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# High temperature uniaxial tensile stress rupture strength of sintered alpha SiC

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Uniaxial tensile stress rupture testing of sintered $\alpha$-SiC was carried out at 1200 and 1300° C in air at various applied stress levels and the corresponding times-to-failure were measured. Fractographic evidence from uniaxial tensile stress rupture testing at 1200° C revealed limited presence of subcritical (slow) crack growth associated with surface connected porosity failure sites. The extent of slow crack growth (SCG) increased with increasing temperature and large regions of SCG were observed in tests made at 1300° C. An estimate of the crack velocity exponent, $n$, in the crack velocity–stress intensity factor relation, $V = AK_{\text{I}}^n$, for SCG has been made. Slow crack growth is characterized primarily by intergranular crack propagation while fast fracture (catastrophic failure) occurs transgranularly.

## 1. Introduction

Sintered $\alpha$-SiC is currently being investigated for use as structural components for gas turbines and diesel engines. The primary reasons for its use in heat engines are good oxidation resistance and strength at high temperatures ($\geqslant 1000°$ C), high thermal conductivity and possibly better creep resistance relative to other structural ceramics (hot-pressed $Si_3N_4$, lithium–aluminium–silicate glass ceramic, etc.). Before this material can be used in any commercial application, its reliability and durability must be established. This usually requires long term testing as a function of temperature and stress in a specified environment.

Most of the mechanical strength properties of ceramics are usually evaluated using 3-point or 4-point bend (flexural) tests because of the simplicity and relatively inexpensive nature of the test compared to other tests such as uniaxial tension. In a bend test, the maximum tensile stress occurs at the surface and, therefore, the majority of the failures occur due to surface-initiated flaws. One seldom sees failure initiation occurring internally (in the cross-section) in a bend test unless randomly occurring inherent (processing) flaws in the material are much larger than surface flaws or surface damage (acting as an initiating site due to machining or grinding damage). However, this behaviour is minimized in a uniaxial tensile test where the whole cross-section of the test specimen and all randomly occurring inherent flaws in the material are subjected to uniform stress* and failure should occur at the largest flaw. Because of this, the failure stress in uniaxial tension is usually smaller relative to the 4-point or 3-point flexural strength and is often much more representative of the material's strength for design purposes.

This study was undertaken primarily to investigate the reliability and durability of sintered $\alpha$-SiC, especially investigating the presence of subcritical (slow) crack growth (SCG) at 1200° C and higher temperatures in air. An estimate of the crack velocity exponent, $n$, or the crack propagation parameter, for SCG has been made and the results will be compared with flexural stress rupture tests at 1200° C in air.

*It is understood that the bending stresses due to misalignment are kept at a minimum and do not play a significant role in influencing the magnitude of the applied stress.

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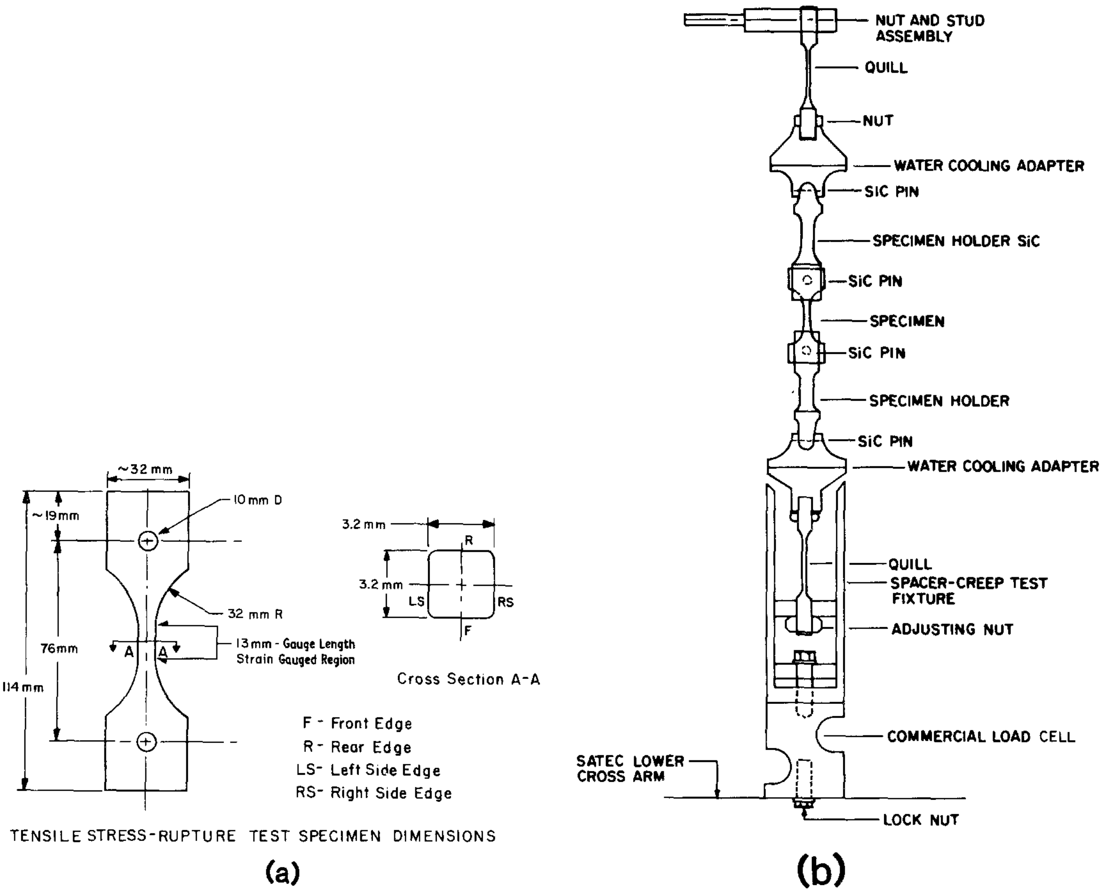


*Figure 1* (a) Geometry and dimensions of a tensile stress rupture specimen. (b) Schematic diagram of load train assembly for tensile stress rupture testing at high temperatures.

