THE MEASUREMENT OF K_{Ic} AND SUBCRITICAL CRACK PROPAGATION RATES IN HOT PRESSED SiC AND Si_3N_4

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INTRODUCTION

In recent years it has become apparent that further significant increases in the efficiency of gas turbines, through increases in gas inlet temperature, will be severely limited by the properties of the superalloys used. Thus, ceramics are now being considered for components in the hot gas stream of advanced turbines. The most promising materials are hot pressed SiC and hot pressed Si_3N_4.

A major design problem is in accurately predicting failure times which requires a knowledge of both the mechanisms and mechanics of fracture. It has been shown [1,2] that the crack velocity v is a unique function of the stress intensity, and therefore failure times can be calculated from the v-K_I relationship. The present paper describes and compares two methods for obtaining the appropriate fracture mechanics data for ceramics. Values of K_{Ic} and v-K_I curves determined from tests in air will be presented for hot pressed SiC (NC203)† and hot pressed Si_3N_4 (HS130)†, and the applicability and usefulness of the two techniques will be discussed.

MEASUREMENT TECHNIQUES

Double Torsion (DT)

Evans [1] has developed the Double Torsion test for the measurement of K_{Ic} and crack velocities in ceramics. This method relies upon the change in compliance of a constant K_I fracture toughness specimen, i.e. $K_I \neq f(a)$ with change in crack length. The three ways of performing the test are:-
(i) When a constant displacement rate is applied, then the crack grows at a constant load, whence the growth rate is given by [1,3]

$$v = \frac{H\ B^3 \mu_s (1 - 5B/4H)}{3PH_m^2} \cdot \frac{dy}{dt} \qquad (1)$$

where y is the loading pin displacement, μ_s is the shear modulus, and the other symbols are defined in Figure 1. Thus using commercially available equipment, for a given specimen size it is possible to obtain crack velocities over approximately three orders of magnitude. This method did

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not prove satisfactory in the present series of tests since it was difficult to obtain equilibrium prior to fracture.

(ii) Also from (1), when a constant load is applied, the displacement rate can be measured and hence the crack growth rates calculated. The problem encountered here was that at high loads fracture occurred too rapidly, and at lower loads, prohibitively long test times were required to obtain a measurable crack extension on the fracture surface.

(iii) For specimens subjected to a fixed displacement [1]

$$v \approx \frac{-a_o P_o}{P_i^2} \cdot \left(\frac{dP}{dt}\right)_{t=t_i} \qquad (2)$$

where P_o is the initial load, and P_i is the load at time $t=t_i$. Therefore for a fixed displacement, the relaxation curve can be recorded and the crack growth rates calculated. Using the present experimental apparatus of a commercial testing machine, and point and line loading at high temperature, it was found that the machine relaxation was a significant fraction of the total relaxation. Hence, P_o was initially fairly low, and a number of relaxations were performed, P_o being increased slightly each time. It was then possible to analyse this relaxation data to determine the cycle on which crack growth started. The machine relaxation was taken as the curve prior to the commencement of crack growth. To overcome random fluctuations and provide flexibility of calculation, it is necessary to represent the machine relaxation curve mathematically. Three equations were used for curve fitting, i.e.:-

$$P_i = \sum_{j=1}^{j=n} C_j t_i^{(j-1)} \qquad C_j = \text{constants, } j = \text{integer}$$

$$P_i = \exp(-t_i) \sum_{j=1}^{j=n} C_j' t_i^{(j-1)} \qquad C_j' = \text{constants, } j = \text{integer}$$

$$P_i = P_c - f\, t_i^j \qquad P_c = \text{constant} \approx P_o,\ j \neq \text{integer}$$

$$f = \text{constant}$$

The full details are given elsewhere [3], where it was shown that the latter equation gave the best fit to the data, particularly since it is required to calculate dP/dt. The amount of crack growth occurring during one relaxation can be obtained by intergration of (2) i.e.:-

$$\Delta a = a_o \left[P_o/P_e - 1 \right]$$

where P_e = final load. This value of Δa correlated well with those measured on the fracture surface. The crack front was very nearly perpendicular to the direction of crack motion during propagation, so that no correction for crack front ellipticity was required, as is the case with other materials [1].

Single Edge Notched Beam (SENB)

An alternative method for measuring fracture mechanics parameters is to use SENB loaded in three or four point bending. Both K_{Ic} and time to failure curves for dead weight loading, i.e. delayed fracture have been

determined for HS130 [4], using the specimen and test configuration shown in Figure 2. A preliminary study of this test method had shown that pre-cracking was not required [5]. The results of delayed fracture tests have recently been re-analysed to yield crack growth data i.e. v-K_I curves [6]. This requires that t_f, a_o, and final crack length a be measured and a relationship between v and K_I be assumed, e.g. $v = v_o K_I^\beta$ where v_o and β are empirical constants. They are related by the equation

$$\log t_f = \log \left\{ \frac{1}{\sigma^\beta} \int_{a_o}^{a_c} \frac{da}{Y^\beta a^{\beta/2}} \right\} - \log v_o$$

where $Y = [3.86 - 6.15(a/W) + 21.7(a/W)^2]^{1/2}$ [6], which gives best fit values for β and v_o.

