JOURNAL OF MATERIALS SCIENCE 17 (1982) 1297–1302

Evaluation of slow crack growth parameters for silicon carbide ceramics

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The difficulties in applying existing dynamic and static fatigue theories to model the slow crack growth behaviour of silicon carbide ceramics are examined critically. The assumptions that the geometric parameter, Y, in the fracture mechanics relation remains unchanged for all flaws during crack growth and that the final crack size is much larger than the initial crack size are found to be unrealistic for silicon carbide ceramics.

1. Introduction

In designing ceramics for structural applications, two important aspects of the strength behaviour of these materials must be carefully considered. The first aspect is the statistical variation of strength. This property is conveniently characterized by a Weibull probability function and can be successfully incorporated into the probabilistic ceramic design. The second aspect pertinent to ceramic design is the slow (or subcritical) growth of pre-existing flaws under service environments. This time-dependent behaviour is not as well characterized as strength in most ceramic materials. The importance of an accurate evaluation of the slow crack growth aspect in reliable lifetime predictions of ceramic components cannot be overemphasized. Many pioneering investigations on glass [1, 2] under various chemically active environments have established several functional forms for slow crack growth behaviour. This methodology has been adapted for crystalline ceramics as well [3–5]. The purpose of this paper is to critically evaluate this slow crack growth model; and to examine the data reduction procedures as applied to silicon carbide ceramics (Hexoloy SA and a fine-grain, reaction-sintered SiC)* which are inherently less susceptible to slow crack growth than most other ceramics.

2. Slow crack growth in ceramics

A function form that is commonly used to describe the slow crack growth of ceramics has evolved from the investigations on glass and can be written as:

$$v = da/dt = A\,(K_I/K_{Ic})^n, \quad (1)$$

where v is the rate of growth of the crack with a characteristic dimension, a, when the applied stress intensity factor equals K_I. The critical stress intensity factor is denoted by K_{Ic}. The parameters A and n define the slow crack growth behaviour of the material.

In the past, many life-prediction methodologies have been developed [6–8] based on this relation and the well-known fracture mechanics relation between the applied stress, σ, the stress intensity factor, K_I, and the characteristic flaw dimension, a:

$$K_I = \sigma Y (a)^{1/2}, \quad (2)$$

where Y is a geometric factor that depends on the flaw shape, the orientation, the location and the nature of the applied stress field.

The prediction of the lifetime of a ceramic component or the risk of rupture of the component after a specified service history necessitates precise estimation of the strength distribution parameters as well as the slow crack growth parameters.

3. Evaluation of slow crack growth parameters

The parameters A and n can be estimated from direct velocity measurement techniques or some indirect techniques. The direct velocity experi-

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0022–2461/82/051297–06$03.22/0

ments are generally difficult to perform for most high-performance ceramics, because the temperatures at which slow crack growth becomes significant often exceeds 1000° C. These experiments involve measuring the compliance of notched specimens (double torsion, single-edge notched beam, etc.) as the crack grows under an applied stress. The compliance is related to crack size and therefore the crack velocity can be computed from the measured compliance changes. The primary requirement for the success of such tests is the ability to obtain stable crack growth under the desired experimental conditions. Numerous attempts to sustain stable crack growth in Hexoloy SA at temperatures above 1200° C in both double torsion and single-edge notched beam fracture mechanics configurations have been unsuccessful [9–11]. Therefore, two indirect test techniques, which have been successfully used for glass and other ceramics [12, 13], are considered in this study.

Both tests have been used for characterizing the slow crack growth behaviour of SiC [14, 15]. The first technique, the static stress rupture test, involves loading the specimen in constant stress conditions and allowing the flaw to grow. The times to failure obtained at various applied stress levels are used in evaluating the slow crack growth (SCG) parameters. These tests are generally conducted over long periods of time (several hundred hours), imposing a practical limit on the number of specimens evaluated. The dynamic fatigue test, on the other hand, can be completed in a short time and allows a reasonable statistical population to be tested. This test uses several loading rates to obtain the strength distributions of the material. Slow crack growth parameters are calculated based on an analysis of the effect of loading rates on strength. The application of these indirect methods and the corresponding analysis and data reduction procedures to results obtained for Hexoloy SA is questionable and will be detailed from the existing analyses.

