STATIC AND CYCLIC FATIGUE OF CERAMIC MATERIALS

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MATERIALS TESTING DIVISION

March 1972

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### STATIC AND CYCLIC FATIGUE OF CERAMIC MATERIALS

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Examination of fracture surfaces by scanning and transmission electron microscopy revealed the fracture to be of a transgranular and intergranular nature for both materials. Fracture initiated at the tension surface and propagated immediately to failure. There was no change in fracture appearance in either the static or cyclic mode of failure. (Authors)
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Technical Report by
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ABSTRACT

Static and cyclic fatigue characteristics were determined for two ceramic materials, an alumina AD-94 and a hot-pressed boron carbide B₄C. The static fatigue limits determined in three-point flexure were 23,500 psi for Al₂O₃ and 43,000 psi for B₄C, while the cyclic fatigue limits determined by stressing a cantilevered beam were 21,700 psi for the Al₂O₃ and 28,500 psi for the B₄C.

Examination of fracture surfaces by scanning and transmission electron microscopy revealed the fracture to be of a transgranular and intergranular nature for both materials. Fracture initiated at the tension surface and propagated immediately to failure. There was no change in fracture appearance in either the static or cyclic mode of failure.
INTRODUCTION

There is increased emphasis on the structural use of armor material as evidenced, for example, by the proposed Aerial Armored Reconnaissance Vehicle.\textsuperscript{1,2} This vehicle utilizes monocoque construction with steel armor plate serving as both the skin and primary structure. It is also possible that ceramic composite armor, i.e., ceramic facing bonded to a suitable backup material may be considered for a nonprimary but critical application, especially under severe ballistic environment. However, additional information is needed regarding the behavior of candidate materials that will serve in both a load-carrying and ballistic protection capacity. A program is underway with the objective of characterizing candidate materials. The present study covers an initial phase of this characterization of ceramic composite armor by focusing on the static and cyclic fatigue response of two candidate ceramic materials, Al\textsubscript{2}O\textsubscript{3} and B\textsubscript{4}C.

MATERIALS AND TEST PROCEDURES

Two materials were selected for investigation; (1) AD-94, a commercially available alumina Al\textsubscript{2}O\textsubscript{3}, manufactured by Coors Porcelain Company, Golden, Colorado; and (2) a hot-pressed boron carbide B\textsubscript{4}C, manufactured by the Norton Company, Worcester, Massachusetts. The microstructures of these materials are shown in Figure 1, and their properties are given in the following table:

![Figure 1. PHOTOMICROGRAPHS OF MATERIALS TESTED. Mag. 500X](image)

Table I. Physical Properties

<table>
<thead>
<tr>
<th>Material</th>
<th>Flexural Strength</th>
<th>Modulus</th>
<th>Density</th>
<th>Grain Size</th>
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<tr>
<td>Al\textsubscript{2}O\textsubscript{3}</td>
<td>38,000 psi</td>
<td>41.5 \times 10^6 psi</td>
<td>3.71 g/cc</td>
<td>10\mu</td>
</tr>
<tr>
<td>B\textsubscript{4}C</td>
<td>51,000 psi</td>
<td>60 \times 10^6 psi</td>
<td>2.43 g/cc</td>
<td>10\mu</td>
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a. Alumina 94%  
b. Boron Carbide
The static fatigue tests were conducted in three-point bending with a dead-weight load suspended from the center of the specimen. The specimen size for both the static and cyclic fatigue tests was \( \frac{1}{4}'' \times \frac{1}{4}'' \times 3'' \) with a surface finish of \( 10 \) \( \text{RMS} \).

For purposes of this study, cyclic fatigue has been defined as an intermittently applied load to a specimen such that the stress on the surface of the specimen follows a sinusoidal excursion varying from zero to a maximum tensile or compressive stress, depending on the applied load. The dynamic test consisted of applying a load at the end of a cantilevered specimen at the rate of 15 cps. The equipment utilized in this test has been described elsewhere.

The arbitrary cut-off limit for the cyclic test was \( 1 \times 10^6 \) cycles in the event failure did not occur. Several tests beyond this arbitrary limit were conducted; these tests indicated that a limit of \( 1 \times 10^6 \) cycles was justified.

**RESULTS AND DISCUSSION**

The flexural strength of the \( \text{Al}_2\text{O}_3 \) when loaded to failure was 38,000 psi; this will be referred to as the reference strength, Figure 2. At 75\% of the reference strength life was limited to 10 seconds. Decreasing the stress level to 70\% or 27,000 psi increased the static fatigue life to 15-1/2 hours. At 60\% of the reference strength or 23,000 psi, life was extended to 72 hours, at which time the test was terminated, it being assumed that life would be infinite at this stress level. The specimens that were not fractured in the static tests were reloaded to failure, with failure occurring at 35,000 psi or within 6 or 7\% of the reference strength, indicating that virtually no damage was incurred within the test specimen during the static test. It would thus appear that this material can be loaded statically at 60\% of the reference level for an indefinite period of time.

Static fatigue tests on \( \text{B}_4\text{C} \) were conducted under the same atmospheric conditions as for the \( \text{Al}_2\text{O}_3 \), namely 72 F and 35\% R.H. The reference strength of the \( \text{B}_4\text{C} \) was determined to be 51,000 psi. At 50\% of this value or 46,000 psi, fracture occurred after 61 minutes; at 75\% and 73\% of the reference strength, static fatigue life exceeded the arbitrary limit of 72 hours. The specimens that did not fracture in the static tests were reloaded, and in each case fracture occurred at 97\% of the reference strength, indicating once again that static fatigue had no detrimental effect on the strength of the material.
One of the more predominant mechanisms leading to fracture is stress corrosion,* and it is felt further tests at humidities approaching 100% and also tests conducted in a vacuum would elucidate the role of stress corrosion in fatigue. Also, additional tests to definitely establish the observed trend are planned.