RESULTS

K_{Ic} Measurements

Figure 3 shows the temperature variation of K_{Ic} for hot pressed SiC NC203 between 300K and 1773K and for hot pressed Si_3N_4 HS130 between 300K and 1623K. The curve for Si_3N_4 fits data points determined independently using the SENB [4] and DT [7] methods. K_{Ic} for hot pressed SiC was measured using SENB between 300K and 1373K. Above this temperature lightly sidegrooved DT specimens [1,3,8] were used, Figure 1. Below 1000K there is no variation in the critical stress intensity factor which is 6.1 MPa·m$^{1/2}$. K_{Ic} then decreases to 4.0 MPa·m$^{1/2}$ at 1773K, with the rate of decrease being lower at the higher temperatures. The results of Evans and Lange [9] are also shown and are qualitatively very similar to ours. The numerical difference presumably arises from a variation in the starting materials, since the Double Torsion test was used for some of the results in both cases. A detailed material comparison is not possible, since reference [9] does not include sufficient information.

Crack Velocity Measurements

Figure 4 shows v-K_I curves for hot pressed SiC tested at 1573K. The DT data are from four relaxation tests on two specimens and the delayed fracture data are derived from six specimens. The two curves have similar gradients, but there is a displacement of approximately 0.5 MPa·m$^{1/2}$ i.e. 15%, in the K_I value required to give equivalent crack velocities, or alternatively a displacement of an order of magnitude in the crack growth rates for a given K_I, which is within the combined experimental error of the crack velocity ($\sim\pm 2$ times) and K_I ($\sim\pm 0.3$ MPa m$^{1/2}$) measurements.

In Figure 5, crack velocity data are presented for SiC and Si_3N_4 at 1273K, determined in each case from delayed fracture measurements. Comparative data using DT could not be obtained at these lower temperatures due to practical difficulties inherent in this method under conditions of limited slow crack growth.

DISCUSSION

The results presented above indicate that equivalent fracture mechanics data can be obtained from SENB and DT tests. K_{Ic} values determined using the two techniques, for a well characterized ceramic, HS130, are the same within experimental error. For determinations of v-K_I curves, the constant displacement DT test and the delayed fracture analysis yield results which are statistically in agreement, indicating that the test method does not significantly affect the v-K_I curves.

The advantages of the DT method are:-
(a) crack velocity data can be directly calculated,
(b) the total test time required to determine the v-K_I curve is relatively short.
Its disadvantages are:-
(a) it can only be used for materials which have limited plastic displacement,
(b) it is difficult to measure low crack velocities,
(c) in general specimens must be sidegrooved, which can affect the fracture mode in the region of such a machined defect [4], and also the crack tends to grow with an elliptically shaped front, both of which might affect the v-K_I curve,
(d) sophisticated testing equipment is required.

On the other hand, the advantages of the delayed fracture analysis are:-
(a) it can be used to measure low crack velocity data,
(b) it is a more sensitive measure of crack growth for large values of β,
(c) experimentation is simple,
(d) tests can be performed in aggressive environments,
while its disadvantages are:-
(a) a functional relationship between v and K_I must be assumed,
(b) care must be taken with the analysis to check for delayed initiation and departure from a single functional relationship,
(c) it is relatively time consuming.

Clearly these two methods for determining v-K_I curves are generally complementary. In some cases however, specimen size and environmental conditions can limit the choice of method to the SENB. For example, only the delayed fracture analysis could be applied [6] to time to failure data for SiC and Si_3N_4 tested in simulated gas turbine environments [10]. Where applicable therefore, it is expected that the latter technique will be particularly useful in obtaining engineering data, and the former will be more appropriate for research work where the measurement of limited amounts of crack growth is not required. Caution must be exercised though, since further work is still required to prove that the equivalence between the DT and SENB techniques is generally valid.

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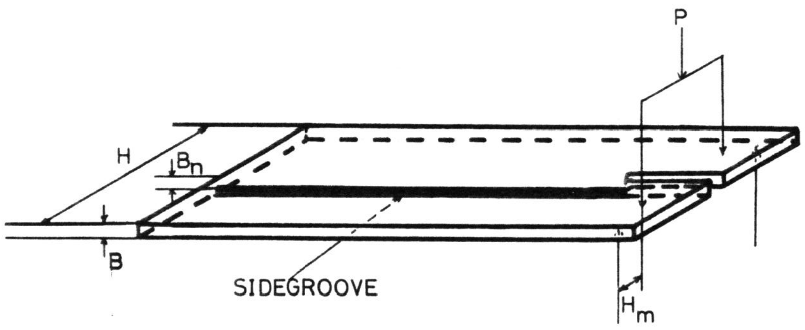