4. Data reduction and analysis

4.1. Static stress rupture

Specimens are normally loaded in flexure until failure occurs. The times to failure, t_f, are then related to the applied stress, σ_a, as follows: from Equations 1 and 2

$$\int_0^{t_f} dt = \int_{a_i}^{a_f} (1/A)(K_{Ic}/\sigma_a Y)^n \, (da/a^{n/2}), \quad (3)$$

where a_i and a_f are the initial and final flaw sizes. Many assumptions are characteristically made at this point and the end results used without careful regard for their importance. For example:

(a) The integral on the right-hand side of Equation 3 contains σ_a which is considered to be a constant. This assumption is reasonable as the size of the flaw is small and the variation of the applied stress across the flaw from the non-uniform stress induced by flexure is insignificant. However, the stress applied at the flaw at failure should be used instead of the maximum outer fibre stress often used in practice.

(b) Generally, the factor Y is also assumed to be invariant during flaw growth to simplify the integral. This assumption is not necessarily valid for realistic fracture origins. In SiC ceramics, failure-initiating flaws are generally three-dimensional voids or agglomerates, often with sharp corners [16]. It must be recognized that the Y factor is a complicated function of the flaw dimensions and more than one variable may be required to characterize the flaw size [17, 18]. Flaw growth may be considered as limited, with a negligible effect on Y, but this assumption will complicate subsequent analysis, as will be shown.

(c) The derivation can be continued, maintaining σ_a and Y invariant, to obtain the lifetime:

$$t_f = [2/A(n-2)] \, (K_{Ic}/\sigma_a Y)^n \, [(1/a_i)^{(n-2)/2} - (1/a_f)^{(n-2)/2}]. \quad (4)$$

A critical and often overlooked assumption is that the final crack size, a_f, is much larger than the initial crack size, a_i, or $(1/a_i)^{(n-2)/2} \gg (1/a_f)^{(n-2)/2}$. Equation 4 then simplifies to

$$t_f = [2/A(n-2)] \, (K_{Ic}/\sigma_a Y)^n \, (1/a_i)^{(n-2)/2}. \quad (5)$$

This assumption contradicts that of the previous step in which the factor Y did not change significantly during flaw growth.

Intrinsic flaws which initiated failure in Hexoloy SA during stress rupture experiments are shown in Fig. 1. Growth patterns are not readily visible along the flaw boundary, so that the assumption $a_f \gg a_i$ cannot be validated for intrinsic flaws in Hexoloy SA by fractographic examination. The errors associated with this step will vary from specimen to specimen depending on the flaw growth ratio, a_f/a_i, and generally will be larger for those specimens which fail after shorter periods and/or under conditions less favourable for SCS.

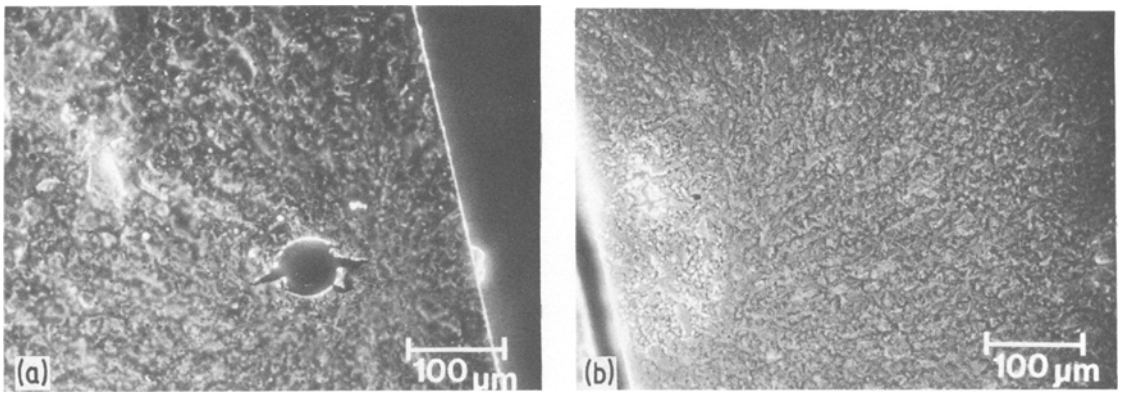

Figure 1 Fracture origins for two Hexoloy SA specimens tested at 1500° C in stress rupture experiments. $\sigma_a = 290$ MPa; t_f = (a) 2.5 h, (b) 90 h.

