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MICROSTRUCTURE AND CREEP FAILURE
OF SILICON CARBIDE

G.H. Campbell
(M.S. Thesis)

December 1986

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Prepared for the U.S. Department of Energy under Contract DE-AC03-76SF00098
Microstructure and Creep Failure of Silicon Carbide

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M.S. Thesis

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Berkeley, California 94720

This work was supported by the U.S. Department of Energy under Contract Number DE-AC03-76SP00098.
Observations of the microstructure and creep behavior of two commercial silicon carbides are presented. A combination of techniques has been used to characterize the microstructure. Sequential creep rupture testing has been carried out in an inert environment at varied temperatures and strain rates, and scanning electron microscopy has been used to observe creep crack propagation and damage development with increasing strain. Basic theory is developed for stress fields and creep rates around a crack tip and is related to the observed brittle to ductile transition in the material as well as room temperature behavior of deformed beams.
Acknowledgement

I would like to express my deepest thanks to Dr. Brian Dalgleish. His broad experience and experimental expertise contributed greatly to this research. All of the TEM photos in this report are his as well as Figures 9, 12, and 16 and the X-ray spectra. I would also like to thank Professor Tony Evans. His insight allowed us to put the analysis of the results to be put in proper mathematical form.
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1. **INTRODUCTION**

The high temperature failure of ceramics has been shown to involve two predominant regimes: high stress ruptures by the extension of pre-existing cracks and low stress fractures that occur by damage accumulation (Fig. 1). The transition between regimes coincides with a relatively abrupt change in rupture strain (Fig. 1) and is accompanied by the creep blunting of pre-existing flaws (Fig. 2). Some aspects of the blunting transition have been studied for various $\text{Al}_2\text{O}_3^{1-6}$, $\text{SiC}^{7-10}$ and $\text{Si}_3\text{N}_4^{11}$ materials. However, present understanding of this vitally important aspect of creep rupture is still speculative. The prevalent speculation is that blunting occurs when the stress and displacement field ahead of the crack reduces below a threshold value, at which the nucleation of a crack tip damage zone (i.e. cavities at grain boundaries and/or in amorphous phases) is suppressed, resulting in a threshold stress intensity, $K_{th}$ (Fig. 3).

At stress intensities below $K_{th}$, rupture occurs by damage accumulation. For $\text{Al}_2\text{O}_3$, failure in this damage controlled regime has been shown to occur by the growth and coalescence of shear bands that nucleate at large microstructural and chemical heterogeneities within the material.$^{1-6}$ When such mechanisms are suppressed, by
'Crack propagation' (pre-existing flaws)

'Damage' control (creep damage)

\[ t_f \sim \sigma^{-n} \]

\[ \dot{\varepsilon}_f \sim g(\sigma) \]

FIG. 1
FIG. 3

- Asymptote to $K_c$
- Power-Law growth
- Crack velocity ($ms^{-1}$)
- $K / K_c$
having superior microstructural and chemical homogeneity,\textsuperscript{12} alternative failure processes can be expected. Indeed, recent research on ZrO\textsubscript{2}\textsuperscript{13} (TZP) and Al\textsubscript{2}O\textsubscript{3}/ZrO\textsubscript{2} composites\textsuperscript{14-16} indicates that, in some cases, sufficient damage does not occur below the blunting threshold and the material then becomes superplastic.

The abrupt change in rupture behavior around the blunting threshold can be regarded as a transition from creep brittleness to creep ductility, involving a competition between flow (creep) and fracture, as depicted schematically in Fig. 4. The rupture characteristics ascertained for constant strain-rate conditions should thus exhibit a transition temperature, $T_t$, below which the material fails at small strains (by crack growth from pre-existing flaws) and above which creep ductility obtains, with failure proceeding by damage mechanism.

