# STRENGTH CHARACTERIZATION AND NATURE OF CRACK PROPAGATION IN SINTERED ALPHA SILICON CARBIDE

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ABSTRACT

A detailed study characterizing the strength and nature of crack propagation has been made in sintered alpha silicon carbide. Fracture phenomenology was studied fractographically using flexural (bend-bar type) specimens in the as machined (uncracked) and precracked condition and to a limited extent using uniaxial tensile specimens. The flexural strength (4-point bend) was found to be independent of temperature (20° to 1400°C). Failure occurred in a brittle, fast fracture (catastrophic) manner. In contrast, uniaxial tensile tests and flexural precracked specimens tested at 1200°C in air showed limited presence of slow crack growth. The extent of slow crack growth increased with increasing temperature. The mode of crack propagation during fast fracture and slow crack growth are discussed.

KEYWORDS

Flexural strength evaluation, crack propagation, slow crack growth, failure initiation sites.

INTRODUCTION

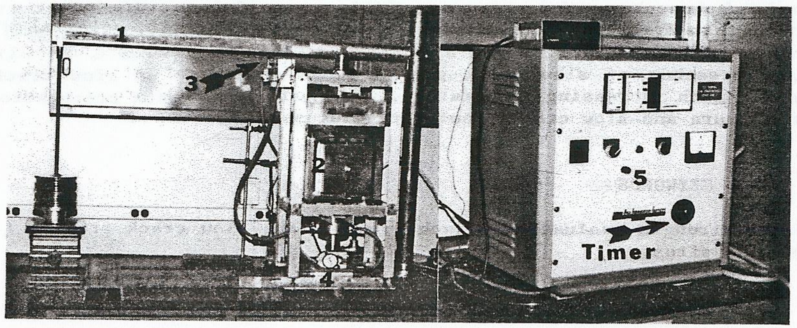
Sintered α-SiC is currently being investigated for use as structural components for gas turbines and diesel engines because of good oxidation resistance and low coefficient of thermal expansion up to about 1000°C. Like many other ceramics, sintered α-SiC is generally regarded as a brittle material with little tolerance for the incipient flaws (primarily porosity) that are present due to processing and fabrication. It is these random flaws that control the mechanical properties. In this paper, we report the results of a series of simple mechanical tests performed on as machined and precracked flexural specimens to measure bend strength as a function of temperature and to reveal the nature of crack propagation during fast (catastrophic) failure and subcritical crack growth (SCG). Fractography is the principal interpretive agent for the presence of SCG and for revealing the nature of crack propagation.

## MATERIAL AND EXPERIMENTAL PROCEDURES

The material used in this study was sintered α-SiC obtained from the Carborumdum Company (Niagara Falls, N.Y.) in the form of square billets of dimensions 100 mm x 100 mm x 10 mm. The material was prepared by cold pressing α-SiC powder, followed by sintering at high temperatures, producing a dense (98% theoretical) material with equiaxed α-SiC grains with an average size of 7-10 microns. Extremely fine porosity was distributed throughout the microstructure along the grain boundaries as shown later.

For flexural strength evaluation, bend bar specimens of dimension 32 mm long x 6 mm wide x 3 mm thick were machined from the α-SiC billets. All faces were ground lengthwise using 220 grit diamond wheel and the edges chamfered lengthwise to prevent notch effects. Specimens were tested in 4-point bending in an Instron machine (Model 1125, Instron Corp., Canton, Mass.) at a crosshead speed of 0.5 mm/min using a specially built self-aligning ceramic fixture (Govila, 1980a) made from hot-pressed SiC. The outer and inner knife-edges of the testing fixture were spaced 19 mm and 9.5 mm apart, respectively. The high temperature bend tests were conducted in air in a furnace attached to the testing machine crosshead.

The flexural stress rupture tests at high temperatures (1300° to 1400°C) in air were also conducted in four-point bending using the self-aligning ceramic fixture and furnace. The load was applied on the test specimen through a cantilever arm type deadweight assembly. The experimental set-up was equipped with a microswitch to cut-off power supply to the furnace and the timer at the instant failure of the specimen occurred and the total time-to-failure was recorded. An overall view of the test set-up is shown in Fig. 1.




