Time Dependent Mechanical Properties of ZrB₂-SiC Ceramic Composites: Room Temperature Fatigue Parameters

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ABSTRACT

Room temperature fatigue of ZrB₂-30 wt% SiC ceramic composite has been studied. The linear regression technique, proposed by Fett and Munz, was used to determine the static and cyclic fatigue parameters of ZrB₂-30 wt% SiC ceramic composite tested in dry air, air with 40% humidity, and water at room temperature. It was found that cracks grow much faster under cyclic test conditions in comparison with constant static loading. This phenomenon can be attributed to (1) the degradation of bridging interactions between the two growing crack surfaces; and (2) a corresponding decrease in shielding stress intensity factor. When cyclic fatigue experiments were performed in water in comparison with the results obtained in air, it was also found that crack grows much faster under 100% of humidity. It was determined that water molecules increase the crack propagation rate in ZrB₂-30 wt% SiC composite. The lifetime of the material decreases from 18 hours at $\sigma = 580$ MPa static loading in air to 5.5 minutes at $\Delta\sigma = 1200$ MPa cyclic loading in air and 0.4 seconds at $\Delta\sigma = 1240$ MPa cyclic loading in water.

KEYWORDS: Static Fatigue, Cyclic Fatigue, Mechanical Behavior, ZrB₂-30 wt% SiC Ceramic Composite, Strength.

1. INTRODUCTION

Delayed failure is an important phenomenon which determines the service lifetime of brittle material. It requires significant attention in order to predict and even extend the service life of the components.^{1–4} The delayed failure occurs well below critical stress values required for instantaneous and catastrophic failure of the brittle material. The magnitude of applied stress significantly affects the material's usable lifetime. At high applied stress of 90–95% of the material's strength, failure occurs in a matter of minutes or even seconds. However, when the stress load is reduced, the time to failure extends to hours, days, and even years. It is assumed that for some materials at a very low threshold of applied stress, an endurance limit could be exhibited.⁵ Such time dependent degradation of mechanical properties, up to a complete failure, is termed "fatigue." Degradation is subject to both the crack/loading

type and the material's properties. For example, it was recognized that a delayed failure in glass and ceramics is caused by the growth of surface cracks subjected to tensile stresses.^{6–16} The surface crack starts growing slowly under appropriate tensile stress conditions until an extension of crack reaches a critical size after which the unstable crack propagates fast and a catastrophic failure occurs instantaneously.

Two types of loading conditions, static and cyclic loading, may contribute to the delayed failure. In static loading, a constant load is applied to the specimen for an extended period of time t . Comparatively, in cyclic loading the stress changes periodically with time. In both cases, the fatigue damage can be described by a relation between the crack growth rate and the stress intensity factor for the initial crack. Crack growth rate $v = da/dt$ under static loading conditions and crack growth rate per cycle da/dN under cyclic loading can be described by power law relationships:¹⁷

$$v = A_s^* (K_I / K_{Ic})^{n_s} \quad (1)$$

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and

$$da/dN = A_c^* (\Delta K_I / K_{Ic})^{n_c} \quad (2)$$

where a is the crack size; K_I is the stress intensity factor; K_{Ic} is the fracture toughness of the material; $\Delta K_I = K_{I\max} - K_{I\min}$ is the range of stress intensity factor; $K_{I\max}$ is the maximum stress intensity factor in a loading cycle; $K_{I\min}$ is the minimal stress intensity factor in the loading cycle; n_s is the static slow crack growth exponent; A_s^* is the material parameter under static loading; n_c is the cyclic slow crack growth exponent; and A_c^* is the material parameter under cyclic loading.

Under static load the lifetime can be estimated using the following expression:¹⁸

$$t_f = B_s \sigma_c^{n_s-2} \sigma^{-n_s} \quad (3)$$

where $B_s = 2K_{Ic}^2 / (A_s^* Y^2 (n_s - 2))$, σ_c is the instantaneous strength of the material; σ is the applied stress; and Y is the constant defining the shape of the crack (typically 1.3 for surface cracks).

The lifetime in cyclic loading (cycles to failure) is presented by:¹⁸

$$N_f = B_c \sigma_c^{n_c-2} \Delta \sigma^{-n_c} \quad (4)$$

where $B_c = 2K_{Ic}^2 / (A_c^* Y^2 (n_c - 2))$, $\Delta \sigma$ is the stress range ($\Delta \sigma = \sigma_{\max} - \sigma_{\min}$); $\sigma_{\max}$ is the maximum stress in a loading cycle; and $\sigma_{\min}$ is the minimal stress in a loading cycle.