The intent of the present research is to examine aspects of the brittle-to-ductile transition for two SiC materials, as needed to further understand this important phenomenon. Some prior research on SiC\textsuperscript{7-10} has provided indirect evidence of a blunting threshold, that varies with temperature, microstructure and environment. This research has also suggested that both the threshold and the creep crack growth above the threshold are dominated by the presence and the characteristics of amorphous
FIG. 4
grain boundary phases that either pre-exist or are formed by exposure to oxidizing environments. Specifically, low viscosity and low surface energy amorphous phases accelerate the crack growth and reduce the relative blunting threshold, $\frac{K_{th}}{K_c}$ (as also established for various $\text{Al}_2\text{O}_3$ and $\text{Si}_3\text{N}_4$ materials, Fig. 5). The present research is performed in an inert environment in an attempt to preserve the initial phase characteristics during testing.

An important constituent of the current research is the thorough characterization of the materials and the direct observation of crack blunting and of damage. These aspects of the research are presented first, followed by measurements of mechanical behavior and interpretation of the brittle to ductile transition.
$T = 1400 \, ^\circ\text{C}$

SIALON
(1\% Mn$_3$O$_4$/MgO)

SINGLE PHASE SIALON

$\beta' + \gamma\text{AG}$

FIG. 5
2. MATERIALS

2.1 Processing Conditions

One silicon carbide* was manufactured\(^{17}\) by mixing a fine, high purity silicon carbide powder with 0.5-5 wt% aluminum in a ball mill, using cobalt bonded tungsten carbide grinding media and hot-pressed at 2075°C and 18 MPa. The other material** was a sintered $\alpha$-SiC containing boron and excess carbon as sintering aids.\(^{18}\) The silicon carbide powder containing excess carbon was mixed with boron carbide powder in a ball mill using tungsten carbide balls. It was sintered at 2100°C in an atmosphere of argon. The carbon is believed to partially remove the silica layer on the silicon carbide powder and thus allow matter transport. The boron is then presumed to diffuse into the silicon carbide grain boundaries and pore surfaces and promote densification.\(^{19}\) The excess carbon may also inhibit exaggerated grain growth by pinning the grain boundaries.

---


**Hexalloy, Sohio Engineered Materials, Niagara Falls, NY
2.2 Specimen Preparation

Surfaces subject to examination were first mechanically polished. The polishing procedure began with 15 μm diamond paste on glass, reduced to 9 μm and then 6 μm: the latter on a lapping wheel. Polishing was completed with 1 μm diamond paste on a vibration polisher. Several specimens were thermally treated to highlight grain boundaries prior to examination. Treatments were conducted in vacuum or argon atmospheres. Temperatures ranged from 1300°C to 1600°C and times from 15 to 45 minutes.

2.3 Characterization Techniques

Chemical analysis was performed on both materials to determine the total boron, silicon, carbon, and oxygen contents. For boron analysis samples were crushed and the boron leached out into an acid solution. This solution was then plasma heated and the photon intensities characteristic of boron determined. The silicon content was determined by fusing powdered SiC. The resultant glass was dissolved in a hydrochloric acid solution and the silicon evaluated using atomic absorption. The carbon content was determined by fusing with an oxidant to evolve carbon dioxide. The carbon dioxide content was then analyzed using a coulometer and a carbonate standard.
Finally, the oxygen content was determined by neutron activation analysis. Other impurities were identified using semi-quantitative spectroscopic procedures.

A microprobe was used to determine the composition of second phase impurities from X-ray spectra and maps. The scanning electron microscope (SEM) was used to obtain information about porosity and carbon inclusions: the former on uncoated, mechanically polished surfaces and the latter on gold coated fracture surfaces. Transmission electron microscopy (TEM) was used to examine grain boundary phases employing both light and dark field techniques. Electron energy loss spectroscopy also identified the principal second phases.