**1.Lever Arm 2. Furnace 3.Microswitch**
**4. Load Indicator 5. Temperature Controller**

Fig. 1 Flexural stress rupture test rig

Several bend bar specimens were precracked at room temperature using a Knoop microhardness indenter (Wilson Instrument Division of ACCO, Bridgeport, Conn.) with indentation load of 19.61 N. This method produced surface cracks of approximately semi-circular geometry of crack depths varying from 85 to 90 μm. Details of the technique are given elsewhere (Govila, 1980b). Precracked specimens were tested in stress-rupture mode at 1200°C in air at varying stress levels in order to determine the material's susceptibility for subcritical crack growth.



## RESULTS AND DISCUSSION

### Flexural Strength vs. Temperature

Fifteen bend bar specimens were tested at 20°C in order to determine the mean strength and the Weibull modulus. The material showed a large scatter (Fig. 2) in fracture strength, $\sigma_F$, primarily because of significant variations in inherent flaw sizes due to processing. The $\sigma_F$ varied from a maximum of 390 MPa to a minimum of 289 MPa with a distribution mean of 337 MPa and a standard deviation of 37 MPa. Assuming a two-parameter Weibull distribution to be representative of the data, a value of Weibull modulus, m=11, was obtained. Two types of porosity were observed in the bulk material on the fracture faces of specimens (as seen later); (i) Fine pores usually of the size 0.5 to 3 μm distributed uniformly along the grain boundaries. (ii) Large pores usually of the size 20 to 80 μm. Failure occurred in a brittle (catastrophic) manner and the majority of the failure initiation sites were associated with porosity both surface and sub-surface, Fig. 3. The flexural strength was also evaluated at higher temperatures

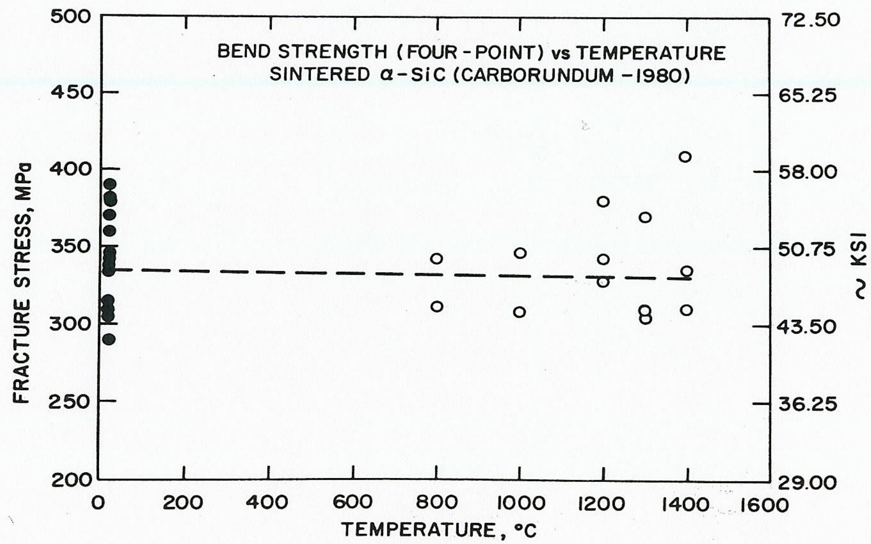



Fig. 2 Variation in fracture strength as a function of temperature

(800° to 1400°C) in order to determine the material's susceptibility for subcritical (slow) crack growth. The variation in fracture strength as a function of temperature is shown in Fig. 2. There is considerable scatter in the data, both in tests made at 20°C and higher temperatures (≥ 800°C). However, it should be noted that the mean strength for the two sets of the data (A total of 15 and 13 bend bar specimens were tested at 20°C and higher temperatures (≥ 800°C), respectively. Individual fracture strength values are given elsewhere (Govila, 1982)) are the same. Therefore, the flexural fracture strength, $\sigma_F$, is shown as constant and independent of temperature, Fig. 2. The fracture surfaces at high temperatures (1200° to 1400°C) were similar to that observed at 20°C, Figs.3(a-d), and did not show any signs of SCG. In all these tests, all fracture faces showed a smooth appearance characteristic of fast fracture similar to cleavage of ionic single crystals and this type of failure is characterized by transgranular crack propagation. In short, the flexural strength evaluation indicates the absence of SCG up to about 1400°C and the primary mode of fracture during crack propagation is transgranular.