## 2. Experimental procedures

### 2.1. Material

The material used in this study was sintered $\alpha$-SiC obtained from the Carborundum Company†, in February 1980, in the form of square billets of approximate dimensions 100 mm x 100 mm x 10 mm. The material was prepared by cold pressing $\alpha$-SiC powder, followed by sintering at high temperatures, producing a dense (98% theoretical) material with equiaxed $\alpha$-SiC grains with an average size of 7 to 10 $\mu$m. The material had extremely fine porosity distributed throughout the microstructure along the grain boundaries, as shown later.

### 2.2. Tensile specimen geometry, preparation and load train design

The tensile stress specimen consisted of a simple rectangular geometry with a narrow cross-section in gauge length and large radii at the shoulders necessitated by the sensitivity of ceramic materials to stress concentrations. The specimen geometry and dimensions are shown in Fig. 1a. This geometry was selected rather than a circular cross-section to facilitate machining. All faces were ground lengthwise using 220-grit diamond wheels. In addition, the two contoured sides of the uniaxial tensile test specimen were hand-polished with diamond paste to give a smooth finish; thus the edges were rounded as well. A total of 24 tensile specimens from 12 different billets of sintered $\alpha$-SiC were machined and the specimens were randomly selected. Prior to testing, each tensile specimen was inspected visually (using optical microscopy at $\times 10$ magnification), radiographed (X-rays) and examined with fluorescent dye penetrants to determine the presence of any machining (surface) damage. Only those specimens which did not display (a total of 20) obvious surface damage were used for testing. In the gauge

†Carborundum Company, Niagra Falls, New York, USA.

length, the 90° corners or edges were hand-ground (with diamond paste) to a smooth curvature to minimize stress concentrations (as shown in cross-section A–A, Fig. 1a). The fracture face of the lower half of each broken specimen was examined fractographically and the edges of the fracture face were identified in a fashion shown in the cross-section A–A, Fig. 1a. This procedure also helped in determining if the fracture initiation sites were random or preferential.

A standard creep testing machine* with a modified load train assembly was used for the tensile stress rupture testing. The test sepcimen is retained in two slotted SiC holders by large SiC pins. The SiC holders are retained in water-cooled metal adaptors which in turn are attached to the standard Satec machine head which includes crossed (90°) knife edges. The assembly procedure includes hanging the load train parts from the Satec machine head as influenced by gravity. At this point the lower Satec cross-arm is lowered to load the train in this position. The load train assembly is shown schematically in Fig. 1b. A high-temperature furnace capable of reaching 1400° C is mounted on the side area of the Satec machine and encloses the full area between the water-cooled metal adaptors. The temperature is controlled and monitored separately with the use of two Pt–13Rh thermocouples placed behind the test specimen. Complete details regarding the load train instrumentation, continuous monitoring of applied stress, test temperature, and time are given elsewhere [1].

Each test specimen contained a total of eight strain gauges (two on each face in the gauge length area) which, for each test, were used for custom axial alignment to keep the bending stresses below 5% at full load. Initial alignment of the specimen is done at 20° C with full load. After alignment at 20° C, the lead wires of the strain gauges are cut and either the gauges are left on the specimen to burn out at high temperatures or the strain gauges are peeled off the specimen surface. During this time full load is maintained on the specimen.

## 3. Results and discussion

Before tensile stress rupture tests could be carried out on sintered $\alpha$-SiC, it was necessary to know the average flexural strength (4-point bend) at 20° C and its variation with increasing temperature (20 to 1400° C). In a separate study [2] it was reported that the mean flexural strength at 20° C of sintered $\alpha$-SiC is 337 MPa, Weibull modulus $m \approx 11$ and a standard deviation of 37 MPa. In addition, the flexural strength, $\sigma_F$, remained constant and independent of temperature up to 1400° C in air and the material did not show the occurrence of subcritical crack growth (SCG) in this temperature regime. Therefore, two temperatures (1200 and 1300° C) were chosen to conduct long term tensile stress rupture studies in order to investigate the presence of SCG as a function of constant applied stresses; the results are discussed separately as given below. A majority of the test specimens failed in the gauge length area but some failed at the lower or upper end of the gauge length [2].