The parameters (n_s and A_s^*) and (n_c and A_c^*) are used to estimate the resistance of the material to the slow crack growth in static and cyclic fatigue, respectively. The slow crack growth exponents n_s and n_c in Eqs. (1)–(4) are parameters for determining the effect of stress intensity factor on crack growth rate. A larger exponent magnitude results in less sensitivity of crack propagation to the stress intensity factor.

For most ceramics, slow crack growth exponents have values greater than 15.¹⁸ For materials with a n_s value of over 200, the materials are considered insensitive to slow crack growth. Additional material crack growth parameters (A_s^* and A_c^*) are used to determine the maximum crack growth rate in static and cyclic fatigue processes respectively.

The fatigue parameters depend strongly on environmental conditions.¹⁷ The value of A_s^* varies from 10^{-5} – 10^{-7} m/s, and the value of A_c^* is in the order of 10^{-17} – 10^{-19} m/cycle for materials with very slow crack growth. However, these parameters can be much higher; especially in aggressive environmental conditions or at high temperatures. As a result, the lifetime of the material before failure can be very limited due to significant crack growth.

There are several methods used to determine the fatigue parameters.^{18,19} The first method of conventional evaluation uses linear regression of $\log(t_f)$ versus $\log(\sigma)$ for static fatigue and $\log(N_f)$ versus $\log(\Delta \sigma)$ for cyclic fatigue. The advantage of this method is that lifetime prediction for different applied stresses during operation of a

component can be obtained from lifetime measurements. The disadvantage to this method is that the procedure is very time consuming; many tests at different stress levels are necessary.¹⁸ Also, if the number of recorded data points is insufficient, high uncertainties in fatigue parameters do not provide a reliable estimate. The second method uses Weibull distribution of lifetimes t_f or N_f . This technique also requires many fatigue tests and is therefore just as time consuming as the linear regression method. The third method is a technique which uses linear regression of $\log(\sigma/\sigma_c)$ versus $\log(t_f \sigma^2)$ for static fatigue and $\log(\Delta \sigma/\sigma_c)$ versus $\log(N_f \Delta \sigma^2)$ for the determination of cyclic fatigue parameters. This technique obtains fatigue parameters, even for data at one stress level or stress range, and provides appropriate accuracy for a reduced number of fatigue tests, comparing with the above methods.

ZrB₂ based ceramic composites are an important class of materials used in different industrial and military applications.^{20–24} Instantaneous mechanical properties of ZrB₂-30 wt% SiC composites were reported in Ref. [25]. The goal of this paper is to report ZrB₂-30 wt% SiC ceramic composite fatigue parameters tested in dry air, air with ~40% humidity, and water, by utilizing linear regression of $\log(\sigma/\sigma_c)$ versus $\log(t_f \sigma^2)$ for static fatigue and $\log(\Delta \sigma/\sigma_c)$ versus $\log(N_f \Delta \sigma^2)$ for cyclic fatigue.¹⁸

2. EXPERIMENTAL DETAILS

Room temperature static and cyclic fatigue experiments were performed with the details of the experiments reported in Ref. [26]. The delayed failure experiments under static loads were performed in air according to Ref. [27]. The tests were performed in specially constructed dead-load equipment. The apparatuses were decoupled from surrounding vibration or shock. The bar-specimens were loaded in four-point bending using 40/20 mm load geometry at stresses equal to 80, 83, and 86% of the Weibull scale parameter σ_0 of our material (724 MPa).²⁵ Eleven specimens were tested at each stress level. The sample bars were kept under a constant stress at room temperature and relative humidity of 30–40%. The time to failure was recorded using an automatic stop watch built on the machine. For these static lifetime tests, a time limit of 168 h (7 days) was chosen.