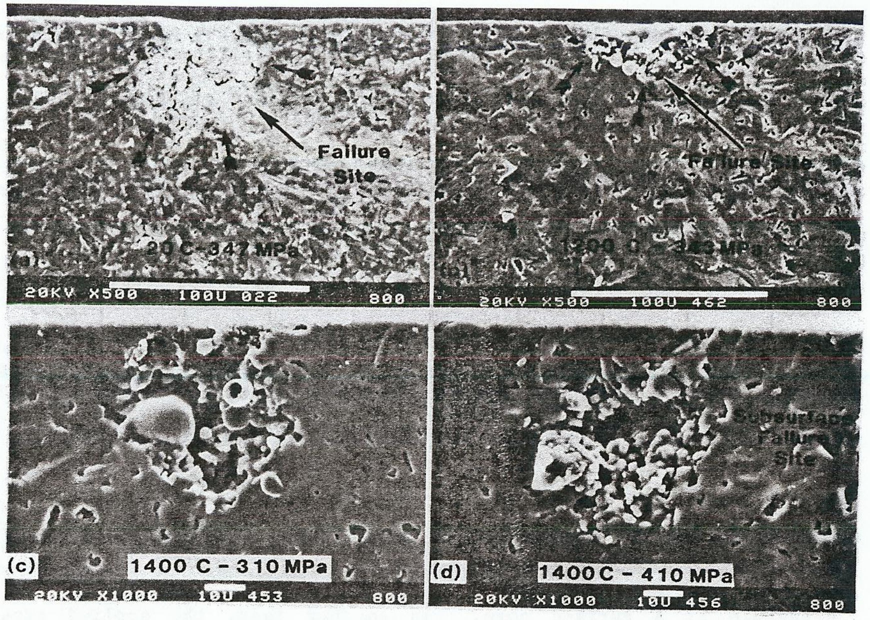



Fig. 3 SEM Fractographs showing typical failure sites in sintered α-SiC. Note uniform distribution of fine pores along the grain boundaries.

Flexural Stress Rupture Evaluation

Sintered α-SiC was examined in flexural stress rupture mode at 1400° and 1300°C for two reasons: (i) To determine the range of applied stress at which slow crack growth occurs. (ii) To characterize and identify the nature and mechanism for crack propagation during slow crack growth. From the temperature dependence of the fracture stress, Fig. 2, the mean flexural strength of the material is about 337 MPa and is essentially independent of temperature. It is important to note that the flexural stress rupture strength of these ceramic materials (in which processing flaws are large and widespread) is usually significantly lower than the mean flexural strength. Therefore flexural stress rupture tests were conducted below the mean strength in order to provide meaningful time-to-failure (> 10 h) data at the following temperatures.

At 1400°C and 1300°C in Air - At 1400°C, fourteen bend bar specimens were tested in a stress rupture mode at three different stress levels and the results are given in Table I. The time-to-failure varied from 0.1 h to 69 h. At an applied stress of 283 MPa, the time-to-failure was 0.3 h. At slightly decreased applied stress level such as 234 MPa, the time-to-failure



varied from 0.1 to 6 h. The majority of the specimens failed in short duration (< 1 h), fracture surfaces did not display clear evidence for the presence of SCG and were similar in appearance to fast fracture mode, Fig. 3.

