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Effect of Strain Rate on the Fracture Toughness of Silicon Nitride at 1400°C

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ABSTRACT

The critical stress intensity factor for fracture, K_{IC}, was measured in Si_3N_4 at 1400°C as a function of strain rate. It was found that K_{IC} increased with decreasing rates of bending of notched bars. This effect could be approximately predicted using a time dependent plastic zone correction to K_{IC}.

1. INTRODUCTION

At very high temperatures, ceramics can deform extensively by time dependent deformation processes like creep. This permanent deformation is stress and time dependent and is not predicted by elasticity theory. Whether or not it is appropriate to apply linear elastic fracture mechanics to creeping materials depends mainly on the extent of creep deformation relative to certain specimen dimensions. With this in mind, the strain rate dependence of the fracture toughness of silicon nitride was investigated at 1400°C.

Work on silicon nitride,[1] aluminum oxide,[2] and silicon carbide[3] have shown that at high temperatures and at moderate to slow strain rates, the fracture toughness is above the room temperature value by a factor of two or more. Under the same conditions, these materials are observed to creep.[3,4] It is believed that it is this creep deformation which is mainly responsible for the increased toughness. Since the stress at which creep deformation is observed is highly rate sensitive, there should be a correspondingly strong dependence of the high temperature K_{IC} on strain rate or loading rate. Figure 1 shows the toughness of silicon nitride measured by various researchers as a function of temperature. Two groups find a strong increase in K_{IC} with temperature, and two groups find no increase or even a slight decrease. The fracture was in all cases a fast fracture; that is, the specimen broke in two under its own elastic strain energy and the load dropped immediately to zero, faster than the loading or straining system could follow. The main difference in these results is the rate at which the tests were carried out. In impact loading studies with displacement rates greater than 100 mm/sec, no enhanced toughness was observed; whereas, increased toughness

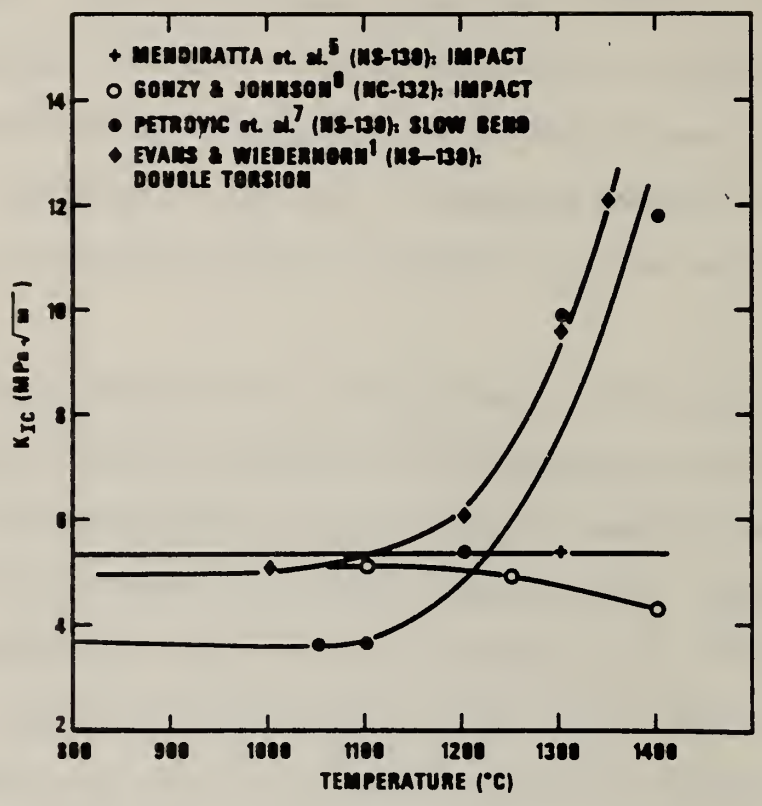

Fig. 1. Effect of Temperature and Strain Rate on the Fracture Toughness of Silicon Nitride as Measured by Various Researchers.

was observed for slow bend tests or double torsion tests carried out at a displacement rate of 1 mm/sec or less. Such a rate effect is observed in metals at much lower temperatures[8] and is generally ascribed to the rate dependence of the yield stress. In such cases, a plasticity correction can be made to K_{IC} to predict with some success the effect of rate on toughness.[9] In the present paper, the effect of strain rate on both the fracture toughness and the stress for the onset of creep is measured for silicon nitride at 1400°C. The creep stress data is used to predict the influence of strain rate on toughness.