### 3.1. Uniaxial tensile stress rupture at 1200° C

A total of 12 tensile stress rupture specimens of sintered $\alpha$-SiC were selected randomly from a batch of 20 specimens. Out of these, four specimens failed during axial alignment at room temperature and as such they were considered premature failures. The remaining eight specimens were tested at applied stresses varying from 100 to 240 MPa and failed in a time-dependent manner. Complete results are given in Table I. The first specimen tested in this temperature series, specimen 1, was subjected to 103 MPa (about 15 000 psi) and survived 1500 h at 1200° C in air with no signs of early failure. It was then decided to test the specimen in a stepped stress rupture series fashion as shown schematically in Fig. 2. The applied stress was increased in increments of about 7 MPa (about 1000 psi), each stress level being maintained for several hours. When the stress reached 200 MPa (about 29 000 psi), the specimen failed after 7 h. The total time to failure was 2035 h. The specimen broke in the centre of the gauge length [2] and the fracture surface for the bottom and upper half are shown in Fig. 3. The failure initiation site or region is in the vicinity of the rear right corner consisting of a small zone, Figs. 3a and c, and possibly be referred as a small SCG region which is distinct in appearance and surrounded by a fast fracture region, Fig. 3c. The morphology of the microstructure inside and outside the failure zone is different and characterizes two different types of crack propagation mechanisms.

*Satec Systems, Inc., Grove City, PA, USA.

TABLE I Uniaxial tensile stress rupture results for sintered α-SiC (Carborundum 1980)

| Specimen number | Applied stress (MPa) | Time-to-failure (h) | Approximate flaw size* (mm) | Failure proximity |
|---|---|---|---|---|
| *Testing Temperature: 1200° C in Air* | | | | |
| 1‡ | 103 | 2035† | 0.10–0.15 | Rear right corner, SCG (Fig. 3). |
| 2‡ | 172 | 1900 | – | Unable to identify failure site, no SCG. |
| 3 | 172 | 100 | 0.060–0.080 | Rear left corner, porosity and machining damage. No SCG (Fig. 4). |
| 4. | 207 | 447 | 0.10–0.30 | Front right corner, porosity and limited SCG (Figs. 6a and b). |
| 5 | 220 | 0.33 | 0.080–0.090 | Internal failure site (near rear left corner) due to an inclusion or foreign particle. No SCG (Figs. 6c and d). |
| 6 | 241 | 0.75 | 0.075–0.085 | Porosity and machining damage. No SCG. |
| 7 | 241 | 4 | 0.070–0.120 | Rear edge, machining damage and porosity. No SCG (Fig. 5). |
| 8‡ | 207 | 1500† | – | Front surface edge and no visible SCG. |
| *Testing Temperature: 1300° C in Air* | | | | |
| 9 | 172 | 171 | AB ≈ 0.20<br>CO ≈ 0.15 | Internal (0.064 mm long) pore and large SCG region. |
| 10 | 172 | 833 | AB ≈ 0.250<br>CO ≈ 0.130 | SCG region along right surface edge (Fig. 7). |
| 11 | 186 | 1 | – | Machining damage near front right corner. |
| 12 | 186 | 23 | 0.125 | Limited SCG region along left side edge. |
| 13 | 186 | 27 | 0.130 | Small SCG region near rear left corner. |
| 14 | 207 | 0.5 | – | Machining damage near front left corner and porosity. |
| 15 | 207 | 7.5 | AB ≈ 0.175<br>CO ≈ 0.100 | Large region of SCG along front edge. |

*These do not take into account the shape of the flaw but simply represent its length or width.
†Stepped stress rupture test and as such not plotted in Fig. 8.
‡During these long tests, the SiC heating elements burned out several times, replaced at room temperature while the test specimen maintained full load and, subsequently, the furnace was restarted.

Inside the failure zone, grain boundary opening and separation of grains is visible which is characteristic of intergranular crack propagation (Figs. 3b and d) while outside the fracture zone shows smooth appearance, characacteristic of fast failure indicating transgranular crack propagation. In this region, no separation of grains is visible. However, in this region a uniform distribution of fine porosity along grain boundaries is visible, Fig. 3b. Since the specimen survived for a considerable length of time, it is unlikely that the failure started due to any machining damage around the rear right corner.

The next specimen, 2, was subjected to a higher stress, 172 MPa (about 25 000 psi) and failed after 1900 h. Both halves of the fractured specimen, examined in SEM, did not show the presence of SCG and a localized failure initiation region [2] similar to the one in specimen 1 was observed, Figs. 3a and c. To confirm the lack of a significant SCG region (especially when the specimen survived for such a long time which suggested SCG may

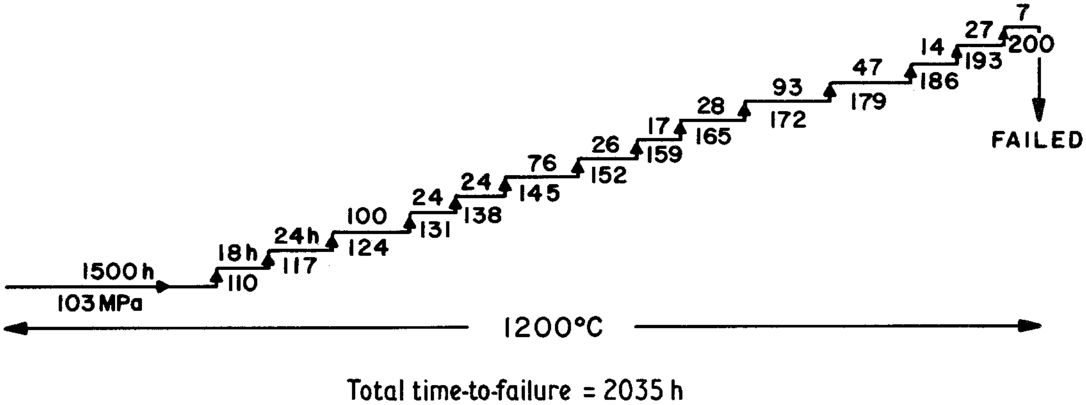


*Figure 2* Schematic illustration of the stepped stress rupture testing in uniaxial tension for sintered alpha SiC specimen 1.