The cyclic fatigue tests were set up to determine the lifetime (cycles to failure) of ceramic specimens using a cantilever bending device (Fig. 1) originally developed by Munz and Fett.²⁸ During the lifetime measurements, a cyclic load was applied to the cantilever by two lateral loudspeakers. The cyclic movement of the loudspeakers' coil-magnet system was transmitted to the cantilever using two flexible metallic wires. A force applied to the bottom of a long cantilever submitted a bending moment to the sample attached to the cantilever's top surface. To ensure a proper fixation between the sample and the cantilever, all the specimens are glued to brass adapters using an epoxy

Fig. 1. Schematics of the mechanical testing device used for samples' oscillatory bending mode loading in cyclic fatigue experiments. (1)—ceramic specimen; (2)—specimen holder; (3) a cantilever; (4)—coil magnet system used in loud speakers; (5)—flexible wire.

resin. The tensile stress developed on the sample surface upon the application of the bending moment M can be calculated based on the simple bending equation:

$$\sigma = \frac{Mz}{I}$$

where z is the distance between the surface and the neutral axis of the specimen (in this case, $h/2$, with h as the sample height); and I is the moment of inertia (in this case, $wh^3/12$, where w is the sample width). A sine waveform stress with constant maximum amplitude $\sigma_{\max}$ equal to 80, 83, and 86% of the Weibull scale parameter ($\sigma_0 = 724$ MPa) of our material (580, 600, and 620 MPa respectively) was applied to the specimen surface at a fixed frequency of 10 Hz ($R = \sigma_{\min}/\sigma_{\max} = -1$). The final load with a maximum amplitude was not applied to the sample from the very first instant of the test, but rather was built up slowly from a low value to prevent overloading. The rate of build-up was such that it took 15 sec, which is equal to 150 load cycles, to reach 95% of the full load. The first 150 cycles have been ignored, and samples that failed before the 150th cycle were classified as an instantaneous failure. Load cycle counting started after 95% of the full load was applied. In such a loading scheme, both surfaces of the specimen are alternately exposed to the same magnitude tensile and compressive stresses. The number of cycles to failure is automatically recorded. The maximum numbers of cycles were chosen to be 10^6 , and if the sample did not fail after this number of cycles the experiment was stopped and the sample was removed from the testing machine. A total of ~ 28 hours were approximately spent to perform 10^6 cycles.

The cyclic fatigue tests were conducted under three different environmental conditions. The first set of cyclic experiments was conducted in a water environment at 100% relative humidity. The second set of cyclic tests was performed in air at about 30–40% relative humidity and the third set of cyclic tests was performed in dried air. To perform experiments under a water environment, a silicone tube was tightly fit over the specimen adapter and filled with distilled water. No silicone tube was used to perform the test in air, and the specimen was exposed to the lab environment. To perform tests in dry air, the silicone tube was filled with silica gel to remove humidity. Eleven specimens were tested for cyclic fatigue in each media (water, air, and dry air).

3. RESULTS AND DISCUSSION

3.1. Static Fatigue

As it was specified in the experimental part of the paper, the static subcritical crack growth was studied at three pre-selected 580, 600, and 620 MPa constant stresses. Due to Weibull related scattering of the strength data of the ZrB₂-30 wt% SiC ceramic composite, there were samples which failed instantaneously during loading and samples which did not fail within 168 hours of testing. Such scattering in the lifetime data depends on the critical flaw size distribution in ceramic composite. Therefore, the scattering in lifetime also correlates to the initial strength scattering.¹⁸ At 580 MPa stress level, there was only one sample that failed instantaneously from the batch of eleven tested. However, when the stress level increased to 600 MPa, already three samples failed instantaneously, and when the stress further increased to 620 MPa, eight out of eleven samples failed before loading started. At the lowest applied stress, there were nine samples which did not fail after a seven day dwelling at 580 MPa, thus leaving only one sample to fail within the limits of pre-determined 168 hour timeframe. At 600 MPa applied stress four samples did not fail after seven days of loading, leaving four samples to fail within the seven days of testing. At the highest stress of 620 MPa, only two samples survived longer than seven days without failure, and one failed within the seven day timeframe. The results when the samples failed instantaneously at or below specified constant load and those that did not fail after seven days of testing were considered as censored data.

3.2. Static Fatigue Parameters

Static fatigue parameters can be estimated by linear regression of $\log(\sigma/\sigma_c)$ versus $\log(t_f \sigma^2)$ plot.¹⁸ In order to calculate $\log(\sigma/\sigma_c)$ and $\log(t_f \sigma^2)$, the lifetime t_f and the instantaneous strength σ_c of a specimen must be known. However, this is not possible because the measurements of lifetime and inert strength have to be done on the same sample. Therefore, while a measurement of lifetime t_f of a sample can be performed using static fatigue experiments,

the instantaneous strength σ_c of the same sample must be determined by applying a statistical approach.