2. EXPERIMENTAL PROCEDURE

Bars having a rectangular cross-section were cut from a billet of NCX34, yttria doped silicon nitride. A narrow notch was introduced using a 1/2 mm thick diamond slitting wheel. Two sizes of bars were tested having the approximate dimensions listed in Table (1). The tests were performed in four point bending (See Figure 2) using a rig made of silicon carbide. The major span was 40 mm and the minor span was 10 mm. All tests were performed in air in furnaces which were brought to temperature (1400°C) in about 3 hours. The furnaces had feed-throughs in the top and bottom for silicon carbide push rods. One push rod was attached to a load cell and the other to the cross-head of a displacement-rate controlled testing machine. The cross-head was driven at rates from 1.25 to 0.005 mm/minute.

In general, a subcritical crack initiated stably from the slit and grew a few millimeters before fast fracture occurred. This avoided ambiguities associated with the sharpness of the slit and eliminated the need to precrack or fatigue crack the specimen. For fracture tests at rapid displacement rates or at room temperature, where little or no subcritical crack growth occurs, relatively long pre-cracks were sometimes grown in by bending at 1400°C with a displacement rate of 0.0125 mm/min. After producing these cracks, the specimens were kept at temperature unloaded for at least one hour before cooling or testing.

Table 1

Nominal Specimen Dimensions (mm)

	Large Specimen	Small Specimen
Length, ℓ	52	52
Width, W	10	5
Thickness, t	5	4.5
Slit depth, c	3 and 5	1.5

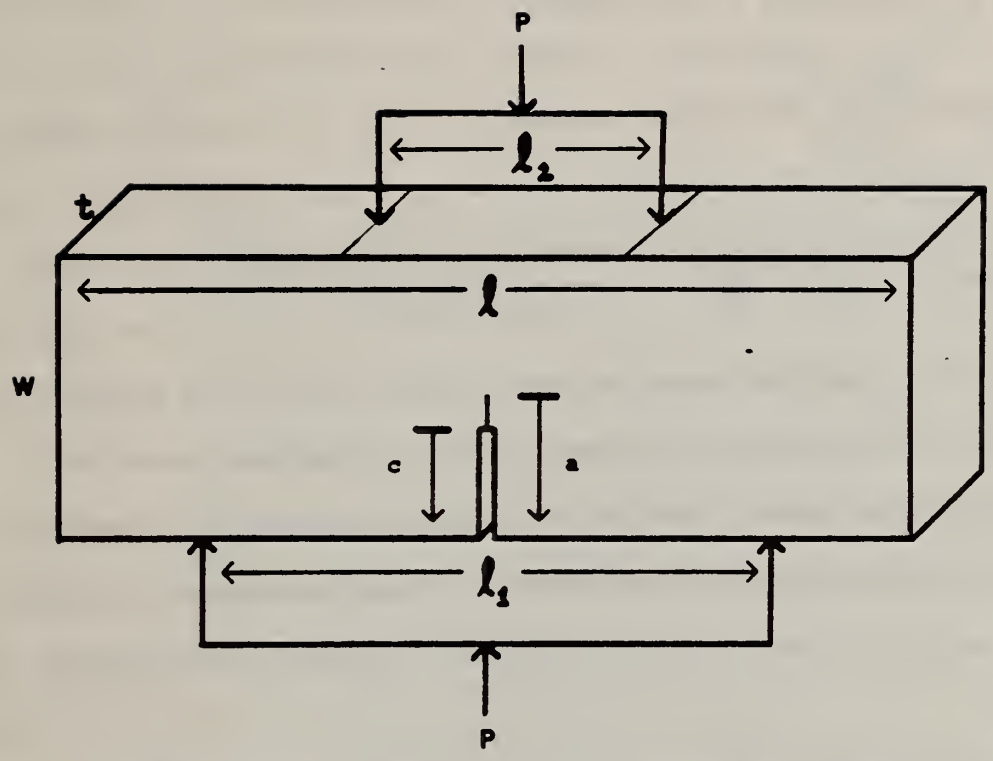

Fig. 2. Schematic of Four-Point Notched Bend Test.

