pressure of oxygen environment.[4,7,8]

The strength dependency on test rate exhibited by these composites at elevated temperatures (see Fig. 2) is very similar to that commonly observed in advanced monolithic ceramics at elevated temperatures. The strength degradation with decreasing stress rate in monolithic ceramics is known to occur by a slow crack growth process (also known as delayed failure, subcritical crack growth or fatigue) and is expressed as follows:[9–11]

$$\log \sigma_{\mathrm{f}} = \frac{1}{n+1} \log \dot{\sigma} + \log D \tag{1}$$

where n and D are slow crack growth parameters, and σ_{f} (MPa) and $\dot{\sigma}$ (MPa/s) are fracture strength and applied stress rate, respectively. Eq. (1) is based on the conventional power-law crack velocity formulation as expressed:

$$v = A\left(\frac{K_{\mathrm{I}}}{K_{\mathrm{Ic}}}\right)^{n} \tag{2}$$

where v, K_{I}, and K_{Ic} are crack velocity, mode I stress intensity factor and fracture toughness, respectively. A is also called slow crack growth parameter. The parameter D is associated with n, A, K_{Ic}, and inert strength of a material. The parameters n and D in Eq. (1) can be obtained by a linear regression analysis from slope and intercept, respectively, when log (fracture strength) is plotted as a function of log (applied stress rate). Constant stress-rate (or called dynamic fatigue) testing based on Eq. (1) has been established as ASTM Test Methods to determine slow crack growth parameters of advanced monolithic ceramics at ambient and elevated temperatures.[10,11]

Notwithstanding the limited number of test specimens used, the data fit to Eq. (1) was very reasonable for the current CMCs with the coefficients of correlation in regression all greater than 0.930. This implies that delayed failure of the composites could be described by the power-law type of slow crack growth formulation, Eq. (2). With this in mind, the apparent delayed–failure parameters n' and D' for the composites were determined based on Eq. (1) using the experimental data shown in Fig. 2 (with the units of σ_f in MPa and $\dot{\sigma}$ in MPa/s). The parameters[1] thus determined were n' = 13 and D' = 127, n' = 7 and D' = 188, n' = 13 and D' = 367, and n' = 20 and D' = 160, respectively, for Nicalon/BSAS (batch A), Hi-Nicalon/BSAS, SiC_f/MAS-5, and SiC_f/SiC. The prime was used here for composites to distinguish them from monolithic ceramic counterparts. The apparent parameters for the C_f/SiC composite were n' = 6 and D' = 196, and n' = 18 and D' = 166, respectively, for the standard and enhanced versions. The value of n' represents a measure of susceptibility to delayed failure, and is typically categorized in brittle materials such that the susceptibility is very high for $n \leq 20$, intermediate for n = 30–50, and very low for $n > 50$. Hence, the current composites exhibited a significant susceptibility to delayed failure as compared with monolithic counterparts such as silicon nitrides and silicon carbides which are typically in the range of $n > 20$ at temperatures ≥1200 °C.[12] Similar results showing greater susceptibility of CMCs to delayed failure at elevated temperatures were also found from the previous study[1] in 1D SiC_f/CAS, 2D SiC_f/MAS, and 2D woven SiC_f/SiC (standard) composites with n' values ranging from n' = 6–18, as depicted in Fig. 4.

Unlike the other CMCs, the C_f/SiC composite, as mentioned before, was subjected to significant oxidation of carbon fibers, resulting in material loss. Therefore, strength degradation was increased, attributed to decreasing fiber volume fraction and subsequently increasing porosity, as the test rate decreased. The strength degradation due to oxidation for the C_f/SiC composite has been described based on the results of stress rupture through a finite difference model on oxygen concentrations and carbon consumption.[13] Although this stress-oxidation model would give a better physical explanation, the phenomenological power-law formulation used in this study still provides a simple, convenient way to quantify