Several techniques have been reported for increasing the strength of ceramic materials. Sample,* for example, introduced a compressive stress on the surface of Al₂O₃ specimens by a heat treat process, and subsequent leading of the heat-treated specimens showed an increase in strength over the untreated specimens. Kirchner et al.,* recognizing that failures occur at surface flaws, treated the surfaces by refiring, chemical and flame polishing, chemical etching, and glazing. These treatments also proved to be beneficial in increasing the strength of the treated material. The effect that these treatments might have on improving the fatigue life of the Al₂O₃ and B₄C should also be investigated.

The results obtained for the cyclic testing of the Al₂O₃ and B₄C are shown in Figure 5. The reference strengths for each material are the same as in the static tests. The threshold level is defined as that level of stress below which an infinite fatigue life is anticipated. The threshold level for both the Al₂O₃ and B₄C in cyclic fatigue was determined to be at 5% of the reference strength for each material. The data for Al₂O₃ compares favorably with that reported by Williams* where he reports a "cyclic endurance limit of 0.56 of the single stroke strength."

Scanning electron microscopy (SEM) of the fracture surface, Figure 4, indicates fracture is of both a transgranular and intergranular nature. The location of the area shown in the micrograph is approximately at the center of the fracture surface. Transmission electron microscopy of the fracture surface of Al₂O₃,

Figure 3. FLEXURAL FATIGUE OF CERAMIC MATERIALS

*Discussion with G. R. Gross, Midwest Research Institute, Kansas City, Missouri.
Figure 5 indicates that the transgranular fracture involves cleavage. It would also appear that there are some cleavage steps shown in the SEM micrograph of the BaC, Figure 4.

Fracture mechanics concepts, based upon the Griffith criterion, were used to determine strain energy release rate and critical flaw size for the two materials. The strain energy release rate was determined by fracturing a V-notched specimen, 1/4" × 1/4" × 2-1/2", having a notch depth of 0.050 inch. The specimens were fractured in three-point bending as outlined by Davidgr and Tappin who defined the strain energy release rate G by
Figure 5. FRACTURE SURFACE OF $\text{Al}_2\text{O}_3$
BY TEM (CARBON REPLICA) Mag. 15,000X

\[ G = \frac{(1-\nu^2)\pi \sigma_F^2 c}{2E} \]  

where $\nu$ is Poisson’s ratio; $E$ is Young’s modulus in pounds per square inch; $\sigma_F$ is the fracture stress in pounds per square inch; and $c$ is the notch depth in inches.

Notched specimens of both $\text{Al}_2\text{O}_3$ and $\text{B}_4\text{C}$ were fractured in an Instron testing machine at a crosshead speed of 0.020 inch per minute. Substituting the test data into Equation 1, the strain energy release rate for $\text{Al}_2\text{O}_3$ was 0.270 in-lb/sq in. ($4.73 \times 10^4$ erg/cm$^2$) and for the $\text{B}_4\text{C}$, 0.192 in-lb/sq in. ($3.36 \times 10^4$ ergs/cm$^2$). For the $\text{Al}_2\text{O}_3$, the strain energy release rate compares favorably with 0.302 in-lb/sq in. reported by McKinney. Care should be exercised in comparing fracture energy data, since Gross and Gotshalk found that the strain energy release rate was grain-size dependent.

From the Griffith criterion

\[ G_C = \frac{\sigma^2 a}{E} \]  

Solving Equation 2 for $a$, $a = (G_CE)/\sigma^2$. If $\sigma$ is assumed to be the fracture stress of an unnotched bar, then it is possible to estimate the critical flaw size from Equation 2. From the test data, it was found that the critical flaw size for $\text{Al}_2\text{O}_3$ was 0.003 inch, approximately 75 μm or about 8 grain diameters; flaws of this size should be readily detected by nondestructive techniques. The critical flaw size for $\text{B}_4\text{C}$ was found to be 0.0015 in., approximately 40 μm, or about 4 grain diameters. It should be pointed out that flaws of this nature were not observed in the examination of the fracture surfaces by scanning electron microscopy. Additional tests are planned in the area of fracture energy measurements.
The static and cyclic fatigue properties of some materials, an alumina AD-94 and a hot-pressed boron carbide B₄C, have been studied. The static fatigue strength of the Al₂O₃ at 72 hours was 60% of the reference strength, and for the B₄C the static fatigue for 72 hours was 74% of the reference strength. Static fatigue was determined by applying a dead-weight load to the center of a specimen of 1/4" x 1/4" x 3" dimensions; the specimen was supported at a span length of 2-1/2 inches.

The cyclic fatigue properties were determined by applying a load to the end of a cantilevered specimen. The specimen size corresponded to that used for static fatigue, with the cantilevered span being 2-1/2 inches; this ensured the stressed volume being the same for both the static and cyclic fatigue tests. The threshold level for safe cyclic fatigue conditions for both Al₂O₃ and B₄C appears to be at 57% of the reference strength level. Examination of the fracture surface revealed the fracture to be of a transgranular and intergranular nature, with the fracture initiating at the tension surface; however, no evidence was present of intermittent progressive crack propagation. This latter observation would indicate that once the crack was initiated, propagation was instantaneous.

From fracture mechanics concepts it was calculated that the strain energy release rate was 0.270 in-lb/sq in. for Al₂O₃ and 0.192 in-lb/sq in. for B₄C, and the critical flaw size was determined to be 75µm for Al₂O₃ and 40µm for B₄C.
LITERATURE CITED