(d) The definition of the initial flaw size, a_i in Equation 5, is also a potential problem. This quantity is related to the "inert" strength, σ_i, (without SCG) and the critical stress intensity factor, K_{Ic}, indirectly through Equation 2. (It should be noted that Equation 2 is used here in a "back-door" fashion to replace flaw dimensions by stress intensity terms.) The initial flaw size is thus expressed as

$$a_i = (K_{Ic}/\sigma_i Y)^2. \quad (6)$$

Again, σ_i should represent the fracture stress at the flaw instead of the maximum outer fibre stress in flexure tests.

The final form of the analysis is obtained by combining Equations 5 and 6:

$$t_f = [2/A(n-2)]\,(K_{Ic}/Y)^2\,\sigma_i^{n-2}/\sigma_a^n. \quad (7)$$

The slow crack growth parameters are computed from the time-to-failure results corresponding to various applied stress levels. The fact that failures may be initiated by different types of flaws in different specimens in some materials is generally ignored. For example, in SiC ceramics, both internal and surface voids have been found to initiate failure [19]. Different flaw shapes also lead to significant differences in Y-values, as illustrated by the fracture origins shown in Fig. 1. Both specimens failed under a maximum applied stress of 290 MPa with failure times of 2.5 and 90 h. Serious errors can be introduced if the variation of the Y-factor is not considered in Equation 7.

It is apparent from Equation 7 that t_f at a specified applied stress condition is not a constant but forms a range of values corresponding to the distribution of σ_i. In many instances [20, 21], the t_f appears to vary by more than an order of magnitude for the same σ_a (Fig. 2). Obviously, a least-square fit between $\log \sigma_a$ and $\log t_f$ to obtain n will introduce significant uncertainty in the estimates. Jakus *et al.* [11] partially circumvented this problem by introducing a Weibull statistical representation for σ_i and conducting a trivariate linear regression procedure to estimate the constants A and n. A subtle correction which should be considered is that the σ_i does not represent the total strength distribution but is truncated at a level corresponding to the proof-stress level equal to the applied stress σ_a.

Typically, stress rupture tests involve a very limited number of specimens at each stress level because of the duration of each experiment. As seen earlier, t_f forms a range with a minimum near zero to a maximum set by the applied stress level and the strength distribution. One can rank the failure times, t_f, and compare the equally

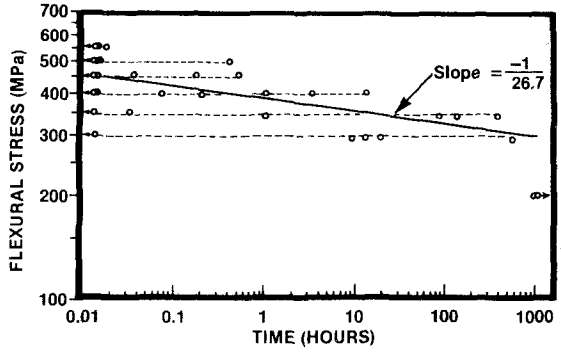

Figure 2 Static fatigue results on Hexoloy SA conducted at 1200° C (from [21]). The broken lines have been superimposed by the present authors.

probable t_f for different values of σ_a to obtain estimates of n. This procedure is impractical, however, as it will require the testing of an enormous number of specimens.

These criticisms are more valid for high-performance ceramics with less susceptibility to SCG, such as Hexoloy SA and fine-grain, reaction-sintered SiC, than for materials with a pronounced SCG tendency. We shall now address the limitations involved in the data reduction of dynamic fatigue results in ceramics.

4.2. Dynamic fatigue

The differential stressing rate or dynamic fatigue method is often preferred in obtaining slow crack growth parameters because the testing of a large statistically significant population can be completed in a much shorter time than with static stress rupture experiments. However, many of the same limitations discussed earlier exist in the analysis of the data.