Using the statistical approach, the cumulative distribution function F (or failure probability) of lifetime and instantaneous strength are considered identical for the same sample.¹⁸ For the evaluation of the failure probability for the lifetime of a sample, all were measured in N lifetime values, where N is the total number of tested samples, and were ranked in an increasing order numbered from 1 to N . Then, the single lifetime t_f values are related to failure probability, and F is determined as $(i - 0.5)/N$ (where i is the number of the corresponding value of lifetime t_f in the experiment). After failure probability for the lifetime of a sample in static fatigue is determined, the instantaneous strength of a sample failed at a certain lifetime can be determined from the Weibull distribution:

$$\sigma_c = \sigma_0 \left(\frac{S_0}{S_{\text{eff}}} \ln \frac{1}{1-F} \right)^{1/m} \quad (5)$$

where m is Weibull modulus and σ_0 is Weibull scale parameter for instantaneous four point bending strength; S_{eff} is the effective surface area of sample for fatigue test; and S_0 is the surface area of sample for four point bending tests to measure the instantaneous strength. Since S_{eff} and S_0 have the same values in static fatigue experiments, Eq. (5) simplifies to:

$$\sigma_c = \sigma_0 \left(\ln \frac{1}{1-F} \right)^{1/m} \quad (6)$$

After determining both t_f and σ_c for each sample, the slope of the linear approximation of $\log(\sigma/\sigma_c)$ versus $\log(t_f \sigma^2)$ provides $d[\log(\sigma/\sigma_c)]/d[\log(t_f \sigma^2)] = -1/(n_s - 2)$ and the intercept $1/(n_s - 2) \log B_s$ for the linear approximation with $\log(\sigma/\sigma_c)$ axis gives A_s^* , where:

$$n_s = 2 - \left(\frac{d[\log(\sigma/\sigma_c)]}{d[\log(t_f \sigma^2)]} \right)^{-1} \quad (7)$$

and

$$A_s^* = \frac{2K_{1c}^2}{Y^2(n_s - 2) \log^{-1}[(n_s - 2)D_s]} \quad (8)$$

and $D_s = (1/(n_s - 2)) \log B_s$ is the intercept value.

For each t_f , the instantaneous strength (σ_c) was calculated using $m = 6.7$ and $\sigma_0 = 724$ MPa for ZrB₂-30 wt% SiC²⁵ substituted in Eq. (6). Overall, 11 samples were tested at each of the stress levels (580, 600, and 620 MPa) during static fatigue experiments, totaling to 33 t_f data points. However, due to a significant number of samples which either failed before time counting began or did not fail during the experiment, certain “censored” data points were not taken into account for the fatigue parameters calculation. After omitting the “censored” data points, only six data points remained, taken within the defined time interval of 168 hours of testing, which were used for plotting $\log(t_f \sigma^2) - \log(\sigma/\sigma_c)$ static fatigue data for test in

Fig. 2. Auxiliary plot for the evaluation of fatigue parameters (the quality of the linear fit $R^2 = 0.95$) (A) and logarithm of crack growth rate, v [m/s], versus logarithm of K_I/K_{Ic} curves for static loading (B).

air Figure 2(A). From this auxiliary plot, the static fatigue parameters are calculated as $n_s = 68$ and $A_s^* = 7.3 \cdot 10^{-7}$ m/s. Static fatigue parameters and the number of data points which were used for static fatigue parameters calculations are summarized in Table I. More detailed statistical analysis of the experimental data gives 90% confidence interval for n_s as $38 \leq n_s \leq 98$.²⁹

Table I. Static fatigue parameters.

	Air
Static slow crack growth exponent, n_s	68
Material parameter, A_s^* , m/s	$7.3 \cdot 10^{-7}$
Number of tested samples	33
Number of censored data points	27
Number of valid data points	6

Using the calculated fatigue parameters, the crack growth rate $v = da/dt$ was determined by utilizing the relationship $K_1/\sigma = K_{1c}/\sigma_c$ (i.e., $\log(\sigma/\sigma_c) = \log(K_1/K_{1c})$)¹⁸ and power law Eq. (1), and $\log(v)$ versus $\log(K_1/K_{1c})$ is plotted (Fig. 2(B)), where the fracture toughness K_{1c} of ZrB₂-30 wt% SiC composite was measured to be equal to $3.8 \pm 0.1 \text{ MPa} \cdot \text{m}^{1/2}$.²⁵ Static fatigue parameters of ZrB₂-30 wt% SiC composite correspond to satisfactory resistance of the material to fatigue crack growth and allow for the prediction of the lifetime of the composite under constant stress level.