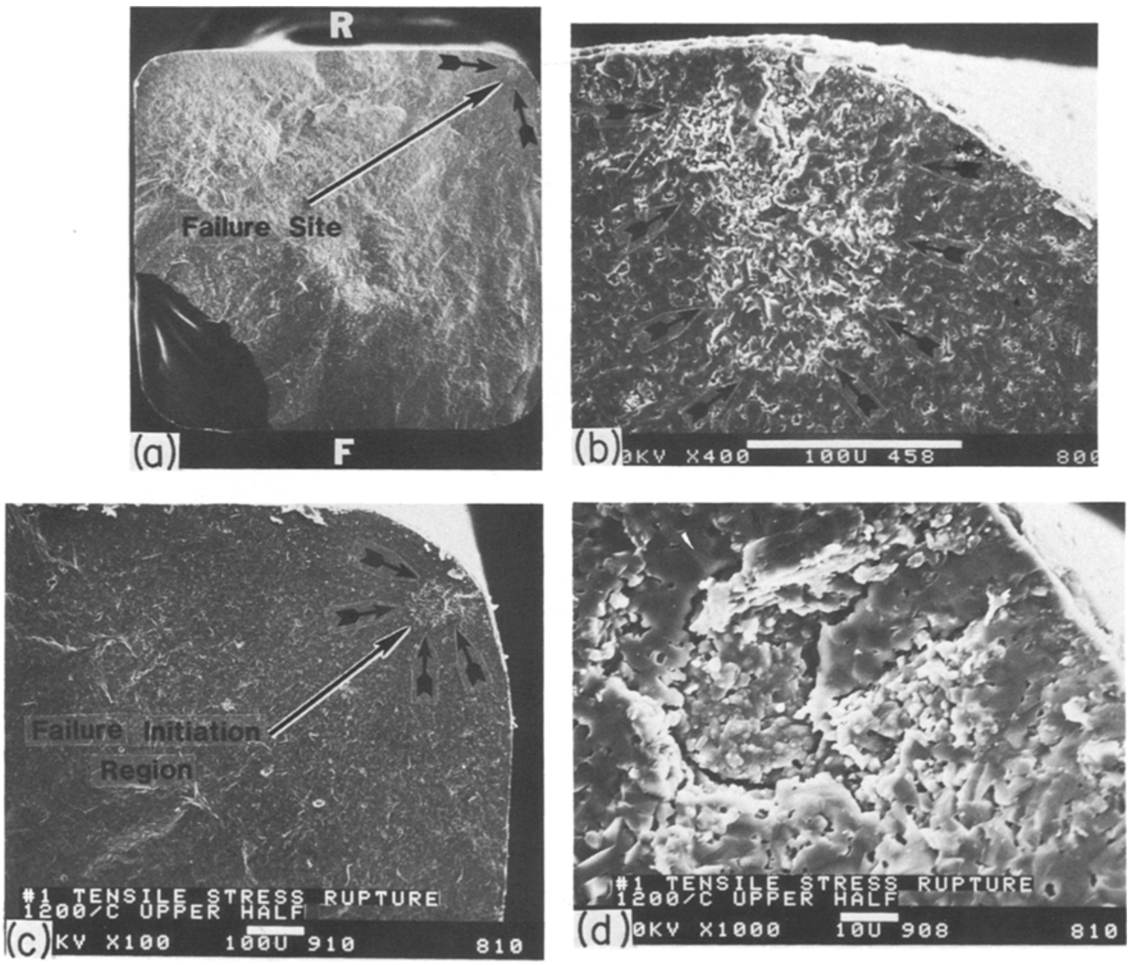


*Figure 3* SEM fractographs of the fracture surfaces of sintered alpha SiC specimen 1 (Table I) tested at 1200° C in air. (a) Overall view of the fracture surface (bottom half) showing failure initiation site; black mark at bottom left is aquadag. (b) Enlarged view of failure site showing that failure region is distinct in appearance from the remainder of the fracture surface and shows limited slow crack growth. (c) Upper half of the specimen showing failure site (slow crack growth region) is internal. (d) Enlarged view of the slow crack growth region seen in (c). Note the separation of grains along the grain boundaries clearly indicates that crack propagation during slow crack growth is primarily intergranular.