Finally, the amounts of each SiC polytype present were determined using X-ray diffraction in conjunction with the analysis described by Rexer, et al.\textsuperscript{20}

\section*{2.4 Material Characteristics}

\subsection*{i. Hot-Pressed Silicon Carbide}

Specimens having a light thermal etch observed in the optical microscope revealed a grain size of about 1.5 \textmu m. The chemical analysis (Table 1) indicated appreciable oxygen, aluminum, tungsten and cobalt. The tungsten and cobalt were presumably introduced by the cobalt bonded tungsten carbide grinding media used to mix the initial powders.
### Table 1

**Material Composition**

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>(quantitative composition)</th>
<th>MAJOR IMPURITIES (semi-quantitative)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hot-Pressed SiC</td>
<td>29.84 wt% C</td>
<td>1.50 wt% Al</td>
</tr>
<tr>
<td></td>
<td>64.89 wt% Si</td>
<td>2.50 wt% W</td>
</tr>
<tr>
<td></td>
<td>50 ppm B</td>
<td>0.10 wt% Co</td>
</tr>
<tr>
<td></td>
<td>1.70 wt% O</td>
<td>0.10 wt% Fe</td>
</tr>
<tr>
<td>Sintered α-SiC</td>
<td>30.50 wt% C</td>
<td>0.18 wt% Al</td>
</tr>
<tr>
<td></td>
<td>69.40 wt% Si</td>
<td>0.07 wt% Ca</td>
</tr>
<tr>
<td></td>
<td>0.15 wt% B</td>
<td>0.06 wt% Cu</td>
</tr>
<tr>
<td></td>
<td>0.23 wt% O</td>
<td>0.16 wt% Fe</td>
</tr>
</tbody>
</table>
Microprobe analysis confirmed appreciable quantities of second phases. Backscattered electron images (Fig. 6) and related X-ray spectra identified silicides (Fig. 7) having variable composition. Some of these are tungsten silicide (dashed line) while others are a mixture of tungsten, cobalt, and iron silicides (solid line). The silicides are also revealed by TEM as intergranular second phase particles (Fig. 8). Attempts to image a grain boundary phase in the TEM were unsuccessful, indicating that amorphous phases, if present, must be less than ~5Å in width. A surface examined by SEM after a short thermal etch in argon shows large scale inhomogeneities (Fig. 9). The crack like features are presumed to be formed by volatilization of SiO formed by the reaction of SiC with Al₂O₃*, subject to low oxygen partial pressure conditions. A longer thermal etch revealed a high number density of bright-contrast second phase particles (Fig. 10) that increase in quantity with time, indicating that these phases migrate to the free surface at high temperature. X-ray analysis of these phases indicates that they are silicides having variable composition (Figs. 11a and b). Even longer thermal treatments cause the particles to assume different

*SiC + Al₂O₃ → SiO + Al₄C₃
morphologies and X-ray mapping shows compositional variations between the differently shaped particles (Fig. 12). Note that the aluminum is present everywhere while the other cations are confined to the second phase.

The X-ray diffraction data were consistent with a predominance of the 6H polytype, and detectable quantities of 15R, 4H and 3C, together comprising about 5% of the volume of the material.

ii. Sintered Silicon Carbide

Optical observations of thermally etched specimens revealed a grain size of about 10 μm. Chemical analysis (Table 1) indicated few impurities. Residual carbon is also implied by these results. Specifically, by assuming that all the silicon and boron present are in the form of carbides the residual carbon content can be estimated as 0.8 wt%. The presence of residual carbon is confirmed by transmission electron microscopy (Fig. 13a). Most definitively, the fine structure of the energy loss spectrum identifies the material as graphite (Fig. 13b). Additionally the laminar structure is consistent with the graphitic form. The graphite is present both at intergranular and intragranular sites (Fig. 14). The residual graphite is also well delineated on fracture faces (Fig. 15). Titanium and vanadium silicides have also been identified (Figs. 16, 17).
(a)

(b)

FIG. 13

Graphite K Edge
Finally, X-ray diffraction indicated mostly 6H polytype with about 5 vol% 3C and trace amounts of 15R and 4H.
3. **MECHANICAL PROPERTIES**

3.1 **Techniques**

The mechanical properties were investigated in four point flexure using 3x3x30 mm beams. Some beams were indented on the tensile surface, with a 200N Knoop indent, such that the long axis of the indenter was oriented perpendicular to the applied stress axis. The indent created a semicircular crack having 125 to 150 μm radius. The residual stress was removed by surface polishing. The edges of the tensile surface were also bevelled to remove edge flaws that might cause premature failure.

