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EFFECT OF TEST RATE ON TENSILE STRENGTH OF VARIOUS CONTINUOUS FIBER-REINFORCED CERAMIC COMPOSITES AT ELEVATED TEMPERATURES

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ABSTRACT

Ultimate tensile strength of three continuous fiber-reinforced ceramic composites, including SiC/CAS-II, SiC/MAS-5 and SiC/SiC, was determined as a function of test rate in air at 1100 - 1200°C. All three composite materials exhibited a strong dependency of strength on test rate, similar to the behavior observed in many advanced monolithic ceramics at elevated temperatures. Both the applicability of the preloading technique and the excellent data fit to log (*ultimate strength*)-vs-log (*test rate*) relation suggested that the overall macroscopic failure mechanism of the composites would be the one governed by a power-law type of damage evolution/accumulation, analogous to slow crack growth commonly observed in advanced monolithic ceramics.

KEY WORDS

Continuous fiber-reinforced ceramic composites (CFCCs), ultimate tensile strength, elevated-temperature mechanical testing, loading rate dependency of ultimate strength, failure mechanism, preload technique, constant stress-rate testing

INTRODUCTION

The successful development and design of continuous fiber-reinforced ceramic composites (CFCCs) depends on a thorough understanding of basic properties such as fracture and delayed failure (slow crack growth, fatigue, or damage accumulation) behavior. In particular, accurate evaluation of delayed failure behavior under specified loading/environment conditions is a prerequisite to ensue accurate life prediction of structural components.

This paper describes the effect of test (or loading) rate on elevated-temperature ultimate tensile strength of three different Nicalon™ fiber-reinforced ceramic composites such as SiC_f/calcium-aluminosilicate (CAS), SiC_f/magnesium-aluminosilicate (MAS) and SiC_f/silicon-carbide (SiC) ceramic composites. For each composite material, strength was determined in air as a function of test rate at elevated temperature of 1100°C (for SiC/CAS and SiC/MAS) or 1200°C (for SiC/SiC). This type of testing, when used for monolithic ceramics, is called "constant stress-rate" or "dynamic fatigue" testing [1-3]. The loading rate dependency of strength was analyzed with the power-law damage or slow-crack-growth propagation, conventionally utilized for monolithic ceramics and glass. Preloading tests were conducted to better understand the governing failure mechanism(s) of the materials. It should be noted that few studies on the subject of loading rate dependency have been done for continuous fiber-reinforced ceramic composites [4], particularly at elevated temperatures.

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EXPERIMENTAL PROCEDURE

All the matrices of the three test composites were reinforced by ceramic-grade Nicalon™ fibers with a fiber volume fraction of about 0.39. The nominal fiber diameters ranged from 10 to 15 µm. The three composite materials tested included Nicalon™ unidirectionally (1D) fiber-reinforced calcium aluminosilicate (designated SiC/CAS-II), Nicalon™ cross-plied (2D) magnesium aluminosilicate (designated SiC/MAS-5), and Nicalon™ plain-woven (2D) silicon carbide composites (designated SiC/SiC). Both SiC/CAS-II and SiC/MAS-5 were fabricated by Corning, Inc. through hot-pressing followed by ceraming of the composites by a thermal process. The silicon carbide matrix in the SiC/SiC composites was fabricated by the DuPont Company through chemical vapor infiltration (CVI) into the fiber perform. SiC/CAS-II and SiC/MAS-5 laminates were 18 and 16 plies thick, respectively, with a nominal thickness of about 3 mm. The plain-woven laminates of the SiC/SiC composite were supplied 12 plies (normally 3.5 mm thick). More detailed information regarding the test composite materials can be found elsewhere [5]. The SiC/CAS-II material has been used in a previous, preliminary study on test rate-effect on tensile strength [6]. The dogboned tensile test specimens measuring 152.4 mm (length) x 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 design of the dogboned tensile test specimen was the result of previous finite element analysis [7].

Monotonic tensile testing was conducted in air at 1100°C for both SiC/CAS-II and SiC/MAS-5 and at 1200°C for SiC/SiC, using a servohydraulic test frame (Model 8501, Instron, Canton, MA). A total of three to four different loading rates (in load control), corresponding to stress rates ranging within 50-0.005 MPa/s, were employed with typically 3 test specimens tested at each loading rate. Detailed experimental procedure on tensile testing and related induction-heating equipment can be found elsewhere [5]. Preload or accelerated testing technique, applied primarily to monolithic ceramics and glass [8], was also conducted at test temperatures using 0.5 MPa/s (for SiC/CAS-II) or 0.005 MPa/s (for SiC/MAS-5 and SiC/SiC) in an attempt to better understand the governing failure mechanism of the materials. Predetermined preloads, corresponding to about 80 to 90 % of the failure strength at 0.5 MPa/s or 0.005 MPa/s with zero preload (regular testing), were applied quickly to the test specimens prior to testing and their corresponding strengths were measured. Typically two to three test specimens were used in preload testing. Tensile testing was performed in accordance with an ASTM Test Method, ASTM C 1359 [9].

