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Load-rate dependency of ultimate tensile strength in ceramic matrix composites at elevated temperatures

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Abstract

Ultimate tensile strength of three continuous fiber-reinforced ceramic matrix composites, including SiC_t/CAS-II (1D), SiC_t/MAS-5 (2D) and SiC_f/SiC (2D), was determined as a function of load (test) rate in air at $1100-1200$ °C. All three composites exhibited a significant dependency of ultimate tensile strength on test rate. The application of the preload technique as well as the prediction of life from one loading configuration (constant stress-rate) to another (constant stress) suggested that the overall phenomenological macroscopic failure mechanism of the composites would be a power-law type of slow crack growth or damage evolution/accumulation. It was further found that constant stress-rate testing could be used as an alternative to life prediction test methodology for ceramic matrix composites, at least for a short range of lifetimes and when ultimate tensile strength is used as a failure criterion.

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Keywords: Continuous fiber-reinforced ceramic matrix composites; Tensile strength; Stress rupture; Slow crack growth

1. Introduction

The successful development and design of continuous fiber-reinforced ceramic matrix composites (CFCCs) depend on thorough understanding of their basic properties such as deformation, fracture and delayed failure (slow crack growth, fatigue, or damage accumulation) behavior. Particularly, accurate evaluation of delayed failure behavior of those materials under specified loading-environment conditions is very important to ensure accurate life prediction of structural components.

This paper describes the effect of load rate on elevated-temperature ultimate tensile strength of three different Nicalon^{M} SiC fiber-reinforced ceramic matrix composites including $SiC_f/calcium-aluminosilicate (CAS),$ $SiC_f/magnesium-aluminosilicate$ (MAS) and $SiC_f/silicon$ carbide (SiC) ceramic composites. Ultimate tensile strength of each composite was determined in air as a function of test rate at 1100 or 1200 \degree C. This type of testing, when used for monolithic ceramics, is called 'constant

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stress-rate' or 'dynamic fatigue' testing [\[1–3\]](#page--1-0). The load-rate dependency of ultimate tensile strength exhibited by these composites was analyzed with a phenomenological, powerlaw slow crack growth. Preload tests were conducted to better understand the governing failure mechanism(s) of the materials. Finally, elevated-temperature constant stress ('static fatigue' or 'stress rupture') testing was conducted for each composite and their results were compared with those of constant stress-rate testing. This was done to further validate the overall governing phenomenological failure mechanism of the composites and to establish constant stress-rate testing as a means of life prediction test methodology for CFCCs. It should be noted that few studies on these subjects of rate dependency and its association with life have been done for CFCCs at elevated temperatures, although some limited rate-dependency data exist for a composite at ambient temperature [\[4\].](#page--1-0)

2. Experimental procedure

All the matrices of three ceramic matrix composites were reinforced by ceramic-grade Nicalon^{m} SiC fibers with a fiber volume fraction of about 0.39. The nominal fiber

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diameters ranged from 10 to 15 μ m. The three composites included Nicalon^{m} unidirectionally (1D) fiber-reinforced calcium aluminosilicate (designated $SiC_f/CAS-II$), Nicalon^{M} crossply (2D) magnesium aluminosilicate (designated $SiC_f/MAS-5$), and NicalonTM plain-woven (2D) silicon carbide (designated SiC_f/SiC) composites. $SiC_f/CAS-II$ and $SiC_f/MAS-5$ were fabricated by Corning, Inc. through hot-pressing followed by ceraming of the composites by a thermal process. The designation -5 in $SiC_f/MAS - 5$ indicates that the matrix was doped with 5% volume fraction of borosilicate glass. The silicon carbide matrix in the SiC_f/SiC composite was fabricated by the DuPont Company through chemical vapor infiltration (CVI) into the fiber preform. SiC_f/CAS -II and SiC/MAS -5 laminates were 18 and 16 plies thick, respectively, with a nominal thickness of 3.2–3.4 mm. The plain-woven laminates of the SiC_f/SiC composite were 12 plies with a nominal thickness of 3.5 mm. More detailed information regarding the test composites can be found elsewhere [\[5\]](#page--1-0). The $SiC_f/CAS-II$ composite has been used in a previous preliminary study of test rate-effect on ultimate tensile strength [\[6\].](#page--1-0) The dogboned tensile test specimens measuring 152.4 mm (length; '1-1' direction) \times 12.7 mm (width) were machined from the composite laminates with the gage section of about 30 mm long, 10 mm wide and 3.2–3.5 mm thick (asfurnished). The longitudinal direction of the SiC/CAS-II test specimens was aligned with the fiber direction. The dogboned tensile test specimen was designed based on the results of finite element analysis [\[7\].](#page--1-0)

Monotonic tensile testing was conducted in air at 1100 °C for both SiC_f/CAS-II and SiC_f/MAS-5 and at 1200 °C for SiC $_f$ /SiC using a servohydraulic test frame (Model 8501, Instron, Canton, MA) together with an experimental setup shown in Fig. 1. Detailed experimental procedure and related induction-heating equipment can be found elsewhere [\[5\]](#page--1-0). A total of three to four different loading rates (in load control), corresponding to stress rates ranging within 50–0.05 or 5–0.005 MPa/s,

were employed with typically three test specimens tested at each loading rate. Preload or accelerated testing, applied primarily to monolithic ceramics and glasses [\[8\]](#page--1-0), was also conducted at test temperatures using 0.5 MPa/s for $SiC_f/CAS-II$ or 0.005 MPa/s for $SiC_f/MAS-5$ and SiC_f/SiC in an attempt to better understand the governing failure mechanism of the composites. Predetermined preloads, corresponding to about 80–90% of the failure strength determined at 0.5 or 0.005 MPa/s with no 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 ASTM Test Standard, ASTM C 1359 [\[9\].](#page--1-0) Each test specimen was held for 20 min at test temperature prior to testing for thermal equilibration.

Constant stress (static fatigue or stress rupture) tensile testing was also carried out in air for the three composites using the same specimen geometry, test fixture, test frame, thermal equilibration, and same test temperatures that were used in constant stress-rate tensile testing. Due to the limited availability of test materials, the number of test specimens was confined to four to nine for a given composite. A total of four to five different constant stresses were applied to test specimens, and their corresponding times to failure were determined.

3. Experimental results

3.1. Constant stress-rate tensile testing

The results of monotonic tensile strength testing with different test rates are presented in [Fig. 2,](#page--1-0) where ultimate tensile strength was plotted as a function of applied stress rate using log–log scales for each composite material. Each solid line in the figure indicates the best-fit regression based on the log (ultimate tensile strength) versus log (applied stress rate) relation. The decrease in ultimate tensile strength with decreasing stress rate, which represents a susceptibility to slow crack growth or delayed failure, was significant for all the composite materials. The strength degradation was about 51, 31 and 62%, respectively, for $SiC_f/CAS-II$, $SiC_f/$ MAS-5 and SiC_f/SiC when stress rate decreased from the highest to the lowest.

Fracture patterns for the $SiC_f/CAS-II$ composite showed significant fiber pullout with jagged faceted matrix cracking often propagating along the test-specimen length, as shown in [Fig. 3](#page--1-0). However, this feature was gradually diminished at lower stress rates of 0.5 and 0.05 MPa/s, resulting in less fiber pullout with increased embrittled region in the outside of specimens. The difference in strength between different fracture patterns was not obvious for either the SiC_f/SiC composite or the Fig. 1. Schematics of the experimental setup used in tensile testing. $SiC_f/MAS-5$. The $SiC_f/MAS-5$ composite exhibited much

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