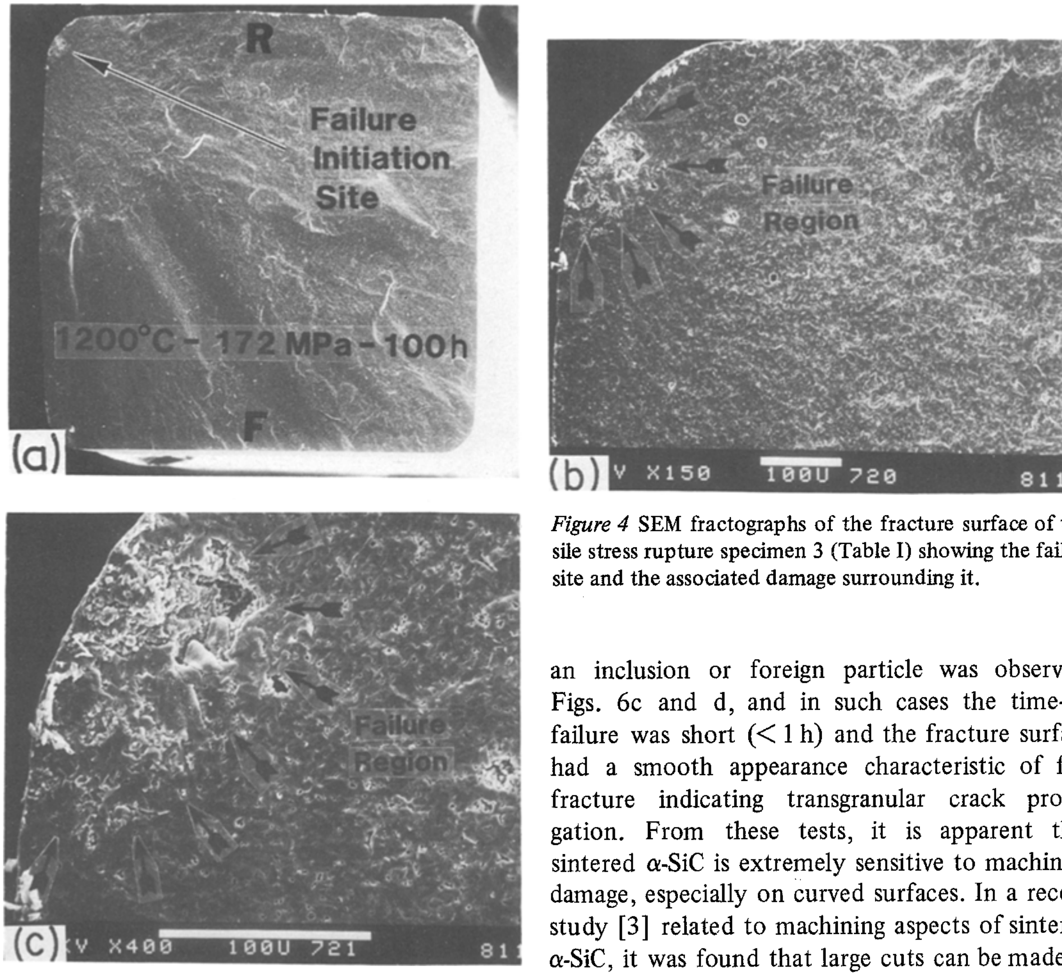


*Figure 4* SEM fractographs of the fracture surface of tensile stress rupture specimen 3 (Table I) showing the failure site and the associated damage surrounding it.

have occurred) another specimen, 3, was tested at the same applied stress (172 MPa) and temperature (1200° C in air) and failed after 100 h. The fracture face showed clearly the failure initiation site, Fig. 4a, in which considerable porosity is evident. It is believed that the early failure relative to specimen 2, was due to some machining damage around the rear left corner which opened up several grains (by joining up fine pores along grain boundaries) in the material. Under the applied stress, the pores spread out, reached a critical size and led to sudden fast failure in a relatively short time without showing signs of SCG, Figs. 4b and c. The same phenomenon occurs more clearly under higher stress and the time-to-failure is reduced drastically, Fig. 5 (see specimens 5 to 7, Table I).

The next specimen was tested at an applied stress of 207 MPa (about 30 000 psi) and failed after 447 h. The failure region is clearly visible, Fig. 6a, showing limited SCG inside the failure region, Fig. 6b. Occasionally, failure occurring at an inclusion or foreign particle was observed, Figs. 6c and d, and in such cases the time-to-failure was short ($< 1$ h) and the fracture surface had a smooth appearance characteristic of fast fracture indicating transgranular crack propagation. From these tests, it is apparent that sintered $\alpha$-SiC is extremely sensitive to machining damage, especially on curved surfaces. In a recent study [3] related to machining aspects of sintered $\alpha$-SiC, it was found that large cuts can be made in the material without introducing surface flaws. But this was only true for planar surface machining and not for curved surfaces.

From the results presented so far (Table I), it is clear that surface initiated flaws (primarily from corners) induced due to poor machining led to early failure, whereas failure initiation occurring inside the cross-section (Fig. 3) leading to the occurrence of SCG usually displayed a much longer life. These results strongly suggest that the occurrence of SCG in sintered $\alpha$-SiC at 1200° C in air is restricted (not widespread) and requires a critical level of applied stress and significantly long periods of time ($\geqslant 400$ h). It should be pointed out that detailed flexural stress rupture tests on sintered $\alpha$-SiC (Carborundum 1978 and 1980) at 1200° C in air have been made by Quinn and Katz [4] and Quinn [5] and the specimens which sustained the applied stress for long periods of time ($\geqslant 500$ h) before failure, did not show the occurrence of SCG on the fracture faces. In their studies [4, 5], all the fracture surfaces had