TABLE 1 Flexural (4-point bending) Stress Rupture Results for Sintered Alpha SiC

| Specimen No. | Applied Stress, MPa | Time-to-Failure h(hours) | Failure Origin |
|---|---|---|---|
| Testing Temperature: 1400°C in Air | | | |
| 1 | 283 | 0.3 | Surface pore, no SCG+. |
| 2 | 234 | 0.1 | Crushed, not visible. |
| 3 | 234 | 0.3 | Sub-surface porosity, limited SCG. |
| 4 | 234 | 0.35 | Surface pore, no SCG. |
| 5 | 234 | 0.75 | Surface flaw with large grain. |
| 6 | 234 | 0.80 | Crushed, not visible. |
| 7 | 234 | 1.83 | Crushed, not visible. |
| 8 | 234 | 5.33 | Sub-surface porosity, limited SCG. |
| 9 | 234 | 0.16* | Crushed, not visible. |
| 10 | 234 | 5.00* | Sub-surface porosity, large SCG. |
| 11 | 234 | 6.00 | Crushed, not visible. |
| 12 | 195 | 0.25 | Surface pore, no SCG. |
| 13 | 195 | 20.00 | Large SCG region (Fig. 4). |
| 14 | 195 | 69.00 | Large SCG region. |
| Testing Temperature: 1400°C in Argon Gas Environment | | | |
| 15 | 234 | 0.2 | From corner, no SCG. |
| 16 | 210 | 60.00 | Large SCG region (Fig. 5). |
| Testing Temperature: 1300°C in Air | | | |
| 17 | 327 | 0.3 | Surface pore, no SCG. |
| 18 | 327 | 2.00 | Crushed, not visible. |
| 19 | 327 | 3.50 | Crushed, not visible. |
| 20 | 293 | 1.30 | Crushed, not visible. |
| 21 | 293 | 26.00 | Broke in 3 pieces, not visible. |
| 22 | 293 | 53.00 | Porosity and SCG. |
| 23 | 244 | 152.00 | Large SCG Region. |
| 24 | 210 | 358.00 | Large SCG Region. |
| Testing Temperature: 1300°C in Argon Gas Environment | | | |
| 25 | 293 | 27.00 | Porosity and SCG. |
| 26 | 244 | 81.00** | Large SCG region. |

\+ Slow Crack Growth
* Specimens annealed at 2000°C in high vacuum for 10 min. prior to testing.
** Stepped Stress Rupture Test

At low stress levels of 195 MPa, the time-to-failure was considerably longer (20 to 60 h) provided the specimen did not have a large processing flaw. These specimen confirmed time dependent failure and fracture surfaces showed large regions of SCG, Fig. 4. Since these specimen were tested in air environment, the fractographic appearance of SCG on fracture faces can be influenced by oxidation. Therefore, in an attempt to find out if SCG also occurs in an inert environment at 1400°C, preliminary tests were carried out in a partially Argon gas environment. Two bend bar specimens were tested in stress rupture mode at applied stress levels of 234 MPa and 210 MPa and failed in 0.2 h and 60 h, respectively, Table I. Specimen tested at the higher stress (234 MPa) basically behaved in the same fashion as those tested in air at the same temperature and stress, and essentially failed in fast fracture mode. The other specimen tested at a slightly lower stress (210 MPa) and sustained the stress for a fair length of time (60 h) showed a large region of SCG, Fig. 5, similar in its appearance to that seen in tests made at the same temperature (1400°C) in air, Fig. 4. Crack propagation