The fracture toughness was determined as a function of temperature by using indented beams tested at a strain-rate of 6x10^{-5} s^{-1}. The initial flaw size due to the indent was measured on the fracture surface in the SEM. The critical stress intensity factor, $K_c$, was determined with the relation\(^{21}\)

$$K_c = \frac{(2/\sqrt{\pi}) \sigma \sqrt{a}}{}$$  \hspace{1cm} (1)

where $\sigma$ is the applied stress and $a$ is the initial flaw radius.

Creep tests were performed at 1600 to 1800°C in an argon atmosphere, and at constant displacement rates of $10^{-7}$ to $10^{-9}$ m/s and the steady state creep properties
characterized by

\[ \dot{\varepsilon} = \dot{\varepsilon}_0 \left( \frac{\sigma}{\sigma_o} \right)^n \]  

(2)

where \( \dot{\varepsilon} \) is the strain rate, \( \sigma \) is the stress, \( n \) is the creep exponent, and \( \dot{\varepsilon}_0 \) and \( \sigma_o \) are constants. The steady-state stress on the tensile surface was calculated using

\[ \sigma = \frac{3(L - \ell)P}{bh^2} \left[ \frac{2n + 1}{3n} \right] \]  

(3)

where \( L \) is the outer span in four point bending, \( \ell \) is the inner span, \( P \) is the load, \( b \) is the thickness, and \( h \) is the height.

Sequential testing was used in some cases, with SEM examinations conducted between each iteration. The evolution of damage on the tensile surface and the behavior of the indentation flaws were thereby observed. At the conclusion of sequential testing (at about 10% strain on the tensile surface) the beams were fractured at room temperature. The material directly in front of the crack tip was then examined for damage in the SEM and a nominal toughness ascertained using Eq. (1).
3.2 Theoretical Results

Stationary cracks in a body subject to steady-state creep generate displacement and strain fields directly analogous to the corresponding non-linear hardening solutions. Consequently the behavior of cracks in the vicinity of the brittle to ductile transition temperature can be addressed and interpreted by invoking the appropriate non-linear solutions. The relevant results are summarized in this section. For a non-linear material subject to steady-state creep (Eq. 2) the stresses outside the blunting region have the form: 24

\[
\sigma_{ij} = \frac{1}{(n+1)} \sigma^* \left[ \frac{C^*}{\sigma_o \dot{\varepsilon}_o} \right] \tilde{\sigma}_{ij} \tag{4}
\]

where \(C^*\) is the loading parameter, \(r\) is the distance from the crack tip, and \(I\) and \(\tilde{\sigma}_{ij}\) are non-dimensional coefficients tabulated by Hutchinson. 25 At the crack tip, blunting occurs, and the stresses are locally reduced below the value predicted by Eq. (4). Within this regime, the stresses are approximately given by the stress at \(r = 2b\) (Fig. 18), 26 with \(b\) being the crack tip opening displacement that evolves during steady-state in accordance with 26
FIG. 18
where \( \sigma \) is the stress at the crack tip. Hence, in steady-state

\[
b = 0.6 \left( \frac{C^*}{\sigma_t} \right) \frac{\varepsilon_\infty}{\varepsilon_0}
\]

where \( \varepsilon_\infty \) is the remote strain. Rearranging Eq. (4) gives (\( r > 2b \):

\[
\sigma_{ij}^{n+1} = \left( \frac{\sigma_\infty^n C^*}{1 r \varepsilon_\infty} \right) \tilde{\sigma}_{ij}^{n+1}
\]

Furthermore, noting that

\[
C^* = \sigma_\infty \varepsilon_\infty a h_s
\]

where \( h_s \) is a constant, gives

\[
\frac{\sigma_{ij}}{\sigma_\infty} = \left[ \frac{h_s a (n+1)}{1 r} \right] \tilde{\sigma}_{ij}^{n+1} \quad (r \gg 2b)
\]

\[
\frac{\sigma_{ij}}{\sigma_\infty} = \left[ \frac{h_s a (n+1)}{2Ib} \right] \tilde{\sigma}_{ij} \quad (r \gg 2b)
\]
The $\sigma_{yy}$ stress in the crack plane predicted by Eqs. (9) is plotted in Fig. 19 for $n=2$ (typical of the present experiments), whereupon $h_s = 1.4$, $I = 5.8$, and $\widetilde{\sigma}_{ij} = 1.8$.