After fracture, the crack length(a) was determined as the sum of slit depth plus the extension resulting from the subcritical crack growth. This, together with the load at the onset of fast fracture (P_f) was used to calculate K_{IC} according to the formula[10]

$$K_{IC} = \frac{3P_f(\ell_1-\ell_2)}{2\,B\,W^2}\sqrt{\pi\ a}\qquad F(a/W) \tag{1}$$

where

$$F(a/W) = \frac{\sqrt{\frac{2W}{\pi a}\tan\frac{\pi a}{2W}}\left(0.923 + 0.199\ (1-\sin\frac{\pi a}{2W})^4\right)}{\cos\ (\pi a/2W)}$$

and where ℓ_1 and ℓ_2 are the major and minor spans of the four point bending rig, B is the specimen thickness, and W is the specimen width. This formula was used rather than the more common polynomial[11] because it is purported to be accurate for any crack length/specimen width (a/W) ratio. The polynomial expression is only good for (a/W) less than or equal to 0.6.

Additionally, two other types of fracture tests were performed. One test consisted of slowly growing a crack at a displacement rate of 0.0125 mm/min., waiting a time (Δt), and then fracturing at a faster displacement rate of 1.25 mm/min. This test was performed for various Δt's. The other type of test consisted of growing in a crack at 0.0125 mm/min and then cooling under load to room temperature and fracturing at 0.05 mm/min.

To describe the influence of displacement rate on the fracture behavior, the creep behavior of this particular material was needed. We made the assumption here that the creep behavior would correlate well with the stresses at which the load-deflection curves became non-linear. These proportional limit stresses were obtained for displacement rates from 0.125 to 0.0125 mm/min. Small cubes (5 mm on a side) were cut

from some of the fracture bars. These were compressed between the silicon carbide push rods. Cross-head displacements agreed with dimensional changes measured on the specimens after the test. No attempt was made to reach steady-state creep. It is known that creep behavior of ceramics is very different in tension and compression.[4] However, the primary stage of creep is probably dominated by some viscous component like grain boundary sliding rather than a cavitational component (which dominates tertiary creep) in the material's mechanical behavior.[12] This viscous deformation should not depend strongly on the sign of the loading.

To investigate the extent of creep deformation in the fracture process, a fine array of Vicker's indentations was made on the side of a flexural specimen (Fig. 3) which was then loaded at a displacement rate of 0.0125 mm/min. The positions of the indentations were measured before and after with a precision of 0.0075 mm.

3. RESULTS

The K_{IC} measured at room temperature for a specimen which was pre-cracked at 1400°C was 8.3 MPa $\sqrt{m}$. This high value is discussed in the next section. The results of measuring K_{IC} at 1400°C as a function of displacement rate are shown in Fig. 4. This shows an increase in toughness from about the room temperature value to more than 18 MPa $\sqrt{m}$ at the lowest displacement rate of 0.0125 mm/min. Evidence that this toughening is due to a time dependent process, is shown in Fig. 5. A specimen was loaded slowly until a crack started growing slowly. The specimen was unloaded rapidly and then reloaded at 1.25 mm/min. If the reloading took place immediately, the toughness value is close to that obtained by breaking the specimen at the slow rate. If the specimen was allowed to remain unloaded for several minutes before reloading, the toughness fell. The maximum effect of delayed reloading was obtained in about 60 minutes after which time the toughness no longer decreased. This effect