3.3. Cyclic Fatigue

The cyclic fatigue was studied at 1160, 1200, and 1240 MPa stress amplitude ranges $\Delta\sigma = 2\sigma_{\max}$. Three different environments, water, air and dry air, were used for the cyclic fatigue tests. Similar to the static fatigue, a large scattering of the lifetime results are obtained during cyclic fatigue experiments due to the Weibull related scattering of defects presenting at the surface and inside ZrB₂-30 wt% SiC ceramics. Both processing related and machine related defects contributed to the fatigue failure, and a certain number of samples failed outside the specified time interval of the testing-either failing instantaneously before reaching the specified cyclic load or surviving the cycling tests without failure. Such samples were classified as censored data similar as it was done in static slow crack growth experiments. The increase in applied stress amplitude range brought an increase in a number of censored samples which failed instantaneously from 3 for $\Delta\sigma = 1160 \text{ MPa}$ to 7 when $\Delta\sigma = 1240 \text{ MPa}$ (cyclic tests performed in water), but the number of samples failing instantaneously at $\Delta\sigma = 1240 \text{ MPa}$ decreased from 7 (when the relative humidity was 100% for tests in water) to 5. When the relative humidity decreased to 30–40% of the typical laboratory environment, it decreased to 2 when the silica gel was used to absorb moisture from the air to further decrease the relative humidity in the testing environment. However, it is important to notice that the number of samples survived more than a selected 10^6 number of cycles is much lower than those survived the static slow crack growth testing. In water, there were only two samples which remained intact after 10^6 cycles at 1200 MPa stress range experiments, while at $\Delta\sigma = 1160 \text{ MPa}$ and 1240 MPa no sample survived one million cycles of the bending loading. In air, two samples survived cyclic tests at $\Delta\sigma = 1160 \text{ MPa}$; one sample survived both at 1200 MPa and 1240 MPa stress ranges. However, in dry air, five samples survived one million cycles at the highest $\Delta\sigma = 1240 \text{ MPa}$ stress range.

3.4. Cyclic Fatigue Parameters

A similar approach that was applied to treat static fatigue results was utilized to determine the cyclic fatigue parameters, which were also estimated by linear regression of $\log(\Delta\sigma/\sigma_c)$ versus $\log(N_f\Delta\sigma^2)$ plot.^{18,28} The cumulative

distribution function F of the lifetime for cycles to failure was estimated using the same method as for the static fatigue data. After the determination of F , the instantaneous strength of the sample failed at a certain lifetime was determined using Eq. (5). Unlike in the static fatigue parameters, the S_{eff} and S_0 are not the same, and their ratio is calculated to be 1.072 in our cyclic fatigue experiments.¹⁸ After the instantaneous strength is determined, the n_c and A_c^* fatigue parameters were then calculated from the slope of linear approximation of $\log(\Delta\sigma/\sigma_c)$ versus $\log(N_f\Delta\sigma^2)$ plot, which is equal to $d[\log(\Delta\sigma/\sigma_c)]/d[\log(N_f\Delta\sigma^2)] = -1/(n_c - 2)$ and the intercept $D_c = (1/(n_c - 2)) \log B_c$ of linear approximation with $\log(\Delta\sigma/\sigma_c)$ axis supplies A_c^* , where

$$n_c = 2 - \left(\frac{d[\log(\Delta\sigma/\sigma_c)]}{d[\log(N_f\Delta\sigma^2)]} \right)^{-1} \quad (9)$$

and

$$A_c^* = \frac{2K_{1c}^2}{Y^2(n_c - 2) \log^{-1}[(n_c - 2)D_c]}$$

The cyclic fatigue experiments were performed at three different stress ranges, $\Delta\sigma = 1160, 1200, \text{ and } 1240 \text{ MPa}$, in air with $\sim 40\%$ humidity and water, and at one stress range $\Delta\sigma = 1240 \text{ MPa}$ in dry air. Similar to the static fatigue experiments, a number of data points for samples tested in cycling failed out of the preset up range of allowable 1,000,000 cycles. The data points for samples which failed prior to the corresponding stress range had been reached were omitted, reducing the number of data points within each trial. The number of data points which were used for cyclic fatigue parameters calculations is shown in Table II. They are used for plotting $\log(\Delta\sigma/\sigma_c)$ versus $\log(N_f\Delta\sigma^2)$ auxiliary graph (Fig. 3(A)), where cyclic fatigue parameters were found. The fatigue parameters were determined for testing in all three different environments and they are summarized in Table II. More detailed statistical analysis of the experimental data gives 90% confidence interval for n_c as $23 \leq n_c \leq 41$ for testing in dry air, $22 \leq n_c \leq 38$ for testing in air, and $21 \leq n_c \leq 57$ for testing in water.²⁹ To determine the crack growth rate da/dN using the fatigue parameters, the relationship between da/dN and $\Delta K_1/K_{1c}$ has to be established.