When test specimens are cooled under load, the stresses given by Eqs. (9) remain. Hence, when the load is removed at room temperature, residual stresses develop. Load removal is an elastic process, whereupon the unloading stresses close to the tip are:

$$\frac{\sigma_{yy}}{\sigma_\infty} \approx - \left( \frac{2a}{b+2r} \right)^{1/2}$$  \hspace{1cm} (10)

Such that the residual stress $\sigma_{yy}^R$ at the near tip for $n=2$ ($r < 2b$) is:

$$\sigma_{yy}^R = \sigma_\infty \left[ .89 \left( \frac{a}{b} \right)^{1/3} - \left( \frac{2a}{b+2r} \right)^{1/2} \right]$$  \hspace{1cm} (11)

where $\sigma_\infty^C$ is now the remote stress on the body during creep and upon cooling.

Conduct of a room temperature fracture test on specimens cooled under load is governed by superposition of the new elastic field upon the residual field. At fracture, when $\sigma^\infty = S$, the stresses near the tip are then

$$\sigma_{yy} = (S - \sigma_\infty) \left( \frac{2a}{b+2r} \right)^{1/2} + .89 \sigma_\infty \left( \frac{a}{b} \right)^{1/3}$$  \hspace{1cm} (12)
FIG. 19
The fracture process may be analyzed on the basis of the stress field.

3.3 **Properties**

The results of the fracture toughness tests (Table 2) show that \( K_{IC} \) is temperature insensitive in the sintered material. The hot-pressed material has a somewhat higher room temperature toughness than the sintered material, but the toughness diminishes at higher temperature to a level comparable to that of the sintered material. Specimens tested at room temperature after exposure to steady-state at high temperature gave nominal toughnesses larger than the sharp-crack toughness, as indicated in Table 2.

The creep testing revealed an abrupt transition from brittle to ductile behavior (Figs. 20 and 21, Tables 3a and b) such that, in the ductile region, the strain on the tensile surface exceeded 10% without failure. At strain-rates of \(~10^{-7} \text{ s}^{-1}\) the transition for the hot-pressed material occurred between 1650 and 1700°C, and for the sintered material between 1750 and 1800°C. In the ductile range the creep exponent of the hot pressed silicon carbide was 2.3 and for the sintered silicon carbide 1.7. These values are in the range expected for superplastic behavior, consistent with the extensive ductility observed above the transition temperature.
Table 2
Fracture Toughness of SiC as a function of Temperature

<table>
<thead>
<tr>
<th></th>
<th>Test #1</th>
<th>Test #2</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sintered SiC</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Room temperature</td>
<td>2.7 Mpa(\sqrt{m})</td>
<td>2.7 MPa(\sqrt{m})</td>
</tr>
<tr>
<td>1500°C</td>
<td>2.7</td>
<td></td>
</tr>
<tr>
<td>1600°C</td>
<td>2.7</td>
<td>3.0</td>
</tr>
<tr>
<td>1700°C</td>
<td>2.9</td>
<td>2.6</td>
</tr>
<tr>
<td>1800°C</td>
<td>2.7</td>
<td>2.8</td>
</tr>
<tr>
<td><strong>Hot-Pressed SiC</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Room Temperature</td>
<td>3.9</td>
<td>4.0</td>
</tr>
<tr>
<td>1400°C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1500°C</td>
<td>2.8</td>
<td></td>
</tr>
<tr>
<td>1600°C</td>
<td>2.7</td>
<td>2.6</td>
</tr>
<tr>
<td>1700°C</td>
<td>2.4</td>
<td>2.6</td>
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</table>

Room Temperature Tests of Deformed Beams

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Nominal Toughness</th>
</tr>
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<tbody>
<tr>
<td>NC11</td>
<td>5.0</td>
</tr>
<tr>
<td>C11</td>
<td>5.5</td>
</tr>
<tr>
<td>C12</td>
<td>6.0</td>
</tr>
</tbody>
</table>
Hot-pressed SiC

温度

$\leq 1650^\circ C$

$\geq 1700^\circ C$

常温変位速度

图20
Figure 21: Stress-strain curve for sintered α-SiC at constant displacement rate.