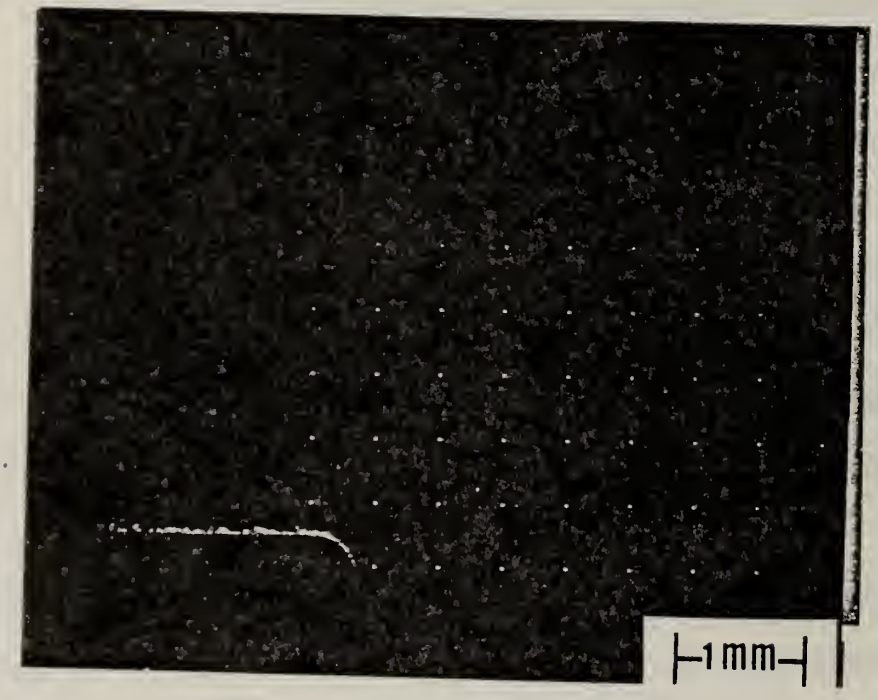

Fig. 3. Array of Vicker's Indentations on Side of Flexural Specimen.

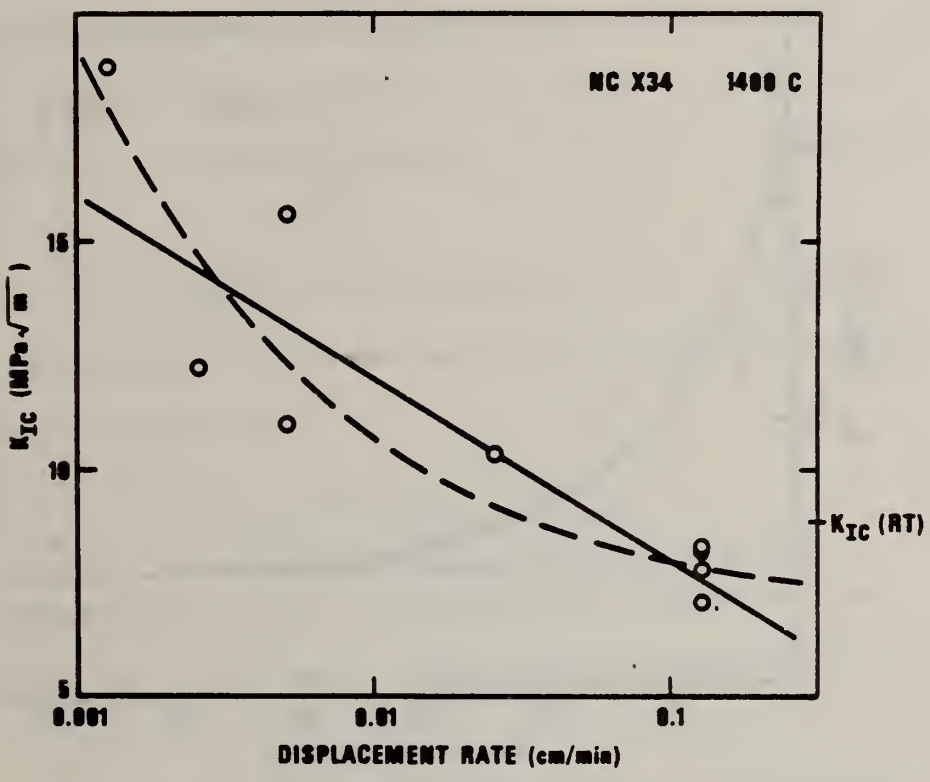

Fig. 4. Fracture Toughness of NCX34 as a Function of Cross-Head Displacement Rate at 1400°C.