RESULTS

Constant Stress-Rate Testing
Results of monotonic tensile strength testing with different test rates are presented in Figure 1, where log (*ultimate strength*) was plotted as a function of log (*applied stress rate*) for each composite material. Each solid line in the figure indicates a best-fit regression line based on the log (*ultimate strength*) versus log (*applied stress rate*) relation. The decrease in ultimate strength with decreasing stress rate, which represents a susceptibility to damage accumulation or delayed failure, was significant for all the composite materials. The strength degradation was about 51, 31 and 62 %, respectively, for SiC/CAS-II, SiC/MAS-5 and SiC/SiC when stress rate decreased from the highest to the lowest. Fracture patterns for the SiC/CAS-II composite showed some fiber pullout with jagged faceted matrix cracking often propagating along the test-specimen length. For a given stress rate, however, the difference in strength between different fracture patterns was not obvious. No appreciable difference in the mode of failure was observed for SiC/MAS-5 and SiC/SiC, where most specimens tested at either high or low stress rate exhibited relatively flat fracture surfaces – possibly termed *brittle fracture*.

Preload Testing
The results of preload tests are also shown in Figure 1, where the ultimate strength with 80 to 90 % preloads is compared with that in regular testing with zero preload. The difference in strength between two preloads (0 and 80-90 %) was negligibly small for each material: 211 MPa (for 0 % preload) and 209 MPa (for a 85 % preload) for SiC/CAS-II; 142 MPa (0 and 80 % preload) for SiC/MAS-5; 77 MPa (0 %) and 80 MPa (90 %) for SiC/SiC. Hence, the maximum strength difference, exhibited by SiC/SiC, amounts to only about 4 %. This indicates that any significant damage that would control ultimate strength of the material did not occur before the applied loads up to 80 to 90 % of fracture load. Conversely, the damage to control final failure

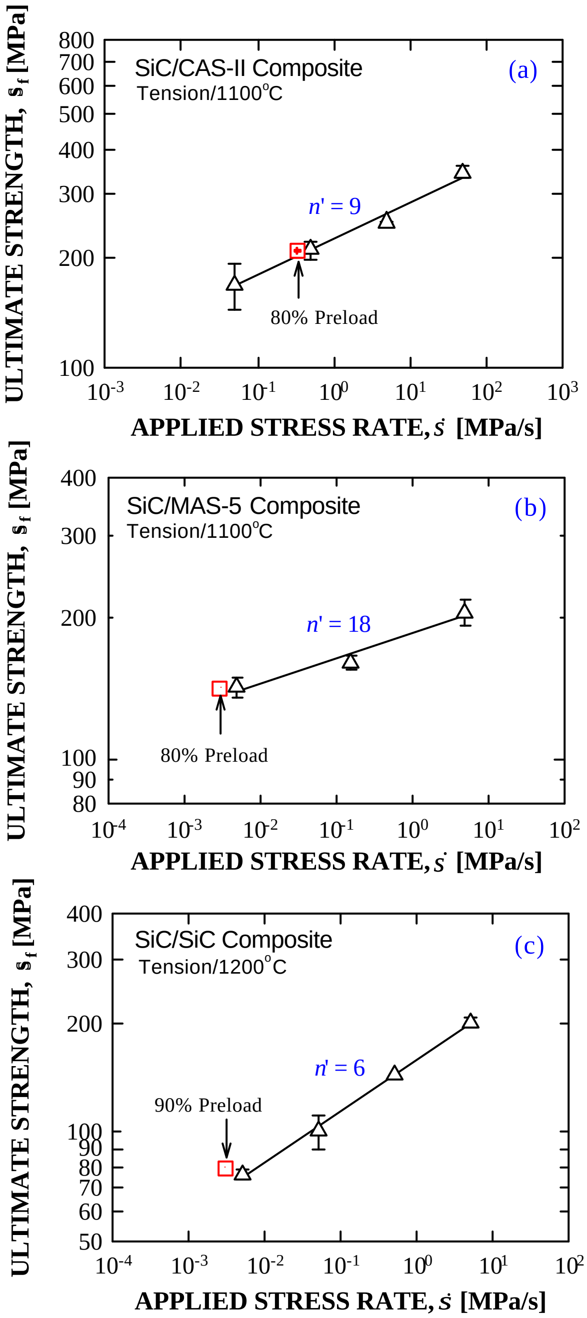

Figure 1. Ultimate tensile strength as a function of applied stress rate for (a) SiC/CAS-II, (b) SiC/MAS-5 and (c) SiC/SiC composites at elevated temperatures in air. The solid lines represent the best-fit regression lines based on Equation 3. Error bar indicates ± 1.0 standard deviation. Ultimate tensile strength with preload is also included for each material for comparison.

would have occurred when applied load or test time was greater than 80 to 90 % of fracture load or total test time. The theory explaining the results of preload testing will be described in the discussion section.