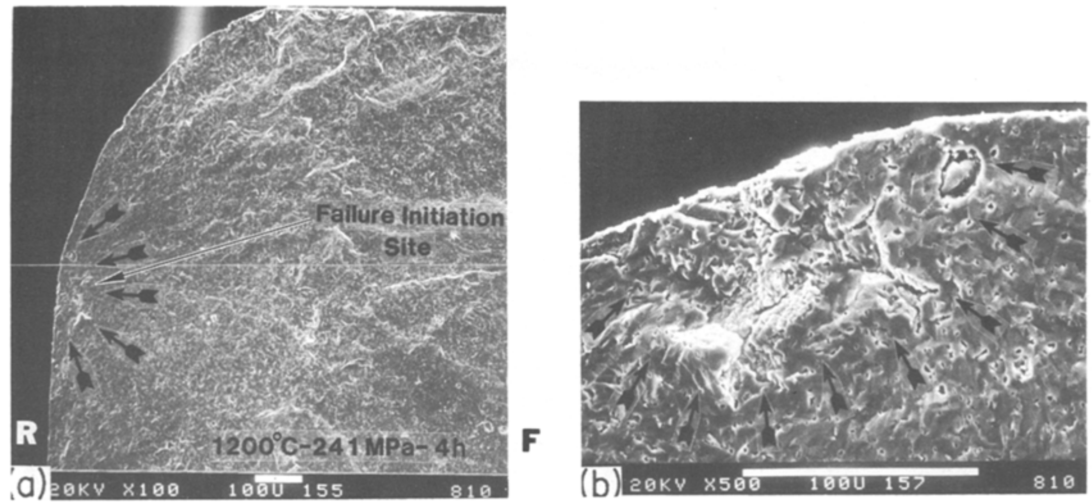


*Figure 5* SEM fractographs of the fracture surface of tensile stress rupture specimen 7 (Table I) showing possible machining damage leading to early failure. (a) Failure site and the surrounding mirror region. (b) Enlarged view of failure site. No visible slow crack growth.

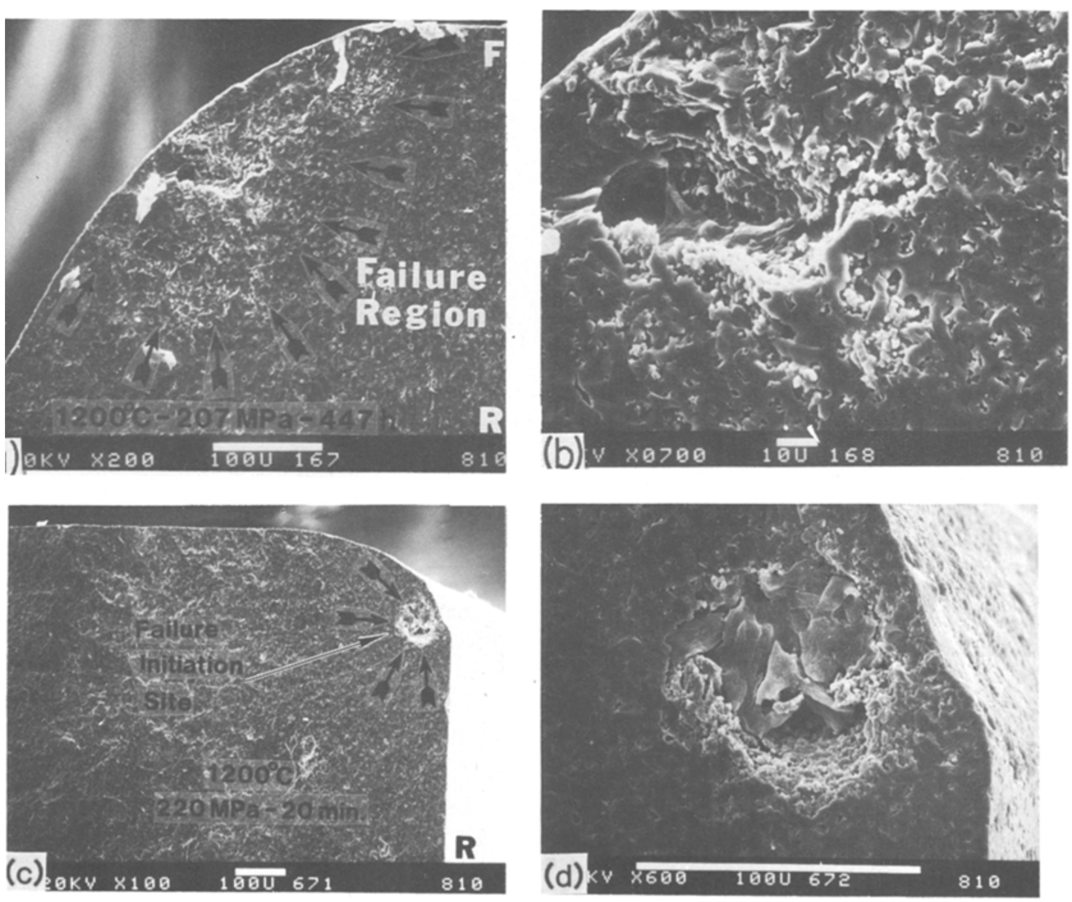


*Figure 6* SEM fractographs of the fracture surface of tensile stress rupture specimen 4(a and b) and 5(c and d), (Table I). (a) Failure region. (b) Enlarged view of failure region showing cavitation and limited slow crack growth. (c) Internally occurring failure initiation site at an inclusion or foreign particle showing smooth fracture surface characteristic of fast failure. (d) Enlarged view of failure site showing no signs of slow crack growth.