Utilizing the relationship $\Delta K_1/\Delta\sigma = K_{1c}/\sigma_c$ (i.e., $\log(\Delta\sigma/\sigma_c) = \log(\Delta K_1/K_{1c})$)²⁸ and Eq. (2), the dependence of $\log(da/dN)$ as a function of $\log(\Delta K_1/K_{1c})$ is

Table II. Cyclic fatigue parameters.

	Dry air	Air	Water
Cyclic slow crack growth exponent, n_c	32	30	39
Material parameter, A_c^* , m/cycle	$6.5 \cdot 10^{-19}$	$2 \cdot 10^{-17}$	$2.2 \cdot 10^{-17}$
Number of tested samples	11	33	33
Number of censored data points	7	13	18
Number of valid data points	4	20	15

Fig. 3. Auxiliary plot for the evaluation of fatigue parameters (A) and logarithm of crack growth rate per cycle, da/dN [m/cycle], versus logarithm of $\Delta K_I/K_{Ic}$ curves (B) for natural surface flaws in water (1), air (2) and dry air (3). Dashed line (4) corresponds to cyclic fatigue parameters evaluated from static fatigue parameters when there is no cyclic effect. The quality of the linear fit in (A): $R^2 = 0.48$ (water); $R^2 = 0.74$ (air); $R^2 = 0.99$ (dry air).

presented in Figure 3(B) for testing in three different environments. As is shown in Figure 3(B), the crack growth is faster when the water content increases. Thus, the fastest crack growth occurs in an environment with water and the slowest crack growth occurs in dry air.

The effect of humidity on cyclic fatigue of the ZrB₂-30 wt% SiC ceramic composite is similar to the effect of water vapor on crack growth in glass, where a crack growth rate is a function of both applied stress intensity factor and a relative humidity.^{5,30} It was proposed that crack growth rate is dependent on the chemical reactions occurring between water vapor and the strained interatomic bonds of the material at the crack tip. Such chemical reactions are responsible for the facilitation of the crack growth rate in materials subjected to

cyclic fatigue when water vapor is present, as shown in Figure 3(B), in ZrB₂-30 wt% SiC in particular.

3.5. Lifetime Prediction

For the ZrB₂-30 wt% SiC ceramic composite, the relationship between applied stress and predicted time to failure is of importance, because it allows for the prediction of the material lifetime. Therefore, a question of prediction of the lifetime at a given level of applied stress can be answered if the fatigue parameters and crack growth rate versus stress intensity factor dependence is known. The calculated lifetimes of ZrB₂-30 wt% SiC ceramic composites under static and cyclic fatigue are shown in Table III. As the applied stress level both in static and cyclic tests is rather high, the lifetime of the samples is overall quite low. It is measured in hours for constant static loading for the 580 MPa and 600 MPa applied stress for testing in air, as well as $\Delta\sigma = 1160$ MPa for cyclic applied stress range for testing in dry air. However, lifetime quickly decreases to minutes, when either applied stress range is increased to $\Delta\sigma = 1200$ MPa and 1240 MPa, for testing either in dry air or air with 40% humidity. The lifetime of ZrB₂-30 wt% SiC ceramics are substantially decreased to seconds when the environment of testing is of water, indicating that the material is highly susceptible to the slow crack growth in high humidity. Such short lifetimes could be of great concern for the application of a ZrB₂ based composite, especially in an environment where water molecules are present in high quantities.

3.6. Cyclic Effect

The prediction of lifetime t_{fc} under cyclic loading from the lifetime t_f in static loading can be made using the following equation^{18,28}

$$t_{fc} = t_f \left(\frac{\sigma}{\sigma_{\max}} \right)^{n_s} \frac{\lambda}{h} \quad (10)$$

under the assumption of no additional cyclic fatigue effects being present if both static and cyclic fatigue experiments are performed in the same environment. By using Eq. (10), one can determine if a material exhibits an increase in a crack growth due to additional cyclic effect or cyclic

Table III. Time to failure 50% samples at given stress levels.