- Peak stress for sintering at ≤1750°C.
- Bowing of curve at ≥1800°C.
Table 3a
HOT-PRESSED SiC (NC203)

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Temp (°C)</th>
<th>Strain Rate (sec⁻¹)</th>
<th>Total Strain</th>
<th>Mode</th>
<th>Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NC2</td>
<td>1600</td>
<td>6.8x10⁻⁷</td>
<td>Fracture</td>
<td>199</td>
<td></td>
</tr>
<tr>
<td>NC3</td>
<td>1600</td>
<td>3.4x10⁻⁷</td>
<td>Fracture</td>
<td>184</td>
<td></td>
</tr>
<tr>
<td>NC5</td>
<td>1600</td>
<td>1.7x10⁻⁸</td>
<td>Fracture</td>
<td>414</td>
<td></td>
</tr>
<tr>
<td>NC7</td>
<td>1600</td>
<td>6.8x10⁻⁷</td>
<td>Fracture</td>
<td>252</td>
<td></td>
</tr>
<tr>
<td>NC8</td>
<td>1650</td>
<td>1.7x10⁻⁷</td>
<td>Fracture</td>
<td>127</td>
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<td>NC9</td>
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<td>Flow</td>
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<tr>
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Indented Beams

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<th>Total Strain</th>
<th>Mode</th>
<th>Stress (MPa)</th>
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Table 3b
SINTERED α-SiC (Carborundum)

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Indented Beams

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<th>Total Strain</th>
<th>Mode</th>
<th>Stress (MPa)</th>
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<td>3.2x10⁻⁷</td>
<td>3.5%</td>
<td>Flow</td>
<td>180</td>
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<tr>
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<td>1800</td>
<td>3.2x10⁻⁷</td>
<td>6%</td>
<td>Flow</td>
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<td>Flow</td>
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</tr>
<tr>
<td>C12</td>
<td>1800</td>
<td>4.3x10⁻⁷</td>
<td>6%</td>
<td>Flow</td>
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Indentation cracks above the transition temperature in the sintered material exhibited blunting and opening without growth (Fig. 22). Furthermore, the only damage evident in the material occurred in the vicinity of the blunt tip in the form of cavities that seemingly initiated at graphite inclusions (Fig. 23). In the hot-pressed material, the behavior above the transition temperature had some differing features. In particular, some crack growth occurred (Fig. 24) albeit at a low crack growth rates. For example, the crack velocity was \( \sim 10^{-9} \) m/s at values of the crack driving force \( C^* \approx 0.013 \) N/ms. viz at a nominal \( K \approx 5.5 \) MPa \( \sqrt{m} \). Appreciable damage was also detected in a crack tip zone (Fig. 25) consisting of lenticular shaped facet cavities typical of those apparent during creep crack growth. \(^{28}\)

4. DISCUSSION

The brittle to ductile transition in ceramics involves local competition between flow and fracture. At this simplest level, flow at elevated temperature is governed by steady-state creep and fracture involves the brittle propagation of an initial crack. For nominally single phase SiC materials, brittle fracture from a sharp crack is expected to be a temperature insensitive process, as affirmed by the fracture toughness measurements
FIG. 22

$10 \mu m$

$\epsilon = 3.5\%$

$\epsilon = 6\%$

$\epsilon = 8.5\%$
FIG. 24
Consequently, the 'brittle strength' of the materials is also temperature insensitive, as plotted on Figs. 26 and 27. Conversely, deformation is strongly temperature dependent, as governed by the creep process (Figs. 26 and 27). A transition from brittle to ductile behavior would be expected to occur when the flow stress becomes smaller than the fracture stress. However, the process must occur everywhere in the body. The most critical region is clearly within the crack tip field (see Figs. 18 and 19). Further discussion thus involves consideration of the competing deformation and fracture processes near the crack tip.