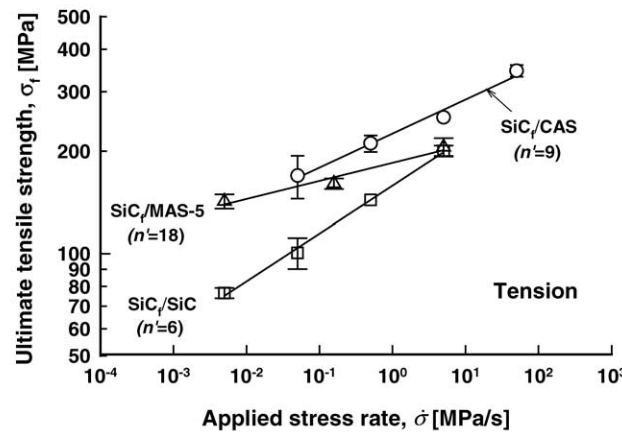

Fig. 4. Ultimate tensile strength as a function of test rates determined for 1D SiC_f/CAS at 1100 °C, 2D SiC_f/MAS-5 at 1100 °C, and woven SiC_f/SiC ("standard") at 1200 °C in air from previous studies.[1]

the degree of strength degradation with respect to test rate. As oxidation prevails into the material system, porosity increases and the effective number and sizes of load-bearing fibers decrease. This oxidation-induced damage would be considered to be equivalent to crack-like flaws growing through matrices and fibers from a fracture-mechanics point of view. The equivalent crack propagates under a driving force (K_I) based on Eq. (2) so that the resulting strength follows in accordance with Eq. (1). Note that oxidation-induced damage increases with decreasing test rate since more time is available for oxidation at lower test rate, and vice versa.[7] In other words, at faster test rates, an equivalent crack has little time to grow, resulting in higher strength; whereas, at lower test rates, the crack has longer time to grow appreciably, thereby yielding lower strength. However, oxidation was not likely a unique source of final fracture responsible in the C_f/SiC material system, as will be discussed in Section 3.2.

3.2. Preload tests results

The results of preload tests carried out at 0.005 MPa/s with an 80% preload are presented in Fig. 5, where ultimate tensile strength was plotted against preload factor (α = 0.8) for each composite. The preload factor α is defined such that a preload tensile stress (σ_p) applied to a test specimen prior to testing is normalized with respect to the ultimate tensile strength (σ_f) with no preload[6,10,11]

$$\alpha = \frac{\sigma_p}{\sigma_f} \tag{3}$$

For advanced monolithic ceramics whose delayed failure is governed by the power-law slow crack growth formulation (Eq. (2)), it has been shown that fracture strength is a function of preload factor and slow crack growth parameter n as follows[6,10,11]

$$\sigma_{fp} = \sigma_f(1 + \alpha^{n+1})^{1/(n+1)} \tag{4}$$

[1] The number of test specimens used in this study, one to three at each test rate, would be insufficient to determine reliable delayed failure parameters. However, considering a relatively small scatter in ultimate strength of many CMCs, typically with coefficients of variation ≤5%, the current delayed-failure parameters provided will not be changed too much even with a large number of test specimens; hence, the variation of the n' and D' to the number of test specimen would be expected to be minimal and statistically insignificant as well.