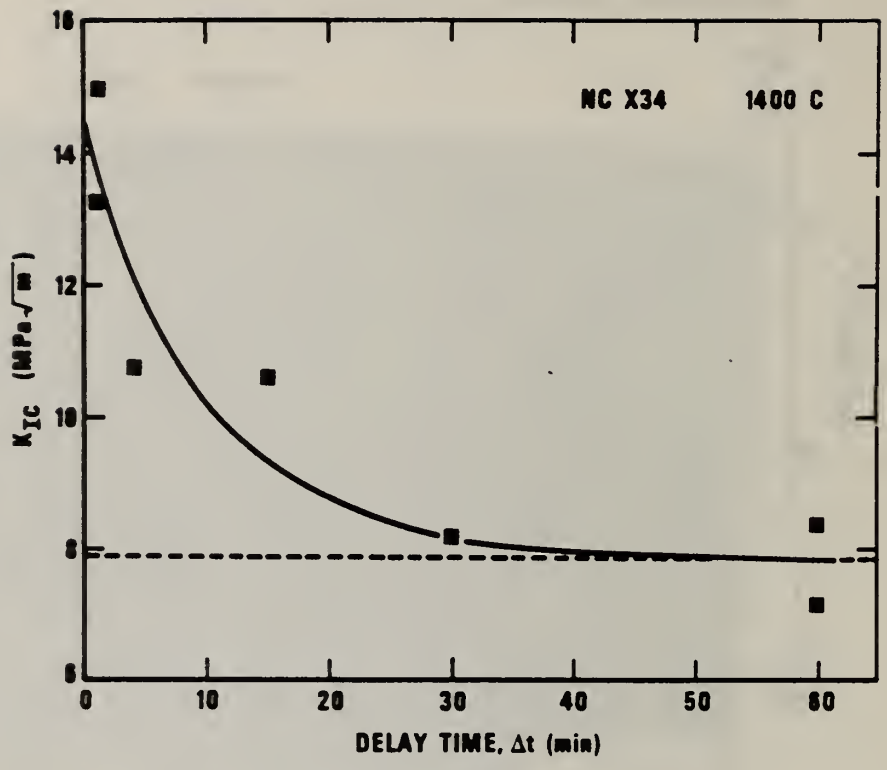

Fig. 5. Decrease in High Temperature Toughness with Time after High Temperature Pre-cracking.

was obtained in specimens which had been heated to 1400°C for several hours as well as in the as-ground specimens.

In an attempt to "freeze-in" the cause of the toughening, a specimen was loaded until a crack began to grow subcritically and then was cooled under a slightly reduced load to room temperature. This resulted in a room temperature K_{IC} of 9.9 MPa $\sqrt{m}$ which is a 20% increase in toughness. Tests like this on alumina have obtained similar increments in room temperature toughness.[2]

The results of the creep tests are shown in Fig. 6.* From this data, the relation between the strain rate and the stress at the onset of creep may be represented by

$$\dot{\varepsilon} = \sigma_{pl}^{4.24}/(2.04 \text{ GPa sec}) \qquad (2)$$

where the pre-exponential term and the power were determined from a least squares analysis of the data. The correlation coefficient was 0.96.

The extent of permanent creep deformation was determined approximately from the flexural specimens on whose side faces an array of indentations had been made. Within the resolution of the technique (0.075 mm in 10 mm), the permanent deformation was confined to a 1 to 2 mm region around the crack.

4. DISCUSSION

The room temperature value for K_{IC}, 8.3 MPa $\sqrt{m}$, is higher than that usually reported for NCX34.[13] This could be due to test technique, since four point bending usually gives higher toughnesses than double torsion tests, and slow creep cracks are probably blunter than indentation cracks. Alternatively, this particular batch of NCX34 might be especially

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*The least squares line has been drawn over the range of strain rates that occur in the bending tests.