4.1 The Flow Stress

The creep process in both materials exhibits characteristics typical of superplasticity: notably a stress exponent, $n \approx 2$, and an activation energy comparable to that for grain boundary diffusion. The creep law thus has the form:

$$\frac{\varepsilon}{D_b} \simeq (\sigma^2 \Omega^{1/3}/\mu kT)(b/d) \quad (13)$$

where $b$ is the Burgers vector, $\Omega$ the molecular volume and $D_b$ the grain boundary diffusion coefficient.
HOT PRESSED

\[ \dot{\varepsilon} = 2.4 \times 10^{-7} \text{s}^{-1} \]

\[ Q = 247 \text{ kJ/mol} \]

\[ \dot{\varepsilon} = 10^{-4} \text{s}^{-1} \]

FIG. 26
\[ \dot{\varepsilon} = 3.2 \times 10^7 \text{ s}^{-1} \]

\[ Q = 563 \text{ KJ/mol} \]
4.2 The Fracture Stress

The onset of crack blunting at elevated temperature clearly changes the brittle fracture process. Prediction of trends in the brittle fracture stress with temperature thus requires that both the operative fracture mechanism in the presence of a blunt crack be established and the rate of blunting be ascertained. Analysis of the tests performed at room temperature on cracks subjected to high temperature blunting provides some information pertinent to these issues.

The present premise is that fracture from blunt cracks is governed by the peak tensile stress in the crack field, \( \sigma \) (Fig. 19). This stress may be derived from the measured strength \( S \) using Eq. (15), and then using \( K \) to estimate the size of the microstructural flaw needed to cause failure; \( a_c \approx (\pi/4)(K_c/\sigma)^2 \). This procedure suggests flaws \( \approx 12\mu m \) in diameter, comparable to the size of the voids observed in the crack tip region. The present hypothesis, therefore, is that the voids are fracture sites and hence that trends in the fracture stress are governed by variations in the peak stress with blunting and by the growth of voids in the crack tip damage zone.
Detailed considerations of trends in fracture stress would include statistical considerations comparable to those used to describe the brittle-to-ductile transition in steels.\textsuperscript{29,30} Insufficient information exists for this purpose. Nevertheless, some insight can be gained by noting the trend in the peak stress during high temperature testing, obtained from Eqs. (6) and (9), viz,

\[
\hat{\sigma}/\sigma_{\infty} \approx 0.93/\sqrt{\varepsilon_{\infty}} \tag{14}
\]

Furthermore, during a typical test, the stress and strain vary as Fig. (28);

\[
\varepsilon_{\infty} \approx \varepsilon_{SS} \tan[(\pi/2)(\sigma_{\infty}/\sigma_{SS})] \tag{15}
\]

where the subscript ss refers to steady-state flow. Combination of Eqs. (14) and (15) reveals a maximum in \(\hat{\sigma}\), which occurs when \(\sigma_{\infty} \approx 0.55 \sigma_{SS}\). Consequently when the steady-state creep stress becomes less than about 1.4 times the fracture stress, creep ductility is assured.
FIG. 28
5. CONCLUSIONS

Silicon carbide exhibits a transition from creep-brittleness to creep-ductility. Below the transition temperature the material fails by brittle crack extension. Above the transition temperature the material is superplastic and can withstand strains in excess of 10%. Indentation cracks above the transition temperature in sintered silicon carbide open and blunt with damage around the crack tip characterized by cavities opening at graphite inclusions. The indentation cracks in the hot-pressed material open, stay sharp, and propagate at a very slow rate with cavity formation on grain facets in front of the crack tip.

The brittle fracture stress of silicon carbide at rapid strain rates is temperature insensitive. However, the deformation stress is strongly dependent on strain rate and temperature. The blunting rate is a deformation process that determines whether the crack will extend. If a crack blunts readily the stress field around its tip is reduced, therefore preventing brittle fracture. Constant strain rate deformation creates a maximum peak stress ahead of a crack tip when the applied stress reaches slightly more than half the flow stress. If the material can survive this point it will continue to deform to large strains.
References


This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

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