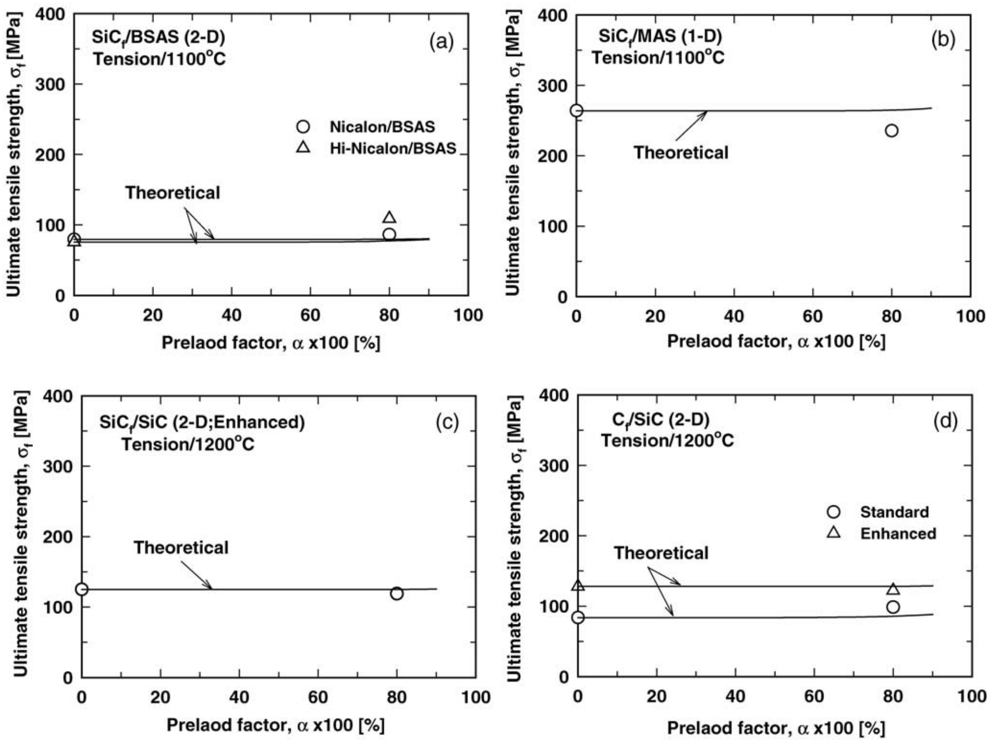

Fig. 5. Results of preload tests, plotted with ultimate tensile strength as a function of preload factor for (a) Nicalon/BSAS and Hi-Nicalon/BSAS, (b) SiC_f/MAS, (c) SiC_f/SiC ("enhanced"), and (d) C_f/SiC ("standard" and "enhanced") ceramic matrix composites at elevated temperatures in air. A theoretical prediction based on Eq. (4) is included for comparison for each composite material.

where σ_{fp} is ultimate tensile strength with a preload and α is in the range of $0 \leq \alpha < 1$. It is noted from Eq. (4) that ultimate tensile strength under preload is more sensitive to higher preload factor α and lower n' value, because of much augmented delayed failure occurring under these conditions.

Each solid line in Fig. 5 indicates the theoretical prediction based on Eq. (4), together with the estimated value of n' and the ultimate tensile strength with no preload. The prediction, despite a limited number of test specimens used, is in good agreement with the experimental data except for the SiC_f/MAS composite. Note that Eq. (4) was derived based on the power-law, slow crack growth formulation, Eq. (2). Therefore, the reasonable applicability of the preload analysis to the current composites suggests that delayed failure process of these composites would be the one governed by the power-law type of slow crack growth, as expressed in Eq. (2). This is consistent with the observations of the previous preload studies using other CMCs.[1] It is also noted from the figure that the overall difference in ultimate tensile strength between two preloads (α = 0 and 80%) was insignificant, resulting in a considerable saving (~80%) of test time by applying the 80% preload. This indicates that any significant crack growth that would control ultimate tensile strength of a composite did not occur even when the applied stress to test specimen during test reached up to 80% of its fracture stress. Conversely, the crack growth or damage to control final catastrophic failure would have occurred when applied stress or test time was greater than 80% of fracture stress or total test time.

The insignificant difference in ultimate tensile strength of the C_f/SiC composite between two preloads of 0 and 80% is particularly noteworthy. The total test time at no preload was about 5–7 h for the C_f/SiC composite (standard and enhanced), while the respective total test time with an 80% preload was about 1–2 h. Despite this appreciable difference in test time (or exposure time for oxidation) between the two preloads, the resulting strength difference was minimal. This suggests that oxidation would not be a sole source of the composite failure as well as of the rate dependency. A further study using increased number of test specimens would reveal more detail aspects of failure mechanism(s) involved in the C_f/SiC composite. However, it is important to state at this point that apart from detailed understanding of a complex oxidation kinetics associated with the C_f/SiC composite, the composite failure can be described phenomenologically by the simple power-law formulation of Eq. (2), based on the results of both ultimate tensile strength and preload tests.

3.3. Constant stress (stress rupture) tests

A summary of the results of constant stress (stress rupture) testing for the Nicalon/BSAS composite (batches A and B) at 1100 °C is presented in Fig. 6, where time to failure was plotted against applied stress in log–log scales. A decrease in time to failure with increasing applied stress, which represents a

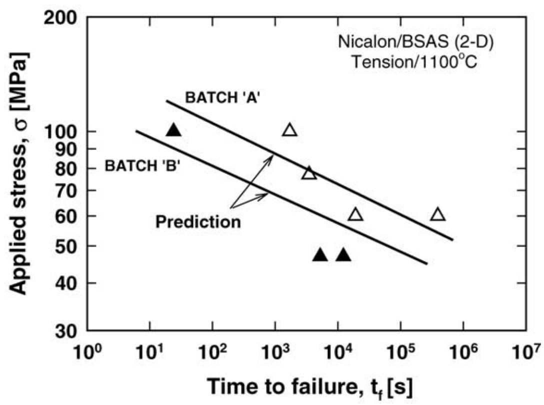

Fig. 6. Results of constant stress ("stress rupture") testing for Nicalon/BSAS composite (batches "A" (open triangle) and "B" (closed triangle)) at 1100 °C in air. The solid lines represent the predictions made based on Eq. (5) from the results of constant stress-rate testing (Fig. 2).