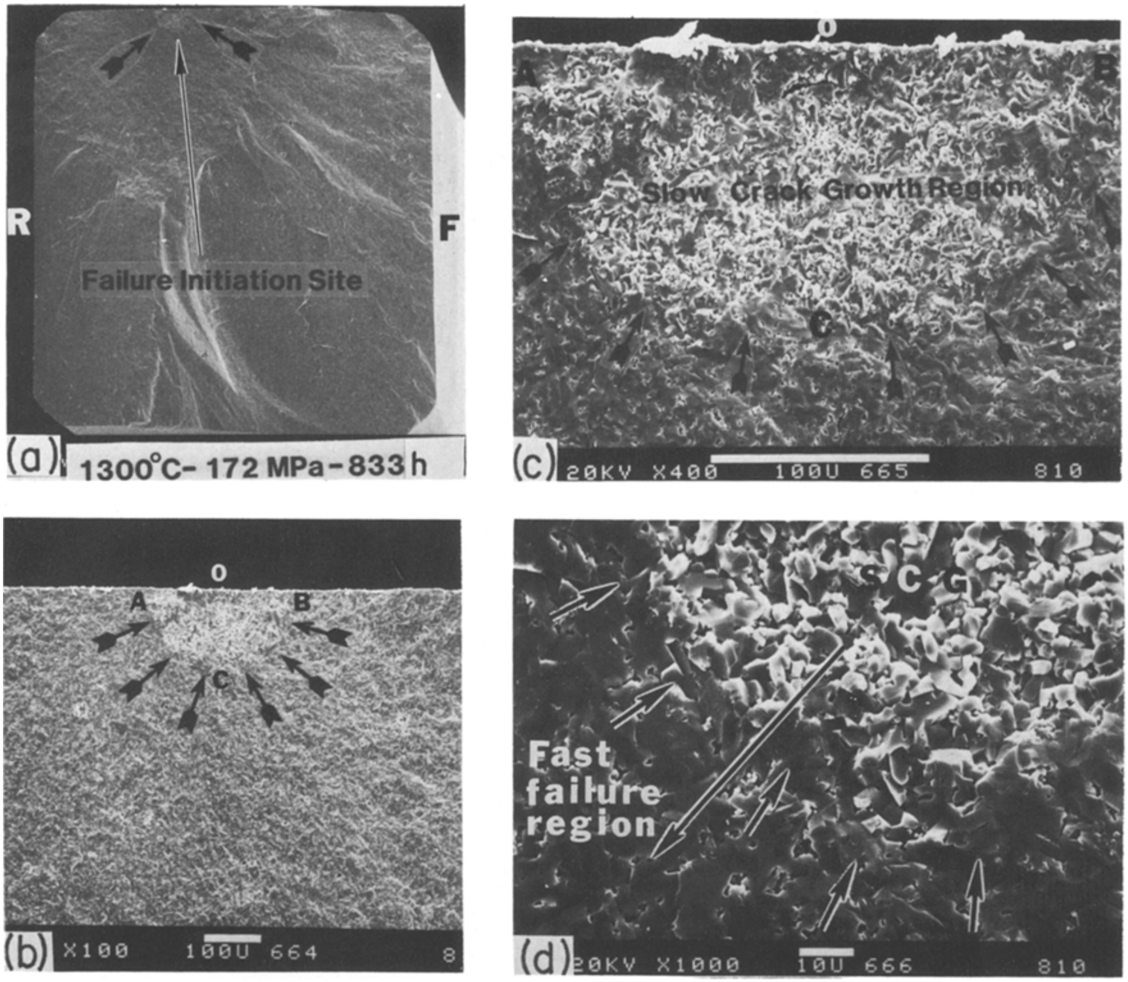


*Figure 7* SEM fractographs of the fracture surface of specimen 10 (Table I) tested at 1300° C in air at 172 MPa and failed after 833 h. (a) Surface associated failure site and the surrounding mirror region. (b) ACB is the failure site and represents the slow crack growth (SCG) region. (c) Enlarged view of the SCG region ACB. (d) Arrow indicates the transition from SCG region to fast failure region. The nature of crack propagation inside the SCG region is primarily intergranular while outside the SCG region, it is transgranular failure.

a fast fracture appearance similar to that observed at 20° C and the majority of the failure origins were associated with surface porosity.

### 3.2. Uniaxial tensile stress rupture at 1300° C

Seven specimens (excluding one that failed prematurely during axial alignment at 20° C) were tested in a uniaxial tensile stress rupture mode at 1300° C in air at various applied stress levels and the results are given in Table I. The first two specimens (9 and 10, Table I) in this temperature series were tested at the same applied stress level (172 MPa) as that used in the 1200° C tests in order to make a direct comparison of the influence of test temperature. Both specimens displayed large regions of SCG and failed after sustaining the stress for 171 h (sample 9) and 833 h (sample 10), respectively. The specimen failing in a shorter time (171 h) had a large void (about 0.064 mm long) inside the semi-circular shaped SCG region. The void possibly acted as a localized stress concentration site and thus led to early failure. The other specimen (10) which failed after a significantly longer time (833 h) did not display such a large single void (see Fig. 7), highlighting the sensitivity of the fracture process to the presence of large stress concentrators such as voids, etc. This points out the large influence of temperature in increasing the extent of the