	σ		
	580 MPa	600 MPa	620 MPa
Static fatigue			
Air	18 h	1.8 h	11.6 min
	$\Delta\sigma$		
	1160 MPa	1200 MPa	1240 MPa
Cyclic fatigue			
Dry air	2.5 h	51 min	18 min
Air	15 min	5.5 min	2 min
Water	5.5 s	1.5 s	0.4 s

effect is not present.¹⁸ The function $\lambda = (S_{\text{eff}}/S_0)^{-(n_s-2)/m}$ takes into account different effective surfaces in static and cyclic tests and $h = 0.395/\sqrt{n_s}$, where n_s is determined by Eq. (7) and m is Weibull modulus.²⁰ The $\log(\Delta\sigma/\sigma_c)$ versus $\log(N_f\Delta\sigma^2)$ dependence is presented by:

$$\log \frac{\Delta\sigma}{\sigma_c} = -\frac{1}{n_s-2} \log(N_f\Delta\sigma^2) + D_s - \frac{1}{n_s-2} \log\left(\frac{0.395}{2^{n_s} f \lambda \sqrt{n_s}}\right) \quad (11)$$

when $t_{fc} = N_f/f$, where N_f is determined by Eq. (4) and f is the frequency of cyclic loading ($f = 10$ Hz in this case). Both n_s (Eq. (7)) and D_s (Eq. (9)) are the fatigue parameters determined by static fatigue tests. The n_c is equal to n_s under such consideration and the second term in the right part of Eq. (11) $D_s - (1/(n_s-2)) \log(0.395/(2^{n_s} f \lambda \sqrt{n_s}))$ is called D' , then the A_c^* for crack growth rate per cycle without consideration of the cyclic effect is equal to $2K_{1c}^2/(Y^2(n_s-2) \log^{-1}[(n_s-2)D'])$.

The described approach was used to calculate the crack growth rate in the cycling of the ZrB₂-30 wt% SiC composite, where no cycling effects were considered. The equation

$$\log(da/dN) = n_s \log(\Delta K_1/K_{1c}) + \log(A_c^*) \quad (12)$$

was used to plot the crack growth rate as a function of stress intensity factor range. As A_c^* fatigue parameter in Eq. (12) was calculated using D' , where cyclic fatigue parameters were determined from static fatigue parameters (when no cyclic effect was taken into account). Such crack growth behavior is presented in Figure 3(B) using a dashed line, where one can see that the crack growth determined from static fatigue parameters, without taking into account extra cyclic effect, does not correspond to the experimental data of cyclic fatigue tests. It is obvious that crack growth is faster in the cyclic fatigue experiment that can be explained by the proposed simple static fatigue mechanism.¹⁸

It was proposed that bridging interactions between two surfaces of the growing crack will affect the slow crack growth during the constant loading in static fatigue experiments.^{31,32} Such interactions will produce shielding stress intensity factor K_{sh} , which decreases total effective stress intensity factor in the crack tip and decelerates the crack growth rate. This effect is significant when there are crack bridging interactions in the ceramic composite, which were shown to be present in ZrB₂-30 wt% SiC (Fig. 4).²⁵ However, the crack interactions will be increasingly reduced in cyclic loading due to presence of wear. Thus, the cyclic effect could be interpreted as shielding stress intensity factor degradation. For the crack bridging effect, the following crack growth rate relationship is proposed:³²

$$v = A' \left(\frac{K_{\max} - K_{sh}}{K_{1c}} \right)^{n'} \quad (13)$$

Fig. 4. Crack bridging in ZrB₂-30 wt% SiC ceramic composite.

where the fatigue parameter A' depends both on frequency and on the shape of the loading cycles. $K_{\max}$ is equal to K_1 for static fatigue tests, $K_{\max}$ is equal to $\Delta K_1/2$ for cyclic fatigue tests with $R = \sigma_{\min}/\sigma_{\max} = -1$, and n' is the effective fatigue exponent. The shielding term K_{sh} is presented as a simple power-law representation:³²

$$K_{sh} = \begin{cases} K_{sh0}, & v \geq v_0, R \geq R_0 \\ K_{sh0}(v/v_0)^{C(R_0-R)}, & v < v_0, R < R_0 \end{cases} \quad (14)$$

where K_{sh0} , v_0 , C and R_0 are material parameters. Using Eqs. (13) and (14), one obtains

$$\log(K_{\max}/K_{1c}) = \log \left(\frac{K_{sh0}}{K_{1c}} + \left(\frac{v}{A'} \right)^{1/n'} \right) \quad v \geq v_0, \quad R \geq R_0 \quad (15)$$

and

$$\log(K_{\max}/K_{1c}) = \log \left(\frac{K_{sh0}}{K_{1c}} \left(\frac{v}{v_0} \right)^{C(R_0-R)} + \left(\frac{v}{A'} \right)^{1/n'} \right) \quad v < v_0, \quad R < R_0 \quad (16)$$