Figure 1 The Double Torsion Specimen

B = specimen thickness = 1 mm
B_n = sidegrooved specimen thickness = 0.9 mm
H = specimen width = 15 mm
H_m = bending arm = 4 mm

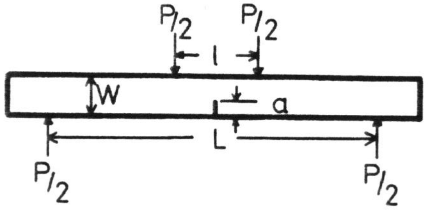

Figure 2 The Single Edge Notched Beam Specimen

B = specimen thickness = 2.5 mm
W = specimen depth = 5 mm
a = crack length = 2 mm
ℓ = minor span length = 10 mm
L = major span length = 40 mm

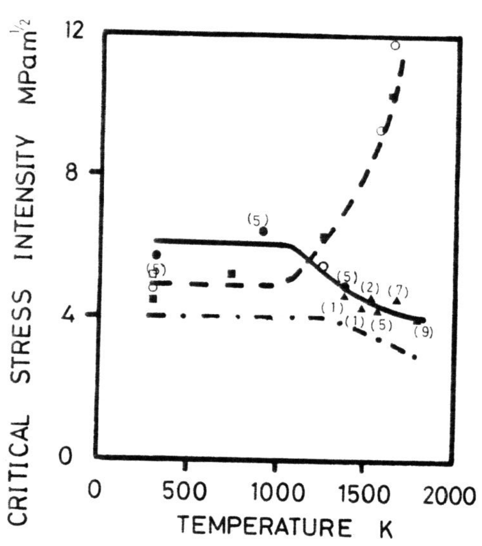

Figure 3 The Temperature Variation of K_{IC} for Hot Pressed SiC and Si_3N_4

——— Hot Pressed SiC; ● SENB (the numbers in parentheses are ▲ DT the number of specimens tested)

—·—·— Hot Pressed SiC; Data from Reference [9]

- - - - Hot Pressed Si_3N_4; Data from Reference [4] {■ SENB ☐ DT and Reference [7] o DT

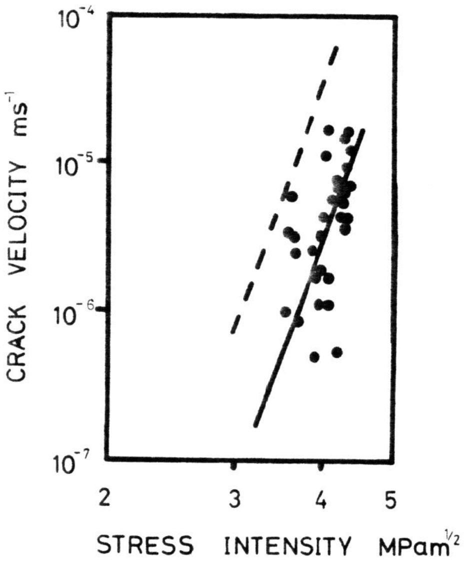

Figure 4 The Relationship Between Crack Velocity and Stress Intensity for Hot Pressed SiC at 1573K. Least Squares Curve Fitting to the Equation $v = v_0 K_I^{\beta}$ gives $v_0 = 5 \times 10^{-14}$ and $\beta = 13$ with Correlation Coefficient $r = 0.81$ for the Double Torsion, and $v_0 = 2 \times 10^{-13}$, $\beta = 14$ and $r = 0.96$ for the Delayed Fracture Analysis

——— Double Torsion; ● Individual Data Points ;

----- Delayed Fracture Analysis

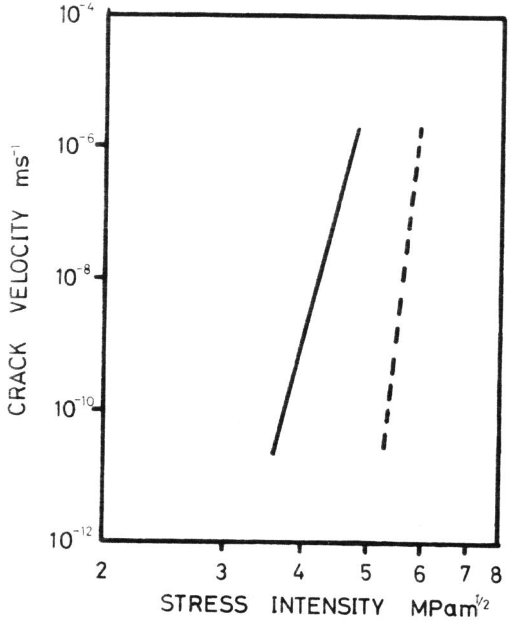

Figure 5 v-K_I Curves for Hot Pressed SiC and Si_3N_4 Obtained from Delayed Fracture Tests at 1273K

——— hot pressed SiC; $v_o = 9 \times 10^{-34}$, $\beta = 40$

----- hot pressed Si_3N_4; $v_o = 2 \times 10^{-76}$, $\beta = 90$