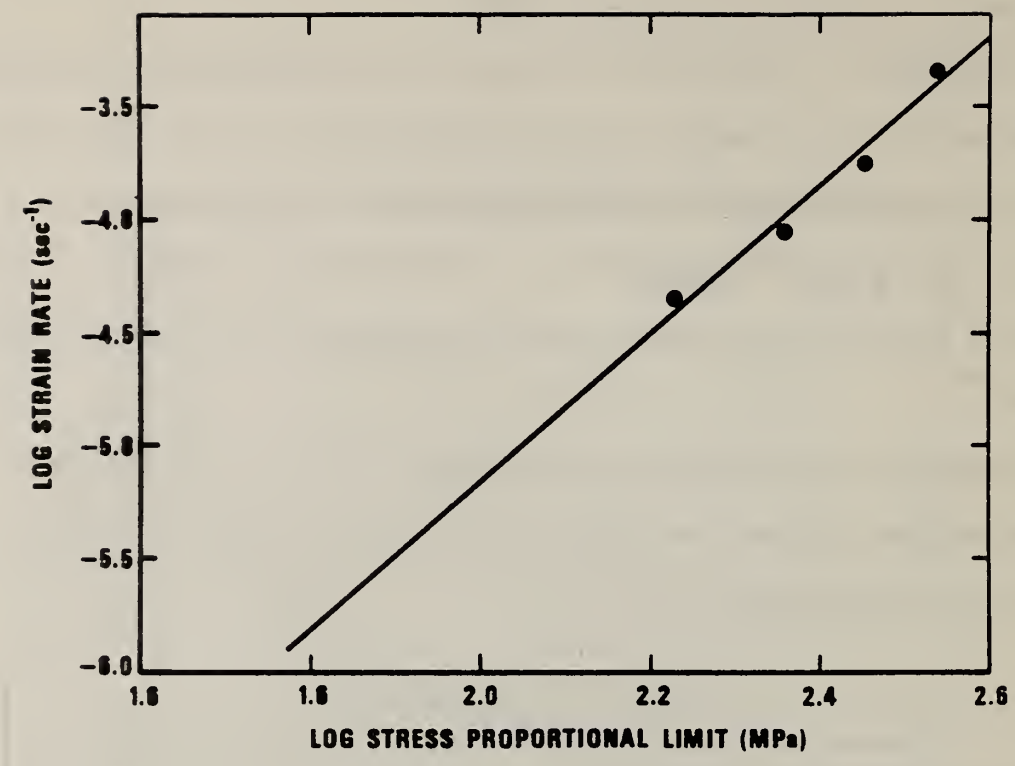

Fig. 6. Proportional Limit Yield Stress as a Function of Strain Rate in NCX34 at 1400°C.

strong as is suggested by other strength studies made on this billet.[14] In any case, regardless of the exact values reported here, the trends with loading rate or delay time should represent general trends in hot-pressed silicon nitrides.

Fig. 4 shows that there is a strong variation in toughness with displacement rate. This raises a serious question as to the existence of a unique fracture toughness which reflects a material's microstructural resistance to fracture. If there is no unique value, then K_{IC} must be measured for each strain rate in question. However, we feel that the time dependent plastic response of this material at 1400°C is responsible for lessening the intensification of the stress near the crack tip. Therefore, the K_{IC} measured at the highest rate will most nearly approach the purely elastic case and ought to be the correct value for comparison of a material's fracture properties. The average K_{IC} for four specimens measured at 1.25 mm/min (i.e. the fastest rate) and 1400°C is $7.9 \pm .6$ MPa $\sqrt{m}$. This value is slightly below the room temperature value in accord with the trend observed in impact studies on NC132. While 1.25 mm/min is not an impact test, it is very fast for a material with good creep resistance like NCX34. It is interesting to note that there was no appreciable difference in toughness between pre-cracked specimens and as-slit specimens.

The K_{IC} measured at lower rates represents some mixture of a material's fracture and deformation behavior. It is probably the more important engineering quantity to specify since it is the effective toughness that will be observed at a given, low strain rate.

Knowing both K_{IC} for the purely elastic case (rapid fracture with little deformation) and the deformation behavior, allows one to predict the apparent K_{IC} for the case of fracture preceded by deformation. One of the simplest

adjustments is to recalculate K_{IC} assuming that the crack is longer than the measured crack by an amount equal to the extent of the deformation zone. Such an assumption works well for metals. It may be particularly valid for silicon nitride in which cavitation is a major component in the tensile creep deformation.[12]

The radius of the deformation zone may be derived from the stress distribution ahead of the crack tip, i.e.

$$\sigma = K/\sqrt{2\pi r} \tag{3}$$

by inserting the appropriate stress and solving for distance ahead of the crack tip(r):

$$r_{pl} = \frac{1}{2\pi} \left(\frac{K_{IC}}{\sigma_{pl}} \right)^2 \tag{4}$$

Note that this analysis relies on the assumption that the stress field outside of the small plastic zone is described by K. The above distance (r_{pl}) is then added to the crack length used in the highest strain rate measurement of K_{IC} (a=5.02 mm). K_{IC} is then recalculated from Eqn. 1 using the new crack length and the original load at fracture for the high rate K_{IC} (471 N). This is the predicted K_{IC} corresponding to the strain rate at which σ_{pl} was measured.