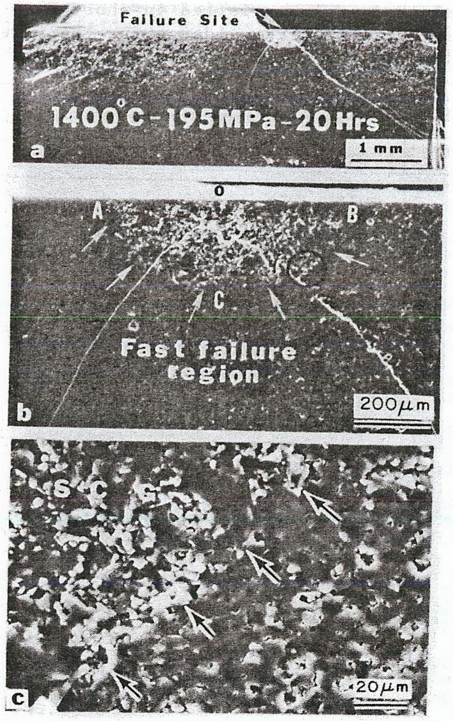



Fig. 4

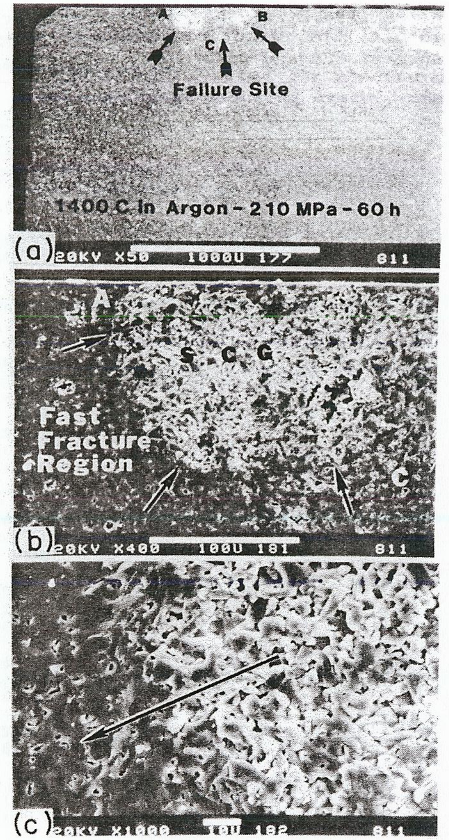



Fig. 5 ➤➤

Fig. 4 SEM fractographs. (a) Failure site. (b) ACB is the slow crack growth region. Note the change in morphology of the fracture surface outside the slow crack growth region. (c) Circled area in (b) shown at a higher magnification.

Fig. 5 SEM fractographs. (a) Failure site. (b) Higher magnification view of the slow crack growth region. (c) Higher magnification view of the boundary along AC. Long arrow indicates the transition from slow crack growth to fast fracture.

during SCG is exclusively intergranular (Figs. 4c, 5c) while the fast failure region displays transgranular mode of fracture, Figs. 4, 5. Other investigators (Coppola and others, 1978; Srinivasan, 1979; Quinn, 1980; Quinn and Katz, 1980; Quinn, 1981) have also observed the presence of SCG in this material at 1400°C in air.

At 1300°C - Ten bend bar type specimens were tested at applied stresses varying from 327 to 210 MPa and the time-to-failure varied from 0.3 to 350 h, Table I. Specimens tested at applied stress, 327 MPa, (close to mean strength, 337 MPa) failed in short time (< 4 h) in an essentially fast fracture mode (brittle manner). As the applied stress was decreased, the time-



to-failure increased considerably (> 10 h) and the fracture surfaces of test specimens displayed large regions of SCG similar to that seen at 1400°C (Govila, 1982).

At 1200°C - Detailed flexural stress rupture studies similar to the one carried out in this study have been made on sintered α-SiC (Carborundum 1980 material or comparable) at 1200°C in air by Quinn (1981) and others (Srinivasan, 1979; Quinn, 1980; Quinn and Katz, 1980). Therefore, no detailed efforts were made in this study to carry out stress rupture tests at 1200°C. However, studies by Quinn and Katz (1980) and Quinn (1981) concluded on the basis of quantitative estimates (using a log $\sigma_F$ vs log $t_F$ time-to-failure plot and determining the crack propagation parameter, n, as shown later) that the material showed time-dependent failure or SCG. In both studies, these authors admitted that the fracture surfaces failed to show the presence of SCG and appeared similar to the fast fracture surface at 20°C. Therefore, in this investigation stress rupture studies were concentrated at 1200°C in air to determine if sintered α-SiC shows SCG and to identify the mechanism for crack propagation. The use of precracked specimens in revealing the occurrence of SCG at a given temperature and applied stress has been successful in several ceramic materials (Govila, Kinsman and Beardmore, 1978, 1979). The precracked specimen contains a localized stress concentration site (a semi-circular type region) and therefore, all the resulting micro-plastic deformation leading to SCG will be concentrated along the boundary of the crack front. This facilitates fractographic examination to identify SCG since the occurrence is confined to this well identified region.