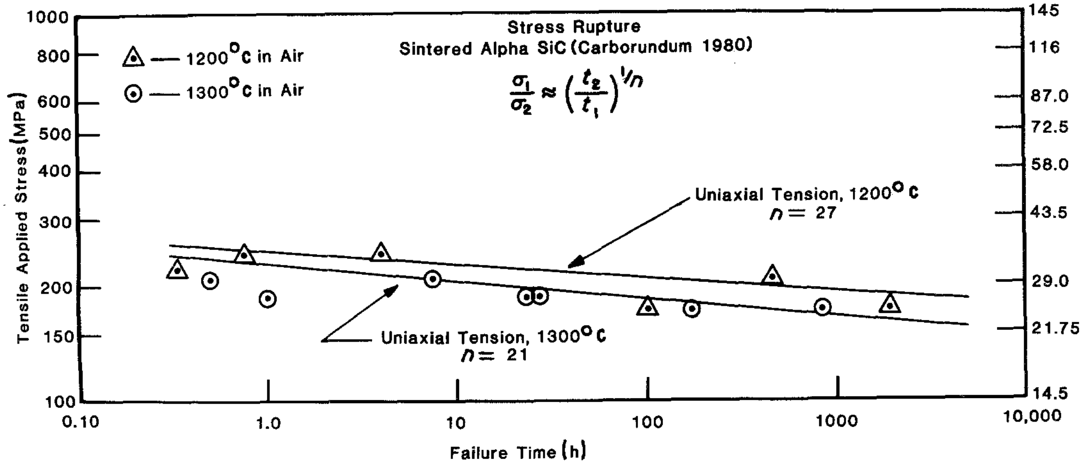


*Figure 8* Uniaxial tensile stress rupture results.

subcritical crack growth region. Note that the specimen 2, tested at the same applied stress (172 MPa), sustaining the stress for 1900 h at 1200° C in air, did not display the occurrence of SCG.

The outstanding feature of failure initiation due to a surface flaw in a uniaxial tensile test is that the growth of the flaw (subcritical or slow crack growth) should occur from the edge of the test specimen and also be symmetrical due to the uniform stress distribution if bending stresses are minimal. Such incidence was displayed by those specimen showing SCG at 1300° C (see Table I) as typically illustrated in Fig. 7 for the specimen 10 failing after 833 h. The fracture face clearly showed the failure site, followed by a mirror region, Fig. 7a. The failure site consisted of a semi-circular region ACB, Fig. 7b, which was actually a slow crack growth region, Fig. 7c, and clearly distinctive from the remainder of the fracture surface (fast fracture region), Fig. 7b. Once the flaw (SCG region) grew to a critical size, catastrophic failure occurred. The nature of the crack propagation during SCG is intergranular while the fast fracture occurred by the transgranular mode as seen clearly and distinctively in Fig. 7d. The long arrow, Fig. 7d. indicates the transition from SCG to fast fracture. Inside the SCG region, separation of grains characteristic of intergranular crack propagation is clearly visible.

In addition to the temperature effect, the influence of small increase in applied stress in promoting the SCG in sintered α-SiC and thus drastically reducing the total time-to-failure is clearly indicated by specimens 12, 13 and 15 (see Table I). Increasing the applied stress by a small magnitude, such as 14 MPa (about 2000 psi) decreased the time-to-failure by over an order of magnitude. In tests at 1300° C in air, all of those specimens which failed in a time-dependent manner, displayed the occurrence of extensive SCG regions. The uniaxial tensile stress rupture tests at 1300° C have clearly demonstrated the extreme influence of applied stress and temperature in revealing the presence of SCG.

### 3.3. Crack propagation parameter

The uniaxial tensile stress rupture results were also analysed to estimate the values of the crack propagation parameter or crack velocity exponent, $n$, in the crack velocity–stress intensity factor relation, $V = AK_I^n$ (assuming that the material obeys this simple power law), for subcritical crack growth following the work of Davidge *et al.* [6] and Ritter [7]. 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:

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{t_2}{t_1}\right)^{1/n}. \qquad (1)$$

A plot of log $\sigma$ against log $t$ would result in a straight line with a slope of $1/n$. The results for the uniaxial tensile stress rupture testing of sintered α-SiC at 1200 and 1300° C are shown in Fig. 8. Because of scatter and limited experimental data points, no least-squares analysis was done for curve fitting. At 1200 and 1300° C, the values of $n$ obtained from the plot (Fig. 8) were about 27 and 21, respectively. Both values are comparable in magnitude but 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 the higher temperature of 1300° C in air which clearly showed the large regions of SCG (e.g. see Fig. 7). Only restricted and limited evidence for the presence of SCG was observed in tensile stress rupture tests at 1200° C after the specimen sustained the stress for a significant length of time (see Figs. 3 and 6). Quinn and Katz [4] and Quinn [5] reported values of $n$ at 1200° C in air to be around 41 for 1978 sintered $\alpha$-SiC and 25 for 1980 sintered $\alpha$-SiC, respectively, using a flexural stress rupture testing method. Both groups of materials (1978 and 1980 $\alpha$-SiC) showed large variations in time-to-failure for a given applied stress, fracture surfaces had a smooth fast fracture surface appearance and did not show the occurrence of SCG similar to that seen in the tensile stress rupture (Fig. 3) studies.

## 4. Conclusions

1. Detailed and careful uniaxial tensile stress rupture tests were made at high temperatures (1200 and 1300° C) in an air environment using sintered $\alpha$-SiC specimens in order to provide information relating to the presence of subcritical crack growth.

2. At 1200° C, fractographic evidence showed the restricted occurrence of subcritical crack growth in sintered $\alpha$-SiC. Extensive subcritical crack growth occurred at 1300° C.

3. The material could sustain a tensile applied stress of 175 MPa at 1200° C in air for a reasonable time ($\geqslant$ 100 h) without showing signs of degradation in strength.

4. The uniaxial tensile stress rupture testing is more sensitive in revealing the presence of slow crack growth fractographically compared to flexural stress rupture testing.


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