R_0 is considered to equal 1;³¹ The crack growth rate v_0 is determined from the intersection point of the $\log(v) - \log(K_{\max}/K_{1c})$ curves for different R , where R is equal to 1 for static fatigue tests and R is equal to -1 for cyclic fatigue tests. The other parameters, K_{sh0}/K_{1c} , n' , A' , and C are obtained by fitting static and cyclic experimental data, where K_{sh0}/K_{1c} , n' , and A' parameters are estimated from static fatigue data, and C is found by fitting the corresponding cyclic fatigue data in air with 40% humidity. After all the parameters determining the shielding effect K_{sh} are known, Eqs. (15) and (16) are used to plot the corresponding $\log(v)$ versus $\log(K_{\max}/K_{1c})$ dependencies (taking the crack growth rate range from Figs. 2(B) and 3(B)). For static tests, v data presented in Figure 2(B) were used and for cyclic tests, v was calculated as a product of da/dN and frequency f where $v = f(da/dN)$ and $K_{\max} = \Delta K_1/2$. Based on the experimental data, the values of $v_0 = 6.34 \cdot 10^{-8}$ m/s, $K_{sh0}/K_{1c} = 0.252$, $n' = 50.7$,

Fig. 5. Logarithm of crack growth rate, v [m/s], versus logarithm of $K_{\max}/K_{1c}$ curves for static and cyclic loading. Squares show static fatigue data. Circles show cyclic fatigue data. Solid lines are plotted according to crack bridging model. Dashed line shows dependence for cyclic fatigue calculated from static fatigue data without crack bridging degradation.

$A' = 2.143$ m/s, and $C = 0.039$ were obtained by fitting the data points for static and cyclic fatigue in air. The $\log(v)$ versus $\log(K_{\max}/K_{1c})$ plots for static and cyclic fatigue tests are combined together in Figure 5. As one can see from Figure 5, the crack propagates faster in cyclic tests because of wear presence during cyclic loading, which decreases the crack bridging interaction. The $\log(v)$ versus $\log(K_{\max}/K_{1c})$ cyclic fatigue dependence calculated from static fatigue data, which does not include crack bridging degradation, is also shown in Figure 5 as a dashed line. When cyclic degradation is not considered in cyclic fatigue, the $\log(v)$ versus $\log(K_{\max}/K_{1c})$ plot is parallel to the static fatigue data line, but shows even lower crack growth rate. Given that only the tensile part of the cycle is considered, time of exposure to the tensile stress is lower than that exposure of the sample to tensile stress under constant static load.

4. CONCLUSIONS

The room temperature fatigue experiments were performed to determine time dependent mechanical behavior of ZrB₂-30 wt% SiC ceramic composite. Static and cyclic fatigue parameters were calculated using the linear regression technique, which permitted accurate estimation of the fatigue parameters where limited numbers of experimental data points were available. The slow crack growth rates were estimated using the calculated fatigue parameters and the lifetime prediction for different applied stresses in different environments was performed. It was found that $\log(da/dN)$ versus $\log(\Delta K_1/K_{1c})$ plots for specimens tested in water, air, and dry air indicate that cracks grow quickest in water environment, and slightly slow down when the environment is dry air. It indicated that water

accelerates crack growth in ZrB₂-30 wt% SiC. It was also found that at such relatively high applied stresses, ZrB₂-30 wt% SiC ceramics will only last for 18 hours, for the most favorable static loading at 580 MPa applied stress, in dry air where the least quantity of water molecules are present. The lifetime quickly deteriorates when either applied stress is increased or cyclic load is used, or when the concentration of water is increased.

It was also found that during the cyclic loading, the cracks grow faster than can be explained by a simple static fatigue mechanism. The fatigue behavior (where $n_s > n_c$) can be explained by degradation of bridging interactions between two crack surfaces and a corresponding decrease in shielding stress intensity factor. The dependencies $\log(v)$ versus $\log(K_{\max}/K_{1c})$ calculated in the crack bridging model correspond well with experimental data.

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