From Equations 1 and 2 and by using $\dot{\sigma} = d\sigma/dt$:

$$\int_0^{\sigma_f} \sigma^n \, d\sigma = \int_{a_i}^{a_f} (K_{Ic}/Y)^n (\dot{\sigma}/A) \, da/a^{n/2}. \quad (8)$$

Again, assuming that the factor Y is invariant and that $a_f \gg a_i$, Equation 8 becomes:

$$\sigma_f^{n+1} = [\dot{\sigma}(n+1)/A(n-2)] (K_{Ic}/Y)^n a_i^{-(n-2)/2}. \quad (9)$$

The initial flaw size a_i can be expressed in terms of an "inert" strength σ_i, as in Equation 6. Then,

$$\sigma_f^{n+1} = [\dot{\sigma}(n+1)/A(n-2)] (K_{Ic}/Y)^2 \sigma_i^{n-2}. \quad (10)$$

In Equation 10, σ_f and the σ_i represent the statistical strength distribution obtained with and without the influence of slow crack growth, respectively. This relationship is also applied to specific ceramics without adequately verifying the validity of the assumptions used in deriving the above relations. For example:

(a) The contradicting assumptions mentioned earlier still occur: (i) that Y is invariant during growth and (ii) that $a_f \gg a_i$. The assumption that $a_f \gg a_i$ is more questionable in this case than in the static stress rupture analysis. The extent of crack growth is very limited due to short loading cycles, especially when fast loading rates and inherently SCG-resistant materials like Hexoloy SA are used. Correspondingly, the assumption that Y is invariant during growth becomes more tenable.

(b) The SCG parameters are again computed from a regression procedure which maintains that the Y-factor is constant for all specimens. This approach was shown as questionable for SiC ceramics in the stress rupture case.

(c) The data reduction using Equation 8 involves some incorrect procedures. The stressing rates are replaced by the cross-head rates of the testing machine on the premise that they are proportional. In actual testing, Young's moduli and specimen dimensions vary somewhat, thus introducing variation in the loading rate. More importantly, in flexure tests, the stressing rate depends on the locations of the flaw with respect to the tensile surface. The variable $\dot{\sigma}$ should represent the location-corrected stressing rate at the failure origin.

(d) One additional objection arises concerning the definition of the "inert" strength, σ_i. The use of strengths obtained at the fastest experimentally achievable loading rate as equal to σ_i, or leaving it as an unknown quantity corresponding to $\dot{\sigma} = \infty$ merits a firmer basis. The σ_f should tend towards a limiting value equal to σ_i, not influenced by SCG behaviour, as the $\dot{\sigma}$ increases. No such tendency is obvious in Equation 10. If the assumption concerning the flaw growth ($a_f \gg a_i$) were not made, then the resulting expression in the place of the Equation 10 could be written as:

$$\sigma_f^{n+1} = [\dot{\sigma}(n+1)/A(n-2)] (K_{Ic}/Y)(\sigma_i^{n-2} - \sigma_f^{n-2}), \quad (11)$$

or,

$$\frac{\sigma_i^{n-2} - \sigma_f^{n-2}}{\sigma_f^{n+1}} \propto \frac{1}{\dot{\sigma}}.$$

It is now apparent that σ_i is the limiting value for σ_f as $\dot{\sigma} \to \infty$. This discussion implies that there is no reasonable definition for σ_i if Equation 10 is employed. This discrepency, however, does not affect the estimate of the parameter n.

5. General discussion

In general, the analysis and data reduction procedures used for fatigue tests for ceramics suffer from the discrepencies discussed. Before these tests can be applied to any material to obtain the slow crack growth data, extensive fractography must be conducted to establish operative failure mechanism(s) and to evaluate the nature of fracture origins and crack growth patterns to ascertain whether the assumptions used in the analysis are valid. Several corrective measures, including deter-

mining the stressing rate and the fracture stress at the flaw, must be included in the data reduction.