Precracked specimens containing a crack, 85-90 μm deep, were tested at 20° and 1200°C and the results are summarized in Table 2. The first specimen was subjected to 137 MPa at 1200°C in air and sustained the load for 300 h without failure. The specimen was unloaded and broken in 3-point bending at 20°C to reveal the extent of SCG at the crack front. The specimen did not fail at the precrack site, possibly due to crack blunting and broke elsewhere at a surface pore. In another test, the applied stress was increased to 150 MPa and the specimen sustained the stress for 144 h without failure. The specimen was unloaded, broken in 3-point bending at 20°C, failed at the precrack site, and showed the occurrence of SCG along the crack front boundary, Fig. 6. The SCG band as seen at 1200°C, Fig. 6(a), is similar in its appearance to that seen at 1400°C, Figs. 4-5, and the morphology of crack propagation during SCG is largely intergranular, Fig. 6(b). Increased applied stress levels led to early failure, Table 2. Long term tests of uniaxial tensile stress rupture testing of sintered α-SiC at 1200°C revealed fractographically limited evidence for the presence of SCG, Fig. 7, and discussed in detail elsewhere (Govila, 1983).

## Crack Propagation Parameter

The flexural stress rupture results were also analyzed to estimate the values of the crack velocity exponent, n, in the crack velocity-stress intensity relation, $V=AK_1^{\ n}$ (assuming that the material obeys this simple power law), for subcritical crack growth following the work of Davidge, McLaren and Tappin, 1973, and others (Quinn, 1980; Ritter, 1978). Under delayed fracture conditions, the ratio of failure times $t_1$, and $t_2$ under constant applied stresses $\sigma_1$ and $\sigma_2$ at a given temperature for a given environment is approximately given by:

$$(\sigma_1/\sigma_2) \simeq (t_2/t_1)^{1/n} \qquad (1)$$

TABLE 2 Flexural Strength and Stress Rupture Results for Precracked (Knoop Indentation Load = 19.61 N) Sintered α-SiC Specimens

| Test No. | Test Temp. | Crack Depth, ~ μm | Fracture Stress at 20°C | Applied Stress and Time | Remarks |
|---|---|---|---|---|---|
| 1 | 20°C | 88 | 187 MPa | - | Failed at the precrack site. |
| 2 | 20°C | 88 | 191 MPa | - | Failed at the precrack site. |
| 3 | 1200°C | - | - | 137 MPa-300 h | Unloaded, did not fail at the precrack site. |
| 4 | 1200°C | 88 | 294 MPa (3-point) | 150 MPa-144 h | Unloaded, failed at the crack site and showed SCG (Fig. 6). |
| 5 | 1200°C | 88 | - | 175 MPa-10 h | Failed at the crack and showed SCG. |
| 6 | 1200°C | 88 | - | 195 MPa | Failed instantly. No SCG. |


Fig. 6 SEM fracture surface of a precracked sintered α-SiC specimen tested in flexural stress rupture. (a) Precracked region ACB is surrounded by the white band as the slow crack growth region. (b) Higher magnification view of the white band region along EACD path showing intergranular crack propagation during slow crack growth.

A plot of log σ against log t would result in a straight line with a slope of 1/n. The results for the flexural stress rupture testing of sintered α-SiC at 1200°, 1300° and 1400°C in air are shown in Fig. 8. Because of scatter and limited experimental data points, no least squares analysis was done for curve fitting at 1300° and 1400°C. Note the extreme scatter displayed by the data obtained at 1200°C (Quinn, 1981). At 1200°, 1300° and 1400°C, the values of n obtained from the plot, Fig. 8, were about 25, 16 and 13, respectively. The n values obtained at 1300° and 1400°C are comparable in magnitude and differ significantly from that obtained at 1200°C and a trend is clearly indicated towards a lower n value at the higher temperature suggesting the presence of slow crack growth. This was confirmed by the fracture surfaces of the specimens tested at 1300° or 1400°C in air which clearly showed the large regions of SCG (Figs. 4,5). Relatively higher value of n obtained at 1200°C is indicative of the absence of SCG or limited presence of SCG.