DISCUSSION

The strength dependency on test rate exhibited by the three composite materials (Figure 1) is very similar to that observed in advanced monolithic ceramics at ambient or elevated temperatures. The strength degradation with decreasing stress rate has been known to be due to slow crack growth (delayed failure or fatigue) of an initial crack, typically governed by the following empirical power-law relation [1-3]

$$v = A(K_I / K_{IC})^n \tag{1}$$

where v, K_I and K_{IC} are crack velocity, mode I stress intensity factor and fracture toughness, respectively. A and n are called slow crack growth (SCG) parameters. Based on this power-law relation, the strength (σ_f) can be derived as a function of applied stress rate ($\dot{\sigma}$) [1-3],

$$\sigma_f = D\,[\dot{\sigma}]^{1/(n+1)} \tag{2}$$

where D is another SCG parameter associated with inert strength, n and crack geometry. Equation (2) can be expressed in a more convenient form by taking logarithms of both sides

$$\log \sigma_f = \frac{1}{n+1}\log \dot{\sigma} + \log D \tag{3}$$

Constant stress-rate ("dynamic fatigue") testing based on Equation (2) or (3) has been established as ASTM Test Methods (C1368 [2] and C1465 [3]) to determine SCG parameters of advanced monolithic ceramics at ambient and elevated temperatures. It has been recommended to use units of MPa for σ_f and MPa/s for $\dot{\sigma}$ [2-3]. As can be seen in Figure 1, the data fit to Equation (3) is very reasonable with the coefficients of correlation (r_{coef}) all greater than 0.980, indicating that the damage evolution/accumulation or delayed failure of the composite materials would be adequately described by the power-law type relation, Equation (1). Assuming this, the *apparent* parameters n' and D' for the composites were determined using a linear regression analysis based on Equation (3) with the data in Figure 1. Values of n' = 9.0 and D' = 226, n' = 18 and D' = 185, and n' = 6 and D' = 158 were obtained for SiC/CAS-II, SiC/MAS-5 and SiC/SiC, respectively (The prime was used here for composite materials to distinguish them from monolithic ceramic counterparts.). It is noteworthy that the value of n', a measure of susceptibility to damage, was very low for both SiC/CAS-II and SiC/SiC, but intermediate for SiC/MAS-5. Typical monolithic silicon nitrides and silicon carbides at high temperatures at ≥1200°C exhibit $n \geq 20$. Hence, compared with monolithic ceramics, the SiC/CAS-II and SiC/SiC composites exhibited a significantly higher susceptibility to damage evolution/accumulation.

The preloading or accelerated testing technique has been developed for monolithic ceramics in order to save test time in constant stress-rate testing [8]. Based on the power-law SCG relation of Equation (1) with some mathematical manipulation, strength of a test specimen under a preload (α_p) was derived as a function of preloading factor as follows [8,2,3]:

$$\sigma_{fp} = \sigma_f\,(1+\alpha_p^{\,n+1})^{\frac{1}{n+1}} \tag{4}$$

where σ_{fp} is strength with a preload and α_p ($0 \leq \alpha_p \leq 1$) is a preloading factor (or percentage of preload) in which a preload stress (applied to the test specimen) is normalized with respect to the strength with zero preload. Equation (4) indicates that strength with a preload is sensitive to the magnitude of preload particularly at lower n and higher α_p values. A theoretical prediction of ultimate strength as a function of preload, based on Equation (4) with estimated values of n' from Figure 1, is presented in Figure 2. The prediction is in excellent agreement with the experimental data for all the three composite materials tested, as seen in the figure. This result obtained from the composite materials is also analogous to that observed in advanced monolithic ceramics and glass [8]. Damage, mainly SCG, of monolithic ceramics occurs substantially close to 90 % of total failure time because of their higher n (≥20) value [8]. The applicability of the preloading analysis for the composite materials strongly suggests that major damage evolution/accumulation process would be the one governed by the power-law relation (Equation (1)) and that the damage would have occurred after a long incubation time, at least after 80 % of total test time.