A significant corrective measure would be to use a known crack instead of inherent flaws for monitoring SCG behaviour. A fracture mechanics configuration like a single-edge notched beam (SENB) can be used because the exact functional form of Y is known and can be included in the integration of Equation 3. This technique has been successfully applied by Henshall *et al.* [23], to static stress rupture experiments. The dynamic fatigue analysis for notched beams becomes somewhat complicated but can be evaluated by computer [24]. Preliminary computations on Hexoloy materials have been completed and results are encouraging. The use of SENB for crack growth studies may be criticized on the basis that the notch is much larger in size than the intrinsic flaws and hence the results may not be representative of the actual material behaviour.

A smaller, well-defined flaw can be produced by placing a Knoop indentation on the surface of the specimen. Such flaws have been utilized in stress rupture experiments [25]. In general, some growth pattern is seen along the flaw boundaries after stress rupture, unlike the situation with intrinsic flaws. The boundaries are not usually well defined and final flaw size, taken as the outermost boundary of the flaw, at the applied stress level corresponds to unrealistically large stress intensity factors [26]. The analysis for the fatigue tests employing controlled-flaw specimens is also more cumbersome and tedious than that for SENB tests.

Application of these fatigue tests to silicon carbide materials like Hexoloy SA and fine-grain, reaction-sintered SiC poses additional problems because the SCG effect is so small as to be difficult to evaluate with much precision. Thermomechanical strengthening has been observed in Hexoloy SA at 1200° C [27, 28] which further complicates the analysis. The estimates of n for this material at 1200° C vary widely (between 40 and 80) when static stress rupture tests are used. Numerically larger but negative values of n have been obtained when dynamic fatigue and proof test results are used [27]. These discrepancies are attributed to drawbacks in the analyses rather than to material variations.

Actual service environments involve lower stress levels and longer times to failure than the conditons evaluated in the tests. For materials resistant to slow crack growth, the errors in the data estimates from the test results are amplified considerably in the predicted lifetimes. It is important that, even after the necessary precautions have been taken to assure that data reduction procedures are valid, confidence bands be constructed for the curve fit. This procedure results in a range in the predicted lifetimes. It may then become necessary to increase the number of specimens and to test at stress levels closer to the service stress in order to improve the confidence in the predicted lifetimes.

The location of the fracture origin can also influence the growth behaviour because of exposure to the testing environment. In Hexoloy SA, surface-connected voids have shown more extensive growth than the interior pores after long-term loading experiments [20, 29]. From the foregoing discussion, it is clear that caution must be exercised when using results from fatigue tests for design purposes. Extensive fractography and other supporting information must be obtained before any practical significance can be assigned to the fatigue data.

It is also recognized that both A and n need to be evaluated to adequately represent the SCG behaviour of any material [19, 30]. Generally, the fatigue test results are used to estimate n only. The pre-exponential factor, A, as seen in the fatigue expression, can be obtained from the intercept. The uncertainty involved in A is very large considering that the intercept is a function of many factors, including n.

Frequently, comparison of slow crack growth tendency among different materials [30], environments [31, 32], and even testing techniques [33] are made based solely on the estimates for the parameter n. It is conceivable that a material with a higher n-value could fail before a material with a lower n-value when subjected to the same loading conditions as the failure time is also influenced by other factors, such as A, K_{Ic} and the initial flaw size and location (Equation 3). For a more accurate comparison, this equation may be applied, in a general sense, incorporating the uncertainties in the SCG parameters and K_{Ic} values as well as taking into account the nature of the failure-causing flaws. Although cumbersome, this procedure may be necessary for justifiable comparisons concerning the SCG behaviour.

Finally, it should be noted that some of the objections regarding the theoretical development and the application of these fatigue techniques to silicon carbides may not be significant for many

other ceramics exhibiting a well-defined flaw population and a pronounced SCG tendency.

6. Conclusions

The indirect techniques used to evaluate the SCG behaviour of ceramics have many inherent problems arising from both the analysis and the failure characteristics of the materials. There are often invalid and unsupported assumptions that are employed in the analysis, making these tests and the data reduction procedures not necessarily suitable for application to silicon carbide ceramics. Some modified procedures such as the use of notched beams can be developed to reduce the ambiguity of the results from these testing techniques. It is suggested that direct velocity measurements or other indirect techniques should be pursued to obtain these important design parameters more accurately for silicon carbides.

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Received 24 June
and accepted 28 September 1981