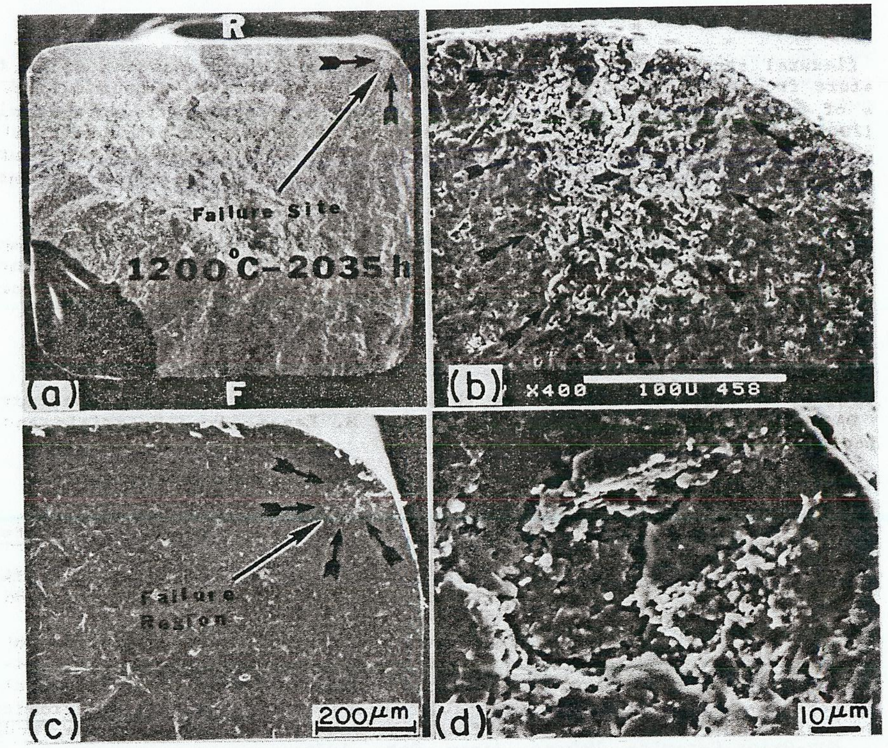



Fig. 7 SEM fracture surfaces of sintered α-SiC specimen tested in uniaxial tensile stress rupture mode at 1200°C in air. (a) Overall view (bottom half) showing failure site; black mark at bottom left is aquadag. (b) Enlarged view of failure site showing that failure region is distinct in appearance from the remainder of the fracture surface and shows limited SCG. (c) Upper half of the specimen showing failure site is internal. (d) Enlarged view of failure site seen in (c).

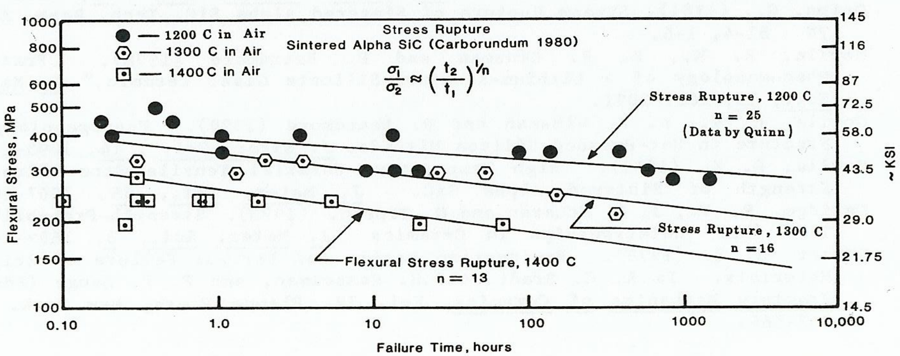



Figure 8 Flexural stress rupture results

## CONCLUSIONS

The flexural strength of sintered α-SiC is essentially independent of temperature from 20° to 1400°C. Failure occurred in a brittle manner and the mode of crack propagation during fast fracture is primarily transgranular. At 1200°C, fractographic evidence shows restricted occurrence of subcritical crack growth in sintered α-SiC. Extensive subcritical crack growth occurred with increasing temperatures (> 1300°C) and the mode of crack propagation during SCG is primarily intergranular.

At 1400°C, the limiting stress is about 175 MPa while for the temperature range 1300° to 1200°C, it is about 250 MPa, which the material can sustain for a reasonable time (< 100 h) before significant slow crack growth occurs and lead to sudden failure.

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