To carry this procedure out for the present data, the strain rate was determined from the displacement rate in a bend test.[15] The relation used is:[16]

$$\dot{\varepsilon}_{max} = \frac{6\ (W-a)}{(\ell_2-\ell_1)\ (\ell_2+2\ell_1)}\ \dot{\Delta} \tag{5}$$

where $\dot{\varepsilon}_{max}$ is the strain rate in the outer fiber, $\dot{\Delta}$ is the displacement rate, ℓ_2 and ℓ_1 are the major and minor spans, and (W-a) is the ligament width. The assumption is made here that the strain rate at the crack tip

is equal to the outer fiber strain rate in an elastic bar having a width equal to the remaining ligament width. Since most of the bar is deformed elastically, Eqn. 5 seems more appropriate than an equation which assumes a creeping or perfectly plastic material. At longer times than were studied here, however, creep deformation occurs on a scale comparable to the specimen's dimensions and other relations would have to be used. In this regime, however, the validity of measuring K_{IC} would also be questionable.

In Table 2, the strain rates at which the tests were carried out are listed in decreasing order in the first column. In the second column, the corresponding yield stresses calculated from Eqn (5) are shown. These yield stresses are then used (Eqn. (4)) to determine the plastic zone size. Finally the corrected K_{IC} is calculated from the highest rate K_{IC} by altering the effective crack length by an amount equal to the extent of the deformation. These values may be compared with the experimentally determined ones in the last column. This comparison is shown graphically in Fig. (7). The agreement between the predicted and experimental toughness is as good as the scatter between the experimental values. The predicted line tends to be below the experimentally determined points. This could be due to the measurement of the creep stress in compression rather than in tension. Creep should occur more readily at lower stresses in tension. This would tend to shift the predicted line upward and give better agreement.

The effect of the deformation or plastic zone on the toughness decreases with time after unloading as shown in Fig. (5). It takes about 1 hour at 1400°C. to recover fully. The effect can be frozen in by cooling to room temperature under load. This is additional evidence that it is the time dependent deformation which is responsible for increasing the apparent K_{IC} by reducing the crack tip stress field intensification. The delay time

Table 2

Summary of Results

Strain Rate Sec^{-1}	Proportional Limit MPa	Plastic Radius mm	K_{IC}(corr.) $MPa(m^{1/2})$	K_{IC}(meas.) $MPa(m^{1/2})$
$3.51x10^{-4}$	334	.089	8.10	7.88
$3.49x10^{-4}$	333	.089	8.10	8.31
$3.40x10^{-4}$	331	.090	8.10	8.39
$1.76x10^{-4}$	270	.136	8.22	7.16
$3.29x10^{-5}$	162	.377	8.90	10.41
$1.05x10^{-5}$	114	.760	10.17	15.69
$7.34x10^{-6}$	102	.950	10.91	11.07
$3.25x10^{-6}$	80	1.544	13.71	12.31
$2.82x10^{-6}$	76	1.711	14.98	18.87