The strength dependency on test rate, the very reasonable data fit to Equation (3) and the applicability of preloading technique all support that the damage evolution/accumulation of the composite materials tested was controlled by a process very similar, in principle, to the power-law type of SCG of monolithic ceramics.

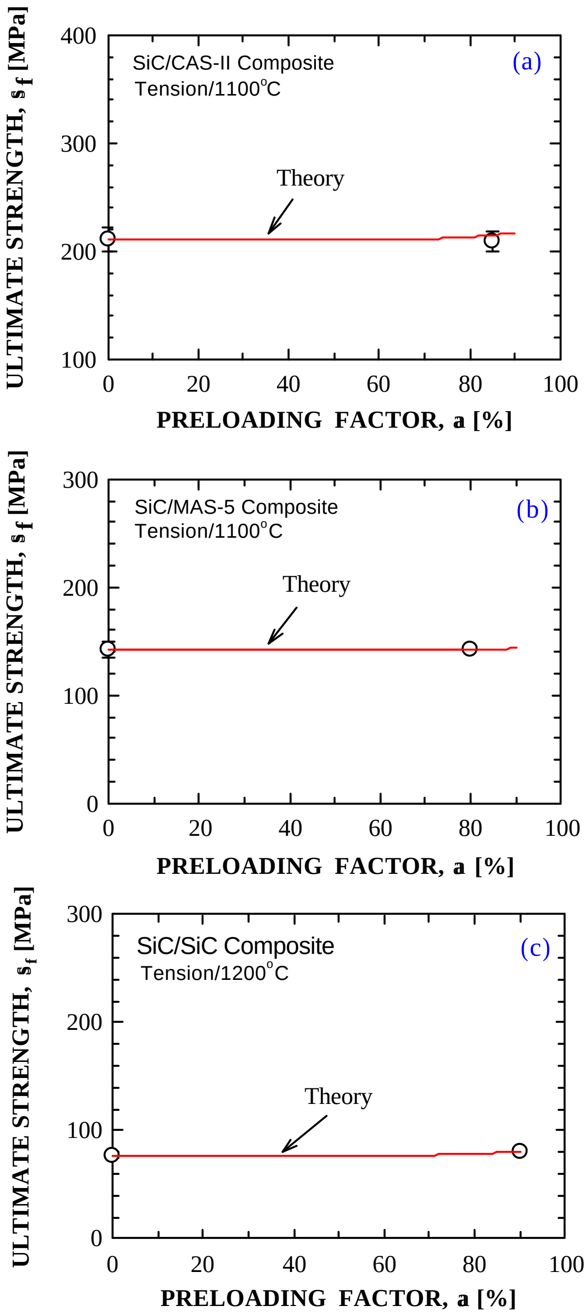

Figure 2. Results of preloading tests (ultimate strength as a function of preloading) for (a) SiC/CAS-II, (b) SiC/MAS-5 and (c) SiC/SiC composites at elevated temperatures in air. A theoretical line based on Equation (4) [8] is included for comparison for each composite material.

The previous results obtained from constant stress ("stress rupture") tensile testing for the same test materials also showed the power-law type of damage evolution/accumulation mechanism [10]. This all indicates that constant stress-rate testing, commonly utilized in determining life prediction parameters of monolithic ceramics, could be applicable even to composite materials as a means of life prediction test methodology. The merit of constant stress rate testing is enormous in terms of simplicity and test economy (shortened test time and less test specimens required) over other stress rupture or cyclic fatigue testing, especially for short lifetimes. A continuing effort to establish a database in elevated-temperature constant stress rate testing is in progress by adding more CFCC materials. At the same time, a more detailed understanding regarding microscopic failure mechanisms [4,11-14] associated with matrix/fiber interaction, matrix cracking and its effect on slow crack growth, and delayed failure of sustaining fibers near catastrophic fracture, etc. is also needed. The results of this work also 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: strength is simply dependent on which test rate one chooses (Figure 1). Therefore, at least two test rates (high and low) are generally recommended to better characterize high-temperature strength behavior of a composite material.

CONCLUSIONS

Elevated-temperature strength of three continuous fiber-reinforced ceramic composites, including SiC/CAS-II, SiC/MAS-5 and SiC/SiC, exhibited a strong dependency on test rate, similar to the behavior observed in many advanced monolithic ceramics at elevated temperatures. The applicability of the preloading technique as well as the reasonable data fit to log (*ultimate strength*)-vs-log (*test rate*) relation suggested that the distinct, overall failure mechanism of the composite materials would be a process primarily governed by a power-law type of damage evolution/accumulation, analogous to the mechanism observed in monolithic counterparts.

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