susceptibility to delayed failure, was evident for the composite. The mode of fracture was similar to that in constant stress-rate testing showing some fiber pullout with jagged matrix cracking through the specimen-thickness direction. At lower applied stresses, however, the fracture surfaces of both batches were somewhat flat with decreased fiber pullout, a change in fracture mode more likely to brittle fracture. The lines in Fig. 6 indicate life prediction from the constant stress-rate data. The prediction, primarily applied to brittle monolithic materials, was made using the following relation based on the power-law formulation of Eq. (2).[1]

$$t_f = \left(\frac{D'^{n'+1}}{n'+1}\right)\sigma^{-n'} \tag{5}$$

where t_f and σ are time to failure and applied constant stress, respectively. Use of Eq. (5) together with n' and D' determined in constant stress-rate testing allows one to predict life under constant stress (stress rupture) loading. Even though the small number of test specimens was used here due to limited material availability, the prediction was in reasonable agreement with experimental data at least for the Nicalon/BSAS composite. This indicates that the governing failure law of the Nicalon/BSAS composite was very similar either in constant stress-rate or in constant stress loading. Since the prediction (Eq. (5)) was made based on Eq. (2), the governing delayed–failure mechanism of the Nicalon/BSAS would be the one controlled by the power-law type of slow crack growth (Eq. (2)). Other CMCs also showed reasonable agreement in life between constant stress-rate and constant-stress loading configurations.[1]

3.4. Implications

As seen in the preceding Sections 3.1–3.3, the strength dependency on test rate, the applicability of the preload technique, and the reasonable life prediction from one loading configuration (constant stress-rate testing) to another (constant stress) for a selected composite all support that delayed failure of the composites was controlled by the power-law type of slow crack growth (or damage evolution/accumulation), confirmed with not only the current CMCs but the previous CMCs.[1] It is noted that despite many differences in their processing, architecture, microstructure, and interface, compared with monolithic ceramics, CMCs still exhibit delayed failure, similar in principle to monolithic counterparts. This is simply due to the fact that macroscopically, a CMC is a composite composed of two or more delayed–failure susceptible monolithic materials (constituents of fibers, matrices, interfaces, etc.). The vulnerability to delayed failure would be greater in composite because of its more likelihood of chance to environmental exposure by its inherently more open, porous microstructures, as compared to dense monolithic counterparts. The vulnerability of a composite, of course, will be increased if the constituents added are highly susceptible to delayed failure.

A subsequent importance drawn from the results of this work is that constant stress-rate testing, commonly utilized in monolithic ceramics, could be applicable to CMCs to determine their delayed–failure (or life prediction) parameters, at least for a short range of lifetimes, consistent with the previous observation.[1] The merits of constant stress-rate testing are enormous in terms of test simplicity and test economy (short test time and less data scatter) over other stress rupture or cyclic fatigue testing. A simplistic, phenomenological law of delayed failure was only explored in this study to develop lifetime prediction testing and methodology for CMCs, without accounting for detailed failure mechanisms associated with matrix/fiber interaction, matrix cracking and its effect on slow crack growth, delayed failure of sustaining fibers, and creep-associated deformation.[14–18] A microscopic level of study on this subject is thus needed. Finally, the results of this work suggest that care must be exercised when characterizing elevated-temperature strength of composite materials. This is due to the fact that elevated-temperature strength has a relative meaning if a material exhibits rate dependency: the strength simply depends on which test rate one chooses (see Fig. 2). Therefore, use of at least two test rates (high and low) is recommended to better characterize high-temperature strength behavior of a composite material.

4. Conclusions

Elevated-temperature ultimate tensile strength of five different continuous fiber-reinforced ceramic composites, including Nicalon/BSAS (2D), Hi-Nicalon/BSAS (2D), SiC_f/MAS (1D), SiC_f/SiC (2D woven: enhanced), and C_f/SiC (2D woven: standard and enhanced), exhibited a strong dependency on test rate, consistent with the behavior observed in other CMCs as well as in many advanced monolithic ceramics at elevated temperatures. The rate dependency of ultimate tensile strength, the applicability of the preload technique, and the predictability of life from one loading configuration (constant stress rate) to another (constant stress) for the Nicalon/BSAS composite suggested that the overall failure law of the composites would be governed by a power-law type of slow crack growth (or damage evolution/accumulation). It was further confirmed that constant stress-rate testing could be utilized as a means of life prediction test methodology for composites when short lifetimes are expected and when ultimate tensile strength is used as a failure criterion.

Acknowledgments

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