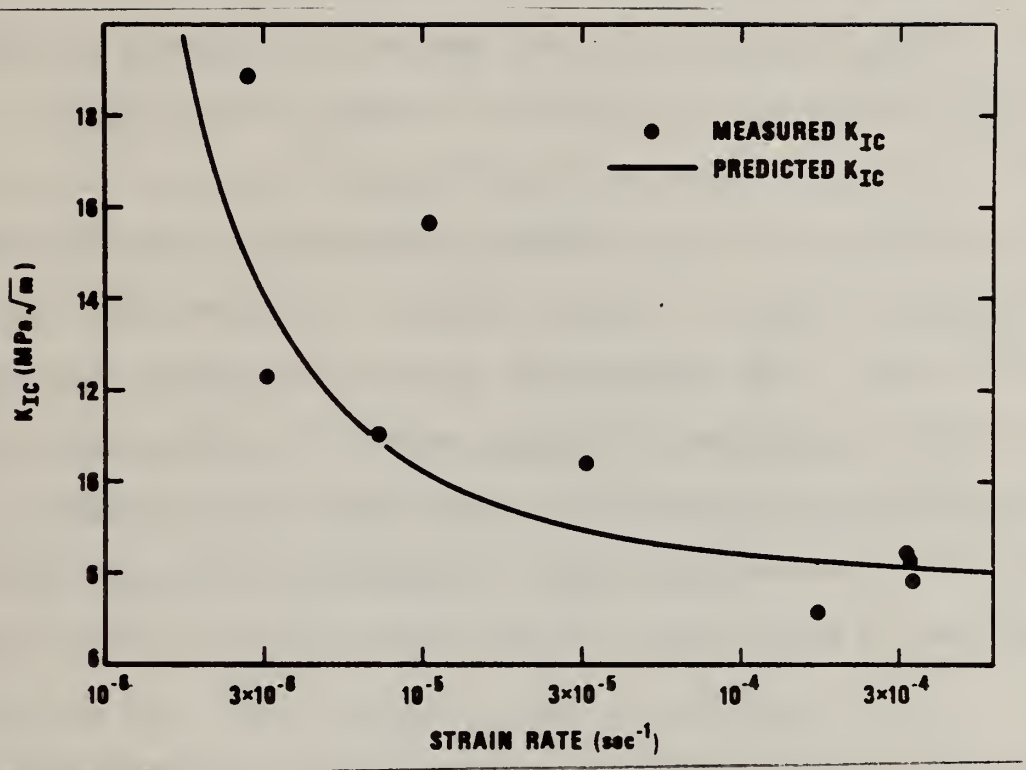

Fig. 7. Measured and Predicted K_{IC} as a Function of Crack Tip Strain Rate.

effect could be explained as follows: on loading, a zone of tensile creep deformation develops around the crack tip. On unloading, the predominantly elastic specimen puts the deformed region into compression and tends to reverse the deformation that has occurred at the crack tip. The time required to return to the low toughness state depends on the creep rate of the material and how fast the stress falls off as the deformation is reversed (that is, the elastic modulus).

It is possible to rule out oxidation and microstructural instability,[17] to some extent, as causes of the above effects. Oxidation could either blunt or sharpen the crack. If it sharpened the crack, then toughness should go down the longer the specimen was held at temperature. This might explain the delay time reloading tests, but it cannot explain the increase in toughness with decreasing strain rate. Alternatively, if oxidation blunted the crack, then it could explain the rate effect but not the delay time effect.

With regard to the microstructural instability, a general degradation of properties in NCX34 is frequently noted after thirty minutes at 1400°C.[14] This has been associated with a volume change in the grain boundary phase causing cracking. With longer exposures, there is only a slow change in properties after the first hour. Since the specimens used here were heated up slowly and equilibrated at temperature before testing, they spent more than an hour at a temperature high enough to reach a relatively stable state. To verify that the microstructure was indeed stabilized, some specimens were kept in the furnace while other tests were run. Later, these specimens themselves were tested. Although these specimens had been at 1400°C for

several hours, no appreciable change in fracture properties was noted when they were compared to the specimens that were tested at room temperature or heated and tested immediately. Also, the microstructural instability cannot simultaneously explain the delay time effect and the strain rate effect.

It is important to note that the range of strain rates over which the plastic zone correction is applicable is limited. At very high strain rates, the zone is so small that it makes a negligible correction to the K_{IC} and the toughness there should be independent of rate. At very low strain rates, the creep deformation extends throughout the whole specimen so that the correction would give effective crack lengths equal to or greater than the specimen width. In such a case, K_I no longer describes the crack tip stress field and it might be better to evaluate J_{IC} in order to predict the K_{IC} that would be obtained in larger bodies.

5. CONCLUSION

At temperatures where creep becomes an important component in the deformation of silicon nitride, the apparent K_{IC} depends on the rate at which the fracture test is carried out. This toughening is due to the relaxation of crack tip stress intensification by high temperature, time dependent plasticity rather than oxidation or microstructural effects. It has been shown here that, when the extent of this deformation is small, its effect on the toughness may be estimated by making a plastic zone correction to the crack length.

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The critical stress intensity factor for fracture, K_{IC}, was measured in Si_3N_4 at 1400°C as a function of strain rate. It was found that K_{IC} increased with decreasing rates of bending of notched bars. This effect could be approximately predicted using a time dependent plastic zone correction to K